SDMS Document
122965

Cornell-Dubilier Electronics Superfund Site

Quality Assurance Project Plan
OU3 Groundwater

Prepared by:

Malcolm Pirnie, Inc

December 2008

Version: 2008/12/17

Document Control Number: CDOU3-05

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APPENDIX

Appendix A Cornell-Dubilier OU-3 Field Sampling Plan

ACRONYMS

ARAR	Applicable, Relevant, and Appropriate Requirement
ASB	Analytical Services Branch
ASTM	ASTM International. ASTM is a non-profit standards organization formerly called the American Society for Testing and Materials
BCI	Bioremediation Consulting Inc.
ca	Carcinogens
CCC	Continuous Calibration Check
CDE	Cornell-Dubilier Electronics
CERCLA	Comprehensive Environmental Response, Compensation and Liability Act
CHSM	Corporate Health and Safety Manager
CLP	Contract Laboratory Program
COC	Chain of Custody
COPC	Compound of Potential Concern
CRQL	Contract Required Quantitation Limit
CVAA	Cold Vapor Atomic Adsorption
DESA	Division of Environmental Science and Assessment
D	Replicate
DNAPL	Dense non-aqueous phase liquid
DoD	Department of Defense
DOT	Department of Transportation
DPM	Deputy Project Manager
DQA	Data Quality Audit
DQI	Data Quality Indicator
DQO	Data Quality Objectives
ECD	Electronic Capture Detector
EDD	Electronic Data Deliverable
FORMS II Lite	Field Operations Records Management System II Lite
FSP	Field Sampling Plan
FTL	Field Team Leader
GC	Gas Chromatography
GC-ECD	Gas Chromatography-Electron Capture Detector
GC-FID	Gas Chromatography-Flame Ionization Detector
GC-HRMS	Gas Chromatography-High Resolution Mass Spectrometry
GC-MS	Gas Chromatography-Mass Spectrometry
HASP	Health and Safety Plan
IC	Initial Calibration
LCS	Laboratory Control Sample

LIMS	Laboratory Information Management System
LNAPL	Light non-aqueous phase liquid
MCLs	Maximum Contaminant Levels
MDL	Method Detection Limit
mg/kg	Micrograms per kilogram
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NA	Not Applicable
nc	Non-Carcinogens
NELAP	National Environmental Laboratory Accreditation Program
NJ	New Jersey
NJDEP	New Jersey Department of Environmental Protection
OSHA	Occupational Safety and Health Administration
OU	Operable Unit
OU-3	Operable Unit -3
PA	Performance Audit
PE	Performance Evaluation
PCB	Polychlorinated Biphenyl
PCDDs/PCFFs	Polychlorinated Dibenzodioxins and Dibenzofurans
PCIH	Project Certified Industrial Hygienist
PE	Performance Evaluation
PT	Performance Test
PID	Photoionization Detector
PO	Project Officer
PM	Project Manager
ppb	parts per billion
ppm	parts per million
PQOs	Project Quality Objectives
PSO	Project Safety Officer
PT	Performance Testing
QA	Quality Assurance
QAM	Quality Assurance Manager
QAPP	Quality Assurance Project Plan
QC	Quality Control
QL	Quantitation Limit
QSM	Quality Systems Manual
R	Recovery
RDCSCC	Residential Direct Contact Soil Cleanup Criterion
RI	Remedial Investigation

RI/FS	Remedial Investigation/Feasibility Study
RL	Reporting Limit
ROD	Record of Decision
RPD	Relative Percent Difference
RSCC	Regional Sample Control Center
SA	Spike added to spiking matrix
SEI	Stone Environmental Inc
SDG	Sample Delivery Group
SMO	Sample Management Officer
SOP	Standard Operating Procedure
SOW	Statement of Work
SR	Spike result
SSR	Spike sample result
SVOCs	Semi-Volatile Organic Compounds
TAL	Target Analyte List
TAT	Turnaround Time
TBC	To Be Considered
TBD	To Be Determined
TCL	Target Compound List
TD	Technical Director
TSA	Technical Systems Audit
µg /kg	Microgram per Kilogram
UFP	Uniform Federal Policy
USACE-KCD	United States Army Corps of Engineers-Kansas City District
USEPA	United States Environmental Protection Agency
VOCs	Volatile Organic Compounds

INTRODUCTION

This Quality Assurance Project Plan (QAPP) is intended to integrate the technical and quality control aspects of the Cornell-Dubilier Electronics (CDE) Superfund Site (Site) groundwater sampling and analysis program for Operable Unit 3 (OU3), and is supplemented by detailed information in the Field Sampling Plan (FSP) in Appendix A. It details the planning processes for collecting data and describes the implementation of the quality assurance (QA) and quality control (QC) activities developed for this program. The purpose of this QAPP is to generate project data that are technically valid and legally defensible. The QAPP consists of four main components:

- Project Management;
- Measurement and Data Acquisition;
- Assessment and Oversight; and
- Data Validation and Usability.

The above components will incorporate QA/QC requirements cited within the following documents:

- U.S. Environmental Protection Agency (USEPA) Requirements for Quality Assurance Project Plans, USEPA QA/R-5, March 2001.
- USEPA Guidance for the Data Quality Objectives Process, QA/G-4, August 2000.
- Uniform Federal Policy for Quality Assurance Project Plans, Final Version March 2005

Project Background

The Site has been divided into four Operable Units (OUs) by the U.S. Environmental Protection Agency (USEPA). Operable Unit 1 (OU1) addresses residential, commercial, and municipal properties in the vicinity of the former CDE facility. On September 30, 2003, the USEPA signed a Record of Decision (ROD) to address OU1. Operable Unit 2 (OU2) addresses contaminated soils and buildings at the former CDE facility. On September 30, 2004, the USEPA signed a ROD to address OU2. Operable Unit 3 (OU3) addresses contaminated groundwater and Operable Unit 4 (OU4) addresses the Bound Brook.

The former CDE facility is located at 333 Hamilton Boulevard, South Plainfield, Middlesex County, New Jersey. The former CDE facility, recently known as the Hamilton Industrial Park, consists of approximately 26 acres. CDE manufactured electronic components including, in particular, capacitors in South Plainfield from 1936 to 1962. Polychlorinated biphenyls (PCBs) and chlorinated organic solvents were used in the manufacturing process. CDE disposed of PCB-contaminated materials and other hazardous substances directly on the facility soils. These activities led to widespread chemical contamination at the facility. Elevated levels of volatile organic chemicals (VOCs) and PCBs have been found in soils at the facility, in soils at adjacent properties (residential, commercial, and municipal), in groundwater from the CDE facility, and in the surface water and sediments of Bound Brook.

This Remedial Investigation/Feasibility Study (RI/FS) for OU3 is designed to collect sufficient data to define the nature and extent of groundwater contamination, assess chemical mobility, identify migration pathways, perform an assessment of human health risks, and evaluate potential remedial alternatives for groundwater.

TOTAL P.002

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

Site Name/Project Name: Cornell-Dubilier Electronics Superfund Site Operable Unit 3
Site Location: South Plainfield, New Jersey

Document Title: Cornell-Dubilier Electronics Superfund Site QAPP OU3 Groundwater

Lead Organization: USEPA Region 2

Preparer's Name and Organizational Affiliation: James McCann and Janis Karn of Malcolm Pirnie, Inc

Preparer's Address, Telephone Number, and E-mail Address:
James McCann and Janis Karn, Malcolm Pirnie, Inc. 17-17 Route 208 North, Fair Lawn, NJ 07410,
e-mail: jmccann@pirnie.com and jkarn@pirnie.com

Preparation Date (Day/Month/Year): 12/17/08

Investigative Organization's Admin. Project Manager/Date: 12/17/08
Signature
Printed Name/Organization: Edward Dudek, P.E., Malcolm Pirnie, Inc

Investigative Organization's Technical Project Manager/Date: 12/17/08
Signature
Printed Name/Organization: Daniel St. Germain., Malcolm Pirnie, Inc

Investigative Organization's Project QA Officer/Date: 12/17/08
Signature
Printed Name/Organization: James McCann, Malcolm Pirnie, Inc

Lead Organization's Project Manager/Date: 12/19/08
Signature
Printed Name/Organization: Pietro Mannino, USEPA Region 2 Project Manager

Approval Signatures/Date: 12/17/08
Signature
Printed Name/Title: Kenneth Maas, USACE-KCD Project Manager

Approval Authority: U.S. Army Corps of Engineers, Kansas City (USACE-KCD)

DEC-19-2008 09:23 EPA REGION 2 1 212 637 4429 P.002

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

Site Name/Project Name: Cornell-Dubilier Electronics Superfund Site Operable Unit 3 (OU3)
Site Location: South Plainfield, New Jersey
Site Number/Code: EPA ID# NJD981557879
Operable Unit: 3
Contractor Name: Malcolm Pirnie Inc
Contract Number: USACE W912DQ-06-D-0006

Title: Cornell-Dubilier Electronics Superfund Site OU3 QAPP
Revision Number: 1
Revision Date: December 2008

Contract Title: Indefinite Delivery/Indefinite Quantity Type Contract for Miscellaneous Military and Civil Hazardous Waste Cleanup Projects and Related Work
Work Assignment Number: Task Order 0025

1. Regulatory Program: Comprehensive Environmental Response, Compensation, & Liability Act (CERCLA)
2. Approval Entity: USEPA Region 2
3. The QAPP is (select one): ☐Generic ☒Project Specific
4. Dates of scoping sessions that were held: April through October 2008
5. Dates and titles of QAPP documents written for previous Site work, if applicable:

Title	Approval Date
Foster Wheeler, 2000, Final Quality Assurance Project Plan for Remedial Investigation Study, Cornell-Dubilier Electronics Superfund Site, South Plainsfield, Middelsex County, New Jersey, Foster Wheeler Envrionmental Corporation	March 2000
Final Quality Assurance Project Plan for Remedial Investigation/Feasibility Study, Cornell-Dubilier Electronics Superfund Site, South Plainfield, Middlessex County, New Jersey, Foster Wheeler Environmental Corporation-Operable Unit 2	March 2002
Final Buildings Quality Assurance Project Plan for Cornell Dubilier Electronics Superfund Site, South Plainfield, NJ, Operable Unit 2	January 2006
Soils Quality Assurance Project Plan, Cornell-Dubilier Electronics Superfund Site, South Plainfield, NJ, Operable Unit 2 (This document describes the requirements for characterizing the nature and extent of hazardous constituents in the soils at the former CDE facility.)	August 2006
Final Quality Assurance Project Plan Cornell-Dubiler Electronics Superfund Site Operable Unit-2: Capacitor Disposal Area Soil Split Sample Analysis Program	March 2008

6. Organizational Partners (stakeholders) and connection with Lead Organization:
The primary project organizational partners include representatives from USEPA Region 2, USACE-KCD, New Jersey Department of Environmental Protection (NJDEP) and Malcolm Pirnie, Inc. (Malcolm Pirnie). USEPA Region 2 and USACE-KCD will provide project and contract management guidance to Malcolm Pirnie. Malcolm Pirnie will be the primary consultant and will be responsible for developing and implementing the investigation and will provide project management for other subcontractors.

7. Data users: USEPA Region 2 and Malcolm Pirnie
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)

Required QAPP Element(s) and Corresponding QAPP Section(s)	Required Information	Crosswalk to Worksheet # or 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 Scoping Session Documentation (including Data Needs tables) - Project Scoping Session Participants Sheet - Problem Definition, Site History, and Background - Site Maps (historical and present)	9 plus meeting minutes Data Needs Table in Attachment 1.2 10 and Attachment 1.1 Introduction and 13 See Field Sampling Plan for maps (Attachment 1).
2.6 Project Quality Objectives (PQOs) 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 –See Attachment 1.1 for Data Quality Objectives (DQOs) 12

QAPP Worksheet #2
QAPP Identifying Information
(Continued)

Required QAPP Element(s) and Corresponding QAPP Section(s)	Required Information	Crosswalk to Worksheet # or Related Documents
2.7 Secondary Data Evaluation	- Sources of Secondary Data and Information - Secondary Data Criteria and Limitations Table	13
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/Standard Operating Procedure (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 See FSP Section 3 18 and FSP 19 20 See FSP 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 Worksheet of Related 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 Attachment 3 SOP No. 1 27 and Attachments 4 and USPEA Forms II Lite software User's Guide
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 Attachment 6 SOP No. 6 32
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		36

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

The following persons will receive a hardcopy of the approved QAPP, subsequent QAPP revisions, addenda, and amendments:

Distribution List

QAPP Recipients	Title	Organization	Telephone Number	Fax Number	E-mail Address	Document Control Number
Pietro Mannino	USEPA Project Manager	USEPA Region 2	212-637-4395	212-637-4429	Mannino.pietro@epa.gov	CDOU3-01
Amy Darpinian	USACE Project Chemist	USACE-KCD	816-389-3897		Amy.F.Darpinian@usace.army.mil	CDOU3-02
TBD	Field Team Leader	Malcolm Pirnie Inc				CDOU3-03

Electronic copies of the final QAPP and related project documents will also be available in the project directory and the project database for the personnel named in the organization chart given in Worksheet 5, Figure 1 and other Malcolm Pirnie personnel who will be assigned to work on the project. Those named above will be responsible for distributing the QAPP and related documents to others in their organization.

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.

Project Personnel Sign-Off Sheet

Organization: USEPA Region 2

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Pietro Mannino	Project Manager –USEPA Region 2	212-637-4395	Peter M	11/26/2008

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.

Project Personnel Sign-Off Sheet

Organization: USACE-KCD

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Kenneth Maas	Project Manager – USACE-KCD	816-389-3709		4 Nov 08

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.

Project Personnel Sign-Off Sheet

Organization: USACE-KCD

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Amy Darpinian	Project Chemist – USACE-KCD	816-389-3897	Amy Darpinian	17 Dec 08

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.

Project Personnel Sign-Off Sheet

Organization: Malcolm Pirnie Inc

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Edward Dudek, PE	Project Manager (Administrative)	914-641-2686		11/4/2008
Dan St. Germain, CPG	Project Manager (Technical)	201-398-4381		11/4/2008
Janis Karn	Deputy Project Manager	201-398-4336		11/5/2008
James McCann	Project Quality Officer	201-398-4310		11/5/2008

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.

Project Personnel Sign-Off Sheet

Organization: Malcolm Pirnie Inc

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
TBD	Field Team Leader			
TBD	Sample Management Officer			
TBD	Field Team Members		Project field team members, when assigned, will be required to sign that they have read applicable sections of the QAPP.	Field team members must read applicable sections of the QAPP and SOPs prior to participating in the project.

QAPP Worksheet #5
(UFP-QAPP Manual Section 2.4.1)

Project Organizational Chart

The Organization Chart, provided as Figure 1, the description of project organization and the roles of the team members are summarized below:

Project/Task Organization Overview

The project management team will consist of representatives from USEPA Region 2, USACE-KCD, the NJDEP, and Malcolm Pirnie. USEPA Region 2 and USACE-KCD will provide technical oversight to the project and contract management guidance to Malcolm Pirnie. NJDEP will provide USEPA with State approval during the planning and investigation. Malcolm Pirnie will be the primary contractor, will be responsible for developing and implementing the investigation, and will provide project management for the other subcontractors. Figure 1 presents the project organization.

Cornell-Dubilier Team Members

This section contains a description of the project organizational structure. Pietro Mannino is the USEPA Project Manager with responsibility for OU3 at the CDE Superfund Site. Kenneth Maas is the USACE-KCD Project Manager. Malcolm Pirnie will be the primary contractor, will be responsible for developing and implementing the investigation, and conduct project management for other subcontractors. Additional project team members from other companies may be subcontracted to Malcolm Pirnie.

Project Officer – The Project Officer (PO) is responsible for the commitment of resources required to fulfill Malcolm Pirnie's obligation to the USACE.

Project Manager – The Project Manager (PM) is accountable to the PO throughout the duration of the project. The PM will be the primary point of contact with the USACE. The PM may delegate authority to expedite and facilitate the implementation of the project

plan. The PM is responsible for:

- Coordination with the USACE.
- Budget control.
- Subcontractor performance.
- Project coordination to implement Work Plans.
- Allocation of staffing and resources to implement the QA/QC program and the Site Safety and Health Plan (SSHP).
- Review of engineering and interim reports.

[Note: For OU-3, PM is a shared role. Ed Dudek is Malcolm Pirnie's Administrative PM responsible for all administrative aspects of the project, and Dan St. Germain is Malcolm Pirnie's Technical PM responsible for all technical aspects of the project. This breakout of duties is warranted due to Malcolm Pirnie's involvement in three other OUs at the Site and Mr. Dudek's involvement in the other OUs.]

Deputy Project Manager – The Deputy Project Manager (DPM) reports directly to, and works with, the Project Manager. The DPM is responsible for assisting the Project Manager, as needed, with project related issues.

Project Quality Consultants – The Project Quality Consultants are responsible for independent reviews of project quality. The Project Quality Consultants make an integral contribution to the project success by performing technical reviews throughout all project phases and offering technical guidance.

Corporate Health and Safety Manager – The Corporate Health and Safety Manager (CHSM) is responsible for development and implementation of Malcolm Pirnie's Health and Safety program. The CHSM serves as the administrator of Malcolm Pirnie's Corporate Health and Safety program. He is responsible for:

- Proper training for Malcolm Pirnie field personnel.
- Medical clearance of Malcolm Pirnie field personnel.
- Field personnel having adequate experience with personal protective equipment.
- Providing guidance on data interpretation.
- Determining levels of worker protection.

Project Certified Industrial Hygienist – The Project Certified Industrial Hygienist (PCIH) is responsible for development, implementation and review of the Site Safety and Health Plan. The PCIH functions as a liaison with the USACE, the Occupational Safety and Health Administration (OSHA), and other agencies on health and safety issues.

Project Safety Officer – The Project Safety Officer (PSO) is knowledgeable in safety and worker protection techniques as they relate to the project. Responsibilities include monitoring daily compliance of work to the SSHP, having the ability and authority to make needed changes or additions to the SSHP and providing technical assistance to the Project Manager on problems relating to work safety.

The PSO is responsible for the development and set-up of emergency procedures and personnel decontamination procedures. The PSO or designee will complete a daily diary of activities with health and safety relevance. If unsafe work conditions are encountered, the PSO is authorized to stop work. Resolution of all health and safety problems will be coordinated through the Project Manager with assistance from the PCIH.

Project Quality Control Officer – The Project QC Officer is responsible for project specific supervision and monitoring of the QA program and reports to the Project Manager. Additional responsibilities include:

- Ensuring that field personnel are familiar with and adhere to proper sampling procedures, field measurement techniques, sample identification, and chain-of-custody procedures.
- Coordinating with the analytical laboratory for the receipt of samples, the reporting of analytical results, and recommending corrective actions to correct deficiencies in the analytical protocol or sampling.
- Preparing QA reports to management.

Field Team Leader – The Field Team Leader will serve as the contact person for Malcolm Pirnie for field investigations and activities. The Field Team Leader will be responsible for the logistics of the field activities. The Field Team Leader will:

- Inspect and replace equipment.
- Prepare interim field reports.
- Prepare samples for shipment.
- Coordinate field activities.
- Schedule sampling and other field activities.

Malcolm Pirnie Field Personnel – All field personnel are required to become thoroughly familiar with the FSP, QAPP, and the SSHP, and follow the guidelines outlined in them. Field personnel will implement the plans and contribute any appropriate suggestions and assist in discovering or correcting non-conforming working procedures.

Data Validator (to be assigned if necessary) – For this project we anticipate that the majority of the data validation will be performed by the USEPA, since the majority of the analytical data will generated by the USEPA's Division of Environmental Assessment (DESA) laboratory or USEPA Contract Laboratory Program laboratories. Qualified Malcolm Pirnie data validation specialists will

also be available and will be assigned as required to validate any non-CLP data from a subcontract laboratory.

Subcontractor Team Members

Subcontractors will be utilized for performance of specific work activities associated with the CDE Superfund Site field investigations. The following is a list of possible services to be subcontracted:

Laboratories (to be determined) - It is anticipated that the DESA laboratory or a CLP laboratory will perform most of the required chemical analyses; however, it is anticipated that subcontract laboratories such as Accutest and Golder Associates will be required to perform some specific analyses.

Mobile Lab for Bedrock Processing and Analysis– It has been proposed that Stone Environmental will be retained as a subcontractor to collect and process bedrock samples and provide rapid analyses for selected volatile chlorinated organic compounds.

Other Contractor(s) (to be determined) – Other subcontractors will be employed for functions such as drilling, rock coring, and surveying. The activities of these subcontractors are described in the FSP.

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

Communication Pathways

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, Pathways, etc.)
Approval of Amendments to the QAPP	Malcolm Pirnie, Inc.	Project Quality Officer (Jim McCann or designee)	201-398-4310	Obtain initial approval from the Investigative Organization PM and submit documented amendments within 10 working days to USACE-KCD/USEPA for approval.
Document and Records Control	Malcolm Pirnie, Inc.	Deputy Project Manager (Janic Karn or designee)	201-398-4336	Project Document Preparation and distribution. Document and records control posting procedure implemented within 5 working days of receipt by Malcolm Pirnie.
Stop Work and Initiation of Corrective action	Malcolm Pirnie, Inc.	Project Managers or Deputy Project Manager (Ed Dudek, Dan St. Germain, or Janis Karn, or designees)	E. Dudek: 914-641-2686 Dan St. Germain: 201-398-4381 Janis Karn: 201-398-4336	The PM communicates within 24 hours of stop work to the project organization by phone, with confirming e-mail.
Real time modification, notifications and approval	Malcolm Pirnie, Inc.	Project Quality Officer (Jim McCann or designee) with PM/DPM approval.	201-398-4310	Real time modification to the project will require the approval of the Project Quality Officer and PM or DPM (or designees) and will be documented using the Field Modifications Form in Attachment 4 within 5 working days.
Reporting of serious issues	Malcolm Pirnie, Inc.	Project Managers or Deputy Project Manager (Ed Dudek, Dan St. Germain, Janis Karn or designees)	E. Dudek: 914-641-2686 Dan St. Germain: 201-398-4381 Janis Karn: 201-398-4336	Report any serious issues to the USEPA and USACE-KCD and other concerned parties by e-mail or memo.
Meeting Minutes	Malcolm Pirnie, Inc.	Deputy Project Manager (Janis Karn or desginee)	201-398-4336	Post approved meeting minutes or distribute by e-mail within 5 working days of meeting.
Corrective action, audit finding	Malcolm Pirnie, Inc.	Project Quality Officer (James McCann or designee)	201-398-4310	Problems or negative audit findings are reported to the PM or DPM by e-mail within 3 days.

QAPP Worksheet #7
(UFP-QAPP Manual Section 2.4.3)

Personnel Responsibilities and Qualification Table

Name	Title	Organizational Affiliation	Responsibilities	Education and Experience Qualifications
Kenneth Maas	USACE-KCD Project Manager	USACE KCD	Project Manager	Professional Engineer
Edward Dudek, PE	Senior Associate	Malcolm Pirnie, Inc.	Administrative Project Manager	Professional Engineer (NY), ME Civil Engineering, BE Civil Engineering
Dan St. Germain	Senior Associate	Malcolm Pirnie, Inc.	Technical Project Manager	BS Marine Geology Professional Geologist
Janis Karn	Senior Project Environmental Scientist	Malcolm Pirnie, Inc.	Deputy Project Manager	BS - Experienced Environmental Scientist
James McCann	Senior Project Chemist	Malcolm Pirnie, Inc.	Project Quality Officer	MA/BS in Chemistry, 40+ years of experience in analytical chemistry, environmental testing, and quality assurance

Note: Resumes of Malcolm Pirnie, Inc. project team members can be obtained by contacting the Malcolm Pirnie PM or designee.

QAPP Worksheet #8
(UFP-QAPP Manual Section 2.4.4)

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 Team	Safety and OSHA training and medical monitoring as specified in the SSHP	Malcolm Pirnie, Inc.	Training dates kept in company/project training records	All field team members working on Site.	All Malcolm Pirnie, Inc. and subcontractor personnel working on Site	Malcolm Pirnie, Inc. Project Files
Sample Management and Creation of sample chain of custody (COC) records	Forms II Lite Software Training	USEPA on-line training	Training dates kept in company/project training records	Sample Management Officer or designee assigned to log samples into Forms II Lite	Malcolm Pirnie, Inc. staff preparing COC forms.	Malcolm Pirnie, Inc. Project Files

Note: Training Records will be kept in the Malcolm Pirnie, Inc. project files.

QAPP Worksheet #9
(UFP-QAPP Manual Section 2.5.1)

Numerous project team meetings and conference calls were held to plan the project from April through June 2008. These included weekly conference calls internal to the Malcolm Pirnie Investigative team to review/plan the project and discuss any open issues. In addition, biweekly conference calls have been held with USACE, USEPA, and Malcolm Pirnie team representatives to discuss the project objectives and status. Visits to the Site are made by team members as necessary to evaluate conditions and gather information needed to develop project plans.

Biweekly Project Team Participants:

Project Scoping Session Participants Sheet

Project Name: Cornell-Dubilier Superfund Site OU3 **Projected Date(s) of Sampling:** Fall 2008 **Project Managers:** Edward Dudek, PE and Dan St. Germain	**Site Name:** Cornell-Dubilier Electronics Superfund Site Operable Unit 3 **Site Location:** 333 Hamilton Boulevard, South Plainfield, New Jersey

Date of Session: April thru June 2008
Scoping Session Purpose: Review the status of project plans.

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Kenneth Maas	Project Manager	USACE-KCD	816-389-3709	Kenneth.E.Maas@nwk02.usace.army.mil	USACE-KCD Project Manager
Pietro Mannino	USEPA Project Manager	USEPA Region 2	212-637-4395	Mannino.pietro@epa.gov	USEPA Project Manager
Edward Dudek, PE	Senior Associate	Malcolm Pirnie	914-641-2686	Edudek@pirnie.com	Admin Project Manager
Janis Karn	Senior Project Environmental Scientist	Malcolm Pirnie	201-398-4336	Jkarn@pirnie.com	Deputy Project Manager

Other team members from the Malcolm Pirnie investigative team and representatives of the USACE and USEPA participated as necessary in the bi-weekly planning calls.

Comments/Decisions: Were documented in meeting minutes stored in Malcolm Pirnie files and distributed to team members by e-mail.
Action Items: Malcolm Pirnie personnel were assigned to prepare the OU3 documents.
Consensus Decisions: Decisions were documented in meeting minutes, which were distributed by e-mail. Minutes of meetings are stored in Malcolm Pirnie files.

Weekly Malcolm Pirnie Team Participants:
Project Scoping Session Participants Sheet

Project Name: Cornell-Dubilier Superfund Site OU3 **Projected Date(s) of Sampling:** 2009 **Project Managers:** Edward Dudek, PE and Dan St. Germain, CPG	**Site Name:** Cornell-Dubilier Electronics Superfund Site Operable Unit 3 **Site Location:** South Plainfield, New Jersey

Date of Session: Weekly sessions starting April 2008.
Scoping Session Purpose: Plan the project.

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Edward Dudek, PE	Senior Associate	Malcolm Pirnie, Inc.	914-641-2686	Edudek@pirnie.com	Admin. Project Manager
Daniel St. Germain, CPG	Senior Associate	Malcolm Pirnie, Inc.	201-398-4381	Dst.germain@pirnie.com	Technical Project Manager
Janis Karn	Senior Project Environmental Scientist	Malcolm Pirnie, Inc.	201-398-4336	Jkarn@pirnie.com	Deputy Project Manager
James McCann	Senior Project Chemist	Malcolm Pirnie, Inc.	201-398-4310	Jmccann@pirnie.com	Project Quality Assurance Officer

Other members of the Malcolm Pirnie investigative team participated as necessary in the weekly project planning/status calls.

Comments/Decisions: The data quality objectives and data needs for the project were developed based on the information provided during communications with the USEPA and USACE-KCD..

Action Items: Janis Karn coordinated the preparation of the project documents. Allan Wood was assigned to prepare the draft FSP, while Jim McCann was assigned to prepare the draft QAPP. Mike Barone was assigned to draft the Work Plan and Tara Schmidt and Erika Zamek were assigned to prepare the SSHP.

Consensus Decisions: Per direction from the USEPA, where possible samples will be analyzed by the DESA laboratory and/or an assigned CLP laboratory.

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

Problem Definition

This is a Superfund Site and a RI/FS will be conducted in accordance with the Comprehensive Environmental Response, Compensation and Liability Act, as amended. This task is concerned with determining the nature and extent of the contamination in the groundwater of OU3. The soils at the former Cornell-Dubilier Electronics facility are known to contain polychlorinated biphenyls (PCBs), volatile organic compounds (VOCs), semi-volatile organic compounds pesticides (SVOCs), dioxins/furans, metals and cyanide and other chemical contamination which potentially could impact the groundwater in the area. Previous analyses of groundwater samples collected from existing groundwater monitoring wells at the Site have also revealed the presence of chemical contamination such as VOCs, SVOCS, PCBs, and dioxin/furans. The objectives for this study are the following:

- Characterize the nature and extent of hazardous constituents in the groundwater in support of the RI/FS and obtain sufficient data on the chemical contamination in the groundwater to evaluate the remedial alternatives.
- Determine the horizontal and vertical extent of contaminated groundwater within the study area.
- Determine the extent of contamination emanating from the former Cornell-Dubilier Electronics facility into the fractured bedrock.
- Determine the down-gradient receptors of the contaminated groundwater.
- Determine the presence of light and/or dense non-aqueous phase liquid (LNAPL and/or DNAPL).
- *Identify the level of human health risk associated with the chemical contamination in the groundwater.*

Note: For further discussion regarding the problem definition please see the DQOs, which are given in Attachment 1.1. Maps are available in the Field Sampling Plan (FSP).

QAPP Worksheet #11
(UFP-QAPP Manual Section 2.6.1)

Project Quality Objectives /Systematic Planning Process Statements

Who will use the data?
USEPA Region 2, the USACE-KC, NJDEP, and Malcolm Pirnie's remedial investigation team.

What will the data be used for?

- Characterize the nature and extent of hazardous constituents in the groundwater in support of the RI/FS and obtain sufficient data on the chemical contamination in the groundwater to evaluate the remedial alternatives.
- Determine the horizontal and vertical extent of contaminated groundwater within the fractured bedrock.
- Determine the extent of contamination emanating from the former CDE facility (OU2) into the fractured bedrock.
- Determine the down-gradient receptors of the contaminated groundwater.
- Determine the presence of either light or dense non-aqueous phase liquid (LNAPL or DNAPL) and if present the areal extent.
- Identify the level of human health risk associated with the chemical contamination in the groundwater.

What types of data are needed?

- Analytical data needs include analyses of groundwater for VOCs, SVOCs, PCB Aroclors, Pesticides, TAL metals and as well as other groundwater screening parameters such as nitrate, sulfate, chloride, ferrous iron, alkalinity and hardness.
- A subset of the groundwater samples collected will also be analyzed for PCB Congeners and Dioxin/Furans.
- In addition, samples from bedrock cores collected during the drilling of four of the nine proposed new wells will also be tested for selected chlorinated volatiles and PCB Aroclors. Miscellaneous physical properties (matrix porosity, bulk density, etc.) will also be performed on representative bedrock samples.

(See Attachment 1.2 for a table of the data needs)

How "good" do the data need to be in order to support the environmental decision? The data needs for this project should consider the requirements for risk assessment. The data must be technically defensible and of sufficient quality to support the project DQOs, which are described in Attachment 1. See Worksheet 15, Reference and Evaluation Table, which summarizes the analytical parameters and the associated project action levels and project quantitation limits.
How much data are needed? Samples will include groundwater and bedrock. An estimated 480 bedrock samples will be collected for volatiles plus subset for PCB Aroclors and other tests by off-site laboratory. Groundwater events 1 and 2 will require 140 samples for chemical parameters plus other miscellaneous testing. The number of samples for test parameter to be collected at each sampling location is described in Worksheet 18 of QAPP.
Where, when, and how should the data be collected/generated? Bedrock samples will be collected by rock core drilling and rock crushing in January through March 2009. The samples will be analyzed by a mobile laboratory for VOCs and a fixed-based laboratory for PCB Aroclors and geotechnical parameters. Groundwater samples will be collected by low flow methods and FLUTe multi-port sampling procedures in May, August, and November 2009 per the FSP. The samples collected will be submitted to the USEPA Region 2 DESA laboratory, USEPA CLP laboratories, and/or subcontract laboratories for analyses. (See FSP Section 4 and Worksheet #21)
Who will collect and generate the data? Malcolm Pirnie field personnel will collect the samples. The samples will be analyzed for chemical analytical parameters by USEPA Region 2 DESA laboratory, USEPA CLP laboratories, and subcontract laboratories.
How will the data be reported? The data will be reported by the USEPA-assigned lab, or the subcontract laboratory to Malcolm Pirnie, according to the requirements in Worksheet 29. Laboratory electronic data deliverables (EDD) will be obtained in a USEPA Region 2 compliment format (http://www.epa.gov/Region2/superfund/medd.htm).
How will the data be archived? Electronic data will be archived in the project database to be maintained by Malcolm Pirnie. Hard copies of laboratory reports will also be kept in the Malcolm Pirnie project files. Data will be transferred to the USEPA upon completion of the project. Retrieval of data by others will be at the discretion of the USACE and the USEPA. The length of time that records will be archived will be at the discretion of the USACE and USEPA.

QAPP Worksheet #12

Measurement Performance Criteria Table

Matrix	Groundwater
Analytical Group	TCL Dioxins/Furans
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP[1]	Data Quality Indicators (DQIs)	Measurement Performance Criteria [1]	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
See FSP in Appendix A	EPA-CLP SOW DLM02.0 or EPA Methods 1613B	Sensitivity and Accuracy	Less than CRQLs	Equipment Blank	S & A
		Precision	<RPD 35% for duplicate values greater than or equal to 5 times the CRQL	Field Duplicates	S & A
		Accuracy/Bias	Per DLM02.0 or EPA 1613B	Initial Calibration	A
		Accuracy/Bias	Per DLM02.0 or EPA 1613B	Calibration Verification	A
		Accuracy/Bias	Per recoveries given in DLM02.0 or EPA 1613B	Laboratory Control Sample	A
		Sensitivity	Per DLM02.0 or EPA 1613B requirements the native compounds in the MB must be less than or equal to the CRQL. Dioxin/Furan analytes in the MB must be less than 3 Xs the CRQL	Method Blanks	A
		Sensitivity	Per DLM02.0 or EPA 1613B	Method Detection Limits (MDLs)	A
		Completeness	>90% collection, >90% laboratory analysis	Data Completeness Check	S & A

1 The assigned laboratory must perform and meet all the measurement performance criteria which assess the analytical DQIs specified in EPA CLP SOW DLM02.0 or the applicable EPA method such as initial and ongoing studies to demonstrate precision and accuracy; calibration verification, internal standards and laboratory control samples for accuracy; and blanks and method detection limits for sensitivity.

QAPP Worksheet #12

Measurement Performance Criteria Table

Matrix	Groundwater
Analytical Group	PCB Congeners
Concentration Level	Low

Sampling Procedure	**Analytical Method/SOP[1]**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria [1]**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)**
See FSP in Appendix A	EPA-CLP SOW CBC01.0 or EPA Method 1668A	Sensitivity and Accuracy	Less than CRQLs	Equipment Blank	S & A
		Precision	<RPD 35% for duplicate values greater than or equal to 5 times the CRQL	Field Duplicates	S & A
		Accuracy/Bias	Per CBC01.1 or 1668A	Initial Calibration	A
		Accuracy/Bias	Per CBC01.1 or 1668A	Calibration Verification	A
		Accuracy/Bias	Per recoveries given in CBC01.1 or 1668A	Laboratory Control Sample	A
		Sensitivity	Per CBC01.1 requirements the native compounds in the MB must be less than or equal to the CRQL.	Method Blanks	A
		Sensitivity	Per CBC01.1 or 1668A	MDLs	A
		Completeness	>90% collection, >90% laboratory analysis	Data Completeness Check	S & A

1 The assigned laboratory must perform and meet all the measurement performance criteria which assess the analytical DQIs specified in EPA CLP SOW CBC01.1 or EPA 1668A including initial and ongoing studies to demonstrate precision and accuracy, and calibration verification, internal standards added to all extracts, cleanup standards and laboratory control samples for accuracy, and blanks and method detection limits for sensitivity.

QAPP Worksheet #12

Measurement Performance Criteria Table

Matrix	Groundwater
Analytical Group	TCL Volatiles
Concentration Level	Trace

Sampling Procedure	Analytical Method/SOP[1]	Data Quality Indicators (DQIs)	Measurement Performance Criteria [1]	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
See FSP in Appendix A	EPA-CLP SOW SOM01.2 or DESA method	Sensitivity and Accuracy	Less than CRQLs	Equipment Blank	S & A
		Precision	<RPD 30% for aqueous and 40% for soilds for duplicate values greater than or equal to 5 times the CRQL	Field Duplicates	S & A
		Accuracy/Bias /Precision	Per recovery and RPD% requirements in SOM01.2 Exhibit D, Table 6 for Criteria	MS/MSD	A
		Accuracy/Bias	Deuterated Monitoring Compound recoveries per requirements in SOM01.2 Exhibit D, Table 5 for Criteria	Deuterated Monitoring Compounds	A
		Sensitivity	Per requirements in SOM01.2	(MDLs	A
		Sensitivity	Less than CRQLs, Reference USEPA Region 2 SOP No. 34/Trace VOA Trace VOA - Blank Type Criteria Table	Method Blanks	A
		Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Check	S & A

1 The assigned laboratory must perform and meet all the measurement performance criteria which assess the analytical DQIs specified in EPA CLP SOW SOM01.2 such as laboratory duplicates and matrix spike duplicates for precision, matrix spikes, Deuterated Monitoring Compounds for accuracy, and blanks and method detection limits for sensitivity.

QAPP Worksheet #12

Measurement Performance Criteria Table

Matrix	Bedrock
Analytical Group	Chlorinated Volatiles
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
Stone Environmental Inc. SOPs SEI-6.37.0	Stone Environmental Inc. SOPs 10.18.0 and SEI-10.17.0 or equivalent	Accuracy	Less than QLs	Equipment Blank	S & A
		Accuracy	80-120%	Fortified Blank	A
		Precision	RPD 15%	Laboratory Duplicate	S & A
		Accuracy	Correlation coefficient of 0.995 or grater	Initial multiple point calibration	S & A
		Accuracy	Recovery ±20%	Initial Calibration Verification Standard	S & A
		Accuracy	Recovery 80-120%	Laboratory Control Standard	S & A
		Accuracy	70-130%	Matrix Spike	S & A
		Completeness	>90% collection, >90% laboratory analysis	Data completeness check	S & A

QAPP Worksheet #12

Measurement Performance Criteria Table

Matrix	Groundwater
Analytical Group	TCL SVOC
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP[1]	Data Quality Indicators (DQIs)	Measurement Performance Criteria [1]	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
See FSP in Appendix A	EPA-CLP SOW SOM01.2,or DESA method	Sensitivity and Accuracy	Less than CRQLs	Equipment Blank	S & A
		Precision	<RPD 30% for duplicate values greater than or equal to 5 times the CRQL	Field Duplicates	S & A
		Accuracy/Bias /Precision	Per recovery and RPD% requirements in SOM01.2 Exhibit D, Table 6 for Criteria	MS/MSD	A
		Accuracy/Bias	Deuterated Monitoring Compound recoveries per requirements in SOM01.2 Exhibit D, Table 5 for Criteria	Deuterated Monitoring Compounds	A
		Sensitivity	Per requirements in SOM01.2	(MDLs)	A
		Sensitivity	Less than CRQLs or per SOM01.2 Reference USEPA Region 2 SOP No. 33/Low/Medium VOA - Blank Type Criteria Table	Method Blanks	
		Completeness	>90% soil collection, >90% laboratory analysis	Data Completeness Check	S & A

1 The assigned laboratory must perform and meet all the measurement performance criteria which assess the analytical DQIs specified in EPA CLP SOW SOM01.2 such as laboratory duplicates and matrix spike duplicates for precision, matrix spikes, Deuterated Monitoring Compounds for accuracy, and blanks and method detection limits for sensitivity.

QAPP Worksheet #12

Measurement Performance Criteria Table

Matrix	Groundwater
Analytical Group	Pesticides
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP[1]	Data Quality Indicators (DQIs)	Measurement Performance Criteria [1]	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
See FSP in Appendix A	EPA-CLP SOW SOM01.2 or DESA method	Sensitivity and Accuracy	Less than CRQLs	Equipment Blank	S & A
		Precision	<RPD 30% for duplicate values greater than or equal to 5 times the CRQL	Field Duplicates	S & A
		Accuracy/Bias /Precision	Per recovery and RPD% requirements in SOM01.2, Exhibit D, Table 3 for Criteria	MS/MSD	A
		Accuracy/Bias	Per recoveries per requirements in SOM0.1.2 Exhibit D, Table 2 for Criteria	LCS	A
		Sensitivity	Per requirements in SOM01.2	Method Detection Limits (MDLs)	A
		Sensitivity	No analyte > CRQLs	Method Blanks	
		Completeness	>90% collection, >90% laboratory analysis	Data Completeness Check	S & A

1 The assigned laboratory must perform and meet all the measurement performance criteria which assess the analytical DQIs specified in EPA CLP SOW SOM01.2 such as laboratory duplicates and matrix spike duplicates for precision, matrix spikes, Laboratory Control Samples for accuracy, and blanks and method detection limits for sensitivity.

QAPP Worksheet #12

Measurement Performance Criteria Table

Matrix	Groundwater
Analytical Group	PCB Aroclors
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP[1]	Data Quality Indicators (DQIs)	Measurement Performance Criteria [1]	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
See FSP in Appendix A	EPA-CLP SOW SOM01.2 or DESA method	Sensitivity and Accuracy	No analyte > CRQLs	Equipment Blank	S & A
		Precision	<RPD 30% for duplicate values greater than or equal to 5 times the CRQL	Field Duplicates	S & A
		Accuracy/Bias/ Precision	Per recovery and RPD% requirements in SOM01.2, Exhibit D, Table 1 for Criteria	MS/MSD	A
		Accuracy/Bias	Per recoveries per requirements in SOM0.1.2 Exhibit D, Table 2 for Criteria	LCS	A
		Sensitivity	Per requirements in SOM01.2	(MDLs	A
		Sensitivity	No analyte > CRQLs Reference USEPA Region 2 SOP No. 37/Low/Medium Aroclor - Blank Type Criteria Table	Method Blanks	
		Completeness	>90% collection, >90% laboratory analysis	Data Completeness Check	S & A

1 The assigned laboratory must perform and meet all the measurement performance criteria which assess the analytical DQIs specified in EPA CLP SOW SOM01.2 such as laboratory duplicates and matrix spike duplicates for precision, matrix spikes, Laboratory Control Samples for accuracy, and blanks and method detection limits for sensitivity.

QAPP Worksheet #12

Measurement Performance Criteria Table

Matrix	Solids (Bedrock)
Analytical Group	PCB Aroclors
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP[1]	Data Quality Indicators (DQIs)	Measurement Performance Criteria [1]	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A)
See FSP in Appendix A	EPA-CLP SOW SOM01.2 or SW-846-8082 or DESA method	Sensitivity and Accuracy	No analyte > CRQLs	Equipment Blank	S & A
		Precision	<RPD 40% for duplicate values greater than or equal to 5 times the CRQL	Field Duplicates	S & A
		Accuracy/Bias/ Precision	Per recovery and RPD% requirements in SOM01.2 Exhibit D, Table 1 for Criteria	MS/MSD	A
		Accuracy/Bias	Per recoveries per requirements in SOM0.1.2 Exhibit D, Table 2 for Criteria	LCS	A
		Sensitivity	Per requirements in SOM01.2	Method Detection Limits (MDLs)	A
		Sensitivity	No analyte > CRQLs Reference USEPA Region 2 SOP No. 37/Low/Medium Aroclor - Blank Type Criteria Table	Method Blanks	
		Completeness	>90% collection, >90% laboratory analysis	Data Completeness Check	S & A

1 The assigned laboratory must perform and meet all the measurement performance criteria which assess the analytical DQIs specified in EPA CLP SOW SOM01.2 or SW-846-8082 such as laboratory duplicates and matrix spike duplicates for precision, matrix spikes, Laboratory Control Samples for accuracy, and blanks and method detection limits for sensitivity. If SW-846-8082 is used instead of SOM01.2, the QA criteria should be DoD compliment.

QAPP Worksheet #12

Measurement Performance Criteria Table

Matrix	Groundwater
Analytical Group	TAL Metals
Concentration Level	ICP-AES (ug/L)

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)
See FSP in Appendix A	ILM05.4	Precision (field)	≤20% RPD*	Field Duplicate	S & A
		Accuracy (field)	No analyte > CRQL*	Equipment Blank	S & A
		Precision (laboratory)	≤20% RPD*	Duplicate Sample **	A
		Accuracy (laboratory)	75–125%; 80–120 %	*** Matrix Spike; LCSW****	A A
		Completeness	>90% collection, >90% laboratory analysis	Data Completeness Check	S & A

[1]Reference number from QAPP Worksheet #21.
[2]Reference number from QAPP Worksheet #23.
*Reference USEPA Region 2 SOP No. HW-2, Revision 13/Evaluation of Metals Data for CLP - (include absolute difference criteria)
**Reference USEPA CLP ILM05.4, Exhibit D of ICP-AES for Duplicate Sample Criteria - (include absolute difference criteria)
***Reference USEPA CLP ILM05.4, Exhibit D of ICP-AES for Spike Sample Criteria
****Reference USEPA CLP ILM05.4, Exhibit D of ICP-AES for aqueous Laboratory Control Sample (LCSW) Criteria w/exception of Ag and Sb

QAPP Worksheet #12

Measurement Performance Criteria Table

Matrix	Groundwater
Analytical Group	TAL Metals
Concentration Level	ICP-MS (ug/L)

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)
See FSP in Appendix A	ILM05.4	Precision (field)	≤20% RPD*	Field Duplicate	S & A
		Accuracy (field)	No analyte > CRQL*	Equipment Blank	S & A
		Precision (laboratory)	≤20% RPD*	Duplicate Sample **	A
		Accuracy (laboratory)	75–125%; 80–120 %	*** Matrix Spike; LCSW****	A A
		Completeness	>90% collection, >90% laboratory analysis	Data Completeness Check	S & A

[1]Reference number from QAPP Worksheet #21.
[2]Reference number from QAPP Worksheet #23.
*Reference USEPA Region 2 SOP No. HW-2, Revision 13/Evaluation of Metals Data for CLP - (include absolute difference criteria)
**Reference USEPA CLP ILM05.4, Exhibit D of ICP-MS for Duplicate Sample Criteria - - (include absolute difference criteria)
***Reference USEPA CLP ILM05.4, Exhibit D of ICP-MS for Spike Sample Criteria
****Reference USEPA CLP ILM05.4, Exhibit D of ICP-MS for LCSW Criteria

QAPP Worksheet #12

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

Matrix	Groundwater
Analytical Group	TAL –Total Mercury
Concentration Level	Cold Vapor Atomic Absorption (CVAA)

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)
See FSP in Appendix A	ILM05.4	Precision (field)	≤20% RPD*	Field Duplicate	S & A
		Accuracy (field)	No analyte > CRQL*	Equipment Blank	S & A
		Precision (laboratory)	≤20% RPD*	Duplicate Sample **	A
		Accuracy (laboratory)	75–125%;	*** Matrix Spike;	A
		Completeness	>90% collection, >90% laboratory analysis	Data Completeness Check	S & A

[1]Reference number from QAPP Worksheet #21.
[2]Reference number from QAPP Worksheet #23.
*Reference USEPA Region 2 SOP No. HW-2, Revision 13/Evaluation of Metals Data for CLP - (include absolute difference criteria)
**Reference USEPA CLP ILM05.4, Exhibit D of ICP-MS for Duplicate Sample Criteria - - (include absolute difference criteria)
***Reference USEPA CLP ILM05.4, Exhibit D of ICP-MS for Spike Sample Criteria

QAPP Worksheet #12

Measurement Performance Criteria Table

Matrix	Groundwater
Analytical Group	TAL –Total Cyanide
Concentration Level	Colorimeter or Spectrophotometer

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)
See FSP in Appendix A	ILM05.4	Precision (field)	≤20% RPD*	Field Duplicate	S & A
		Accuracy (field)	No analyte > CRQL*	Field Blank	S & A
		Precision (laboratory)	≤20% RPD*	Duplicate Sample **	A
		Accuracy (laboratory)	75–125%;	*** Matrix Spike	A
		Completeness	>90% collection, >90% laboratory analysis	Data Completeness Check	S & A

[1]Reference number from QAPP Worksheet #21.
[2]Reference number from QAPP Worksheet #23.
*Reference USEPA Region 2 SOP No. HW-2, Revision 13/Evaluation of Metals Data for CLP- (include absolute difference criteria)
**Reference USEPA CLP ILM05.4, Exhibit D of ICP-MS for Duplicate Sample Criteria - (include absolute difference criteria)
***Reference USEPA CLP ILM05.4, Exhibit D of ICP-MS for Spike Sample Criteria

QAPP Worksheet #12

Measurement Performance Criteria Table

Matrix	Groundwater
Analytical Group[1]	Total Organic Carbon and Dissolved Organic Carbon
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP[1]	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)
FSP in Appendix A	SW 846 9060 or DESA method	Precision	RPD ≤ 50% for duplicate values greater than or equal to 5 times the QL	Field Duplicates	S & A
		Precision	RPD ≤ 30%	Laboratory Duplicates	A
		Precision and Accuracy	Per Lab SOP	MS and MSD	A
		Sensitivity	Sufficient to support RLs	MDLs	A
		Sensitivity	Less than RLs	Method Blanks	A
		Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Check	S & A

1. If DESA performs the analyses they will follow the requirements in the DESA methods.

QAPP Worksheet #12

Measurement Performance Criteria Table

Matrix	Groundwater
Analytical Group[1]	Anions including Sulfate, Chloride, and Nitrate
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP[1]	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)
See FSP in Appendix A	SW-846 9056, EPA 300.0 or DESA method (Nitrate can be measured by alternate methods such as EPA 353.2, in which case the methods QC requirements must be followed.)	Precision	RPD $\leq$ 30% for duplicate values greater than or equal to 5 times the RL	Field Duplicates	S & A
		Precision	Per DoD Quality Systems Manual (QSM) Table B-10 or lab limits	Laboratory Duplicates	A
		Accuracy	Per DoD QSM Table B-10 or lab limits	Calibration and Calibration Verification	
		Precision/Accuracy	Per DoD QSM Table B-10 or lab limits	MS/MSD	A
		Accuracy	Per DoD QSM Table B-10 or lab limits	Laboratory Control Standard	A
		Sensitivity	Sufficient to support project QLs	MDLs	A
		Sensitivity	Less than RLs	Method Blanks	A
		Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Check	S & A

1. If DESA performs the analyses they will follow the requirements in the DESA methods.

QAPP Worksheet #12

Measurement Performance Criteria Table

Matrix	Groundwater
Analytical Group[1]	Alkalinity
Concentration Level	NA

Sampling Procedure[2]	Analytical Method/SOP[1]	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)
FSP in Appendix A	SM2320B or DESA method or similar methods such as EPA Method 310.1 or 310.2	Precision	RPD ≤ 30% for duplicate values greater than or equal to 5 times the QL	Field Duplicates	S & A
		Precision	RPD ≤ 20%	Laboratory Duplicates	
		Accuracy	Per Lab SOP	Check Standard	A
		Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Check	S & A

1. If DESA performs the analyses they will follow the requirements in the DESA methods.

QAPP Worksheet #12

Measurement Performance Criteria Table

Matrix	Groundwater
Analytical Group[1]	Ferrous Iron
Concentration Level	Low

Sampling Procedure[2]	Analytical Method/SOP[1]	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)
See FSP in Appendix A	SM 3500 Fe D	Sensitivity and Accuracy	Blank corrected to 0.00 mg/L Fe^{2+}	Blank	S & A
		Accuracy	Per SM3500 Fe D	Accuracy Check Standard	A
		Precision	RPD $\leq$ 50% for duplicate values greater than or equal to 5 times the QL	Field Duplicates	S & A

1. If DESA performs the analyses they will follow the requirements in the DESA methods.

QAPP Worksheet #12 (UFP-QAPP Manual Section 2.6.2)

Measurement Performance Criteria Table

Matrix	Groundwater
Analytical Group[1]	Hardness
Concentration Level	NA

Sampling Procedure[2]	Analytical Method/SOP[1]	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)
See FSP in Appendix A	SM 2340C	Precision	RPD ≤ 30% for duplicate values greater than or equal to 5 times the QL	Field Duplicates	S & A
		Precision	RPD ≤ 20%	Laboratory Duplicates or MSD	A
		Accuracy	Per Lab SOP, recovery 75-125%,	MS	A
		Sensitivity	Sufficient to support RLs	MDLs	A
		Sensitivity	Less than RLs	Method Blank	A
		Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Check	S & A

1. If DESA performs the analyses they will follow the requirements in the DESA methods.

QAPP Worksheet #12 (UFP-QAPP Manual Section 2.6.2)

Measurement Performance Criteria Table

Matrix	Groundwater
Analytical Group	Methane, Ethane and Ethene
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP[1]	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)
See FSP in Appendix A	SW 846 8015B or method equivalent to EPA RSK175	Accuracy	Regression coefficient (r2) greater than 0.995	Initial Calibration (IC)	S & A
		Accuracy	Difference from calculated value ≤20%	Continuous Calibration Check (CCC)	A
		Sensitivity	Less than RLs	Method Blank	A
		Precision	RPD ≤30 % for duplicate values greater than or equal to 5 times the RL	Field Duplicates	S & A
		Accuracy	Recovery 30-150%; RPD ≤ 20%	Matrix Spike/Matrix Spike Duplicates	A
		Precision	RPD ≤20%	Lab Duplicates	A
		Sensitivity	Sufficient to support RLs	Method Detection Limits	A
		Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Check	S & A

1. If DESA performs the analyses they will follow the requirements in the DESA methods.

QAPP Worksheet 12

Measurement Performance Criteria Table

Matrix	Bedrock
Analytical Group	Total Organic Carbon
Concentration Level	Low

Sampling Procedure	Analytical Method/SOP[1]	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)
FSP in Appendix A	EPA Region 2 Lloyd Kahn or ASTM 2794 or equivalent	Precision	RPD ≤ 50% for duplicate values greater than or equal to 5 times the QL	Field Duplicates	S & A
		Precision	RPD ≤ 30%	Laboratory Duplicates	A
		Precision and Accuracy	Per Lab SOP	MS and MSD	A
		Sensitivity	Sufficient to support RLs	MDLs	A
		Sensitivity	Less than RLs	Method Blanks	A
		Completeness	>90% sample collection, >90% laboratory analysis	Data Completeness Check	S & A

Precision, Accuracy (or Bias), Representativeness, Completeness, and Comparability

To measure and control the quality of analyses, certain QA parameters are defined and utilized in data analysis activities. These parameters are defined below. The QA/QC required for the parameters to be analyzed under the USEPA CLP is contained in the USEPA CLP SOW. If non-CLP labs perform these analyses, they would also be required to meet these QA/QC criteria. Detailed information on CLP methods and QA/QC criteria can be found in the USEPA CLP SOW, on the USEPA CLP website at http://www.epa.gov/superfund/programs/clp/.

Precision

Precision measures the reproducibility of data or measurements under specific conditions. Precision is a quantitative measure of the variability of a group of data compared to their average value. Duplicate precision is stated in terms of relative percent difference (RPD) or absolute difference between two measurements. Measurement of precision is dependent upon sampling technique and analytical method. Field duplicate and laboratory duplicate samples will be used to measure precision for project samples. Both sampling and analysis will be as consistent as possible. For a pair of measurements, RPD (or absolute difference) will be calculated to assess precision, as presented below:

$$RPD(\%) = \frac{|D_1 - D_2|}{\left[\frac{(D_1 + D_2)}{2}\right]} \times 100$$

where: D_1 and D_2 = the two replicate values.

RPD will meet USEPA CLP SOW requirements, where applicable, or the QA requirements listed in the applicable laboratory standard operating procedures.

Accuracy/Bias

Accuracy measures the bias in a measurement system. Sources of error include the sampling process, field contamination, preservation, handling, shipping, sample matrix, sample preparation, and analysis technique. Analytical accuracy will be assessed through surrogate spike, matrix spike, laboratory control and/or quality check samples, where applicable. In general, accuracy is measured in terms of percent recovery (%R):

$$\%R = \frac{(SSR - SR)}{SA} \times 100$$

where: SSR = spike sample result
SR = sample result
SA = spike added to spiking matrix

Refer to the CLP SOW for the laboratory analytical method accuracy requirements.

Representativeness

Representativeness expresses the degree to which data accurately and precisely reflect a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. Representativeness is a qualitative parameter that is dependent upon the proper design and implementation of the sampling program and proper laboratory protocol. The sampling design created for this project was designed to provide data representative of site conditions. During the development of the sampling design, consideration was given to the past history of contamination in the study area, existing analytical data, physical setting, and processes. Representativeness will be satisfied by determining that the FSP is followed; proper sampling techniques, preservation, and handling are used; proper analytical procedures are followed; and holding times for the samples are not exceeded in the laboratory.

Completeness

Completeness is a measure of the amount of usable data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions. It is expected that the laboratories used for this project will provide data that meet the QC acceptance criteria for 90 percent, or more, of all samples analyzed. Following the completion of the analytical testing, the percent completeness will be calculated by the following equation:

$$\text{COMPLETENESS } (\%) = \frac{\text{number of usable data}}{\text{number of samples collected for each parameter analyzed}} \times 100$$

The data validation process will be used to determine the quality and quantity of usable analytical data generated.

The completeness acceptance criterion for samples collected in the field will be 90 percent of the quantity of samples planned for collection as described in the FSP. Corrective action may be implemented to re-collect samples where necessary and possible (*e.g.*, modifying a planned sample location, sample jars broken during shipment). Laboratory notification sample receipt and conditions will be used to determine, as soon as possible, whether any problems during sample shipment would necessitate recollection of samples.

Comparability

Comparability expresses the confidence with which one data set can be compared to another. The extent to which existing and planned analytical data will be comparable depends on the similarity of sampling and analytical methods. The procedures used to obtain the planned analytical data are expected to provide comparable data to existing datasets for the Site. The procedures proposed for both soil and settled dust sample collection are similar to those previously conducted by the USEPA. The procedures used will be USEPA-promulgated methodologies or ASTM Standard Test Methods, which are well recognized and commonly used for environmental and geotechnical investigations.

Desired Method Sensitivity

Depending upon the use of the data (see Attachment 1.2 for data needs and data use) and the type of test parameter, specific Quantitation Limits (QLs) will be required. Worksheet 15 lists the required QLs or USEPA CLP contract required QLs (CRQLs), as specified for the definitive chemical parameters required for this project.

QAPP Worksheet #13
(UFP-QAPP Manual Section 2.7)

Secondary Data Criteria and Limitations Table

Secondary Data [1]	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
Historical Data	See Section 3.1.7 of the Work Plan for information on 2000 study	Foster Wheeler	To determine the potential nature extent of contamination within the study areas.	The historical data is considered to be valid data and has been accepted by the USEPA.
Historical Data	See FSP Section 1.2.2 in Appendix A for information on 2008 study	USEPA	To determine the potential nature extent of contamination within the study areas.	The historical data is considered to be valid data and has been accepted by the USEPA.

1. Existing data is discussed in detail in the WP and FSP.

QAPP Worksheet #14
(UFP-QAPP Manual Section 2.8.1)

Summary of Project Tasks

Sampling Tasks: Monitor groundwater wells throughout OU-3 for chemicals such as VOCs, SVOCs, PCBs, Pesticides, Dioxins/Furans, TAL metals, TOC, DOC and for other groundwater parameters as described in the FSP and Worksheets 17 and 18. Collect bedrock samples for analyses for chlorinated volatiles and PCB Aroclors and geological parameters as described in the FSP and Worksheets 17 and 18. The data needs associated with these tasks are summarized in Attachment 1.2.
Analysis Tasks: Analyze groundwater and bedrock samples by the testing methodologies are described in Worksheets 19 and 23.
Quality Control Tasks: The analytical and testing laboratories will be required to analyze QC samples listed in the USEPA CLP SOWs , and USEPA method and the other documents and procedures given in Worksheet 28.
Secondary Data: Historical data available in prior studies will be used for remediation design. See Worksheet 13.
Data Management Tasks: All data, field notes, and analytical information will be placed in an electronic database which will be maintained in the Malcolm Pirnie, Inc. corporate office. If at the time of sampling the assigned CLP or DESA lab are prepared to provide data in the Region 2 Electronic Data Deliverable (EDD) format, they will be required to do so. All electronic data will be backed up. Hardcopies of data will also be stored in project files. See Worksheet 29 for discussion of data management.
Documentation and Records: All hardcopy data (field notebooks, photos, hardcopies of Chain of Custody forms, Airbills, etc.) will be taken to the Malcolm Pirnie, Inc. corporate office and stored in the project files.
Assessment/Audit Tasks: SOPs will be reviewed prior to the performance of tasks. Technical System Audits will be performed per SOP No. 6 in Attachment 6 (see Worksheet 31.)
Data Review Tasks: Verification of sampling and laboratory data will be conducted. Laboratory data produced by USEPA CLP labs will be validated by the USEPA. Any non-CLP chemical data that is generated will be validated by Malcolm Pirnie data validators or subcontract data validators against the criteria in the applicable analytical SOPs (see Worksheets 23, 28, 35 and 36) .

QAPP Worksheet #15 (UFP-QAPP Manual Section 2.8.1)

Reference Limits and Evaluation Table

Matrix: Aqueous (Groundwater)

Analytical Group: PCB Congeners by USEPA SOW CBC01.1 or Method 1668A

Concentration Level: Low

Analyte	Chemical Abstracts Services (CAS) Number	Risk-Based Screening Level [2] (pg/L)	Potential ARARs Screening Levels[3] (pg/L)	Project Quantitation Limit (pg/L)	Analytical Method		Achievable Laboratory Limits Based on USEPA CLP Contract Required Reporting Limits (CRQLs) (Note 1)	
					MDLs	QLs	MDLs (pg/L)	QLs (pg/L)
All individual Congeners PCB-1 through PCB-209	See CLP SOW CBC01.0 and USEPA 1668A	6,800 – 680,000 (ca)	500,000	Equal to the lab achievable QLs for each congener	See CLP SOW CBC01.0 and USEPA 1668A	See CLP SOW CBC01.0 and USEPA 1668A	NA	10 to 1000 pg/L

1 Achievable QLs are expected to range from 10 to 1000 pg/L and are published in CBC01.1, with exceptions (particularly for co-eluting congeners). The lab sample specific detection limits may be different.

2 Refer Table 3-1 in the work plan.
Risk-Based Screening Levels are the USEPA Region 3 Regional Screening Levels for tapwater (12SEP2008) accessed online at: www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm. For non-carcinogens (nc), the range represents screening levels for target hazard quotients of 0.1 and 1. For carcinogens (ca), the range represents screening levels for target cancer risks of 10^{-6} and 10^{-4}.

3 Refer to Table 3-1 in the work Plan. Potential ARARs, selected as the lowest of the Federal MCLs (40 CFR 141), State MCLs (N.J.A.C. 7:10-5.2 & 7.2), and the NJ Groundwater Quality Criteria (N.J.A.C. 7:9-6).

4 Bold – indicates Preliminary COPCs, refer to Table 3-1 in the work plan.

QAPP Worksheet #15 (UFP-QAPP Manual Section 2.8.1)

Reference Limits and Evaluation Table

Matrix: Aqueous Groundwater

Analytical Group: TCL Polychlorinated Dibenzodioxins and Dibenzofurans (PCDDs/PCFFs) by USEPA CLP DLM02.0 or Method 1613B

Concentration Level: Low

Analyte	CAS Number	Risk-Based Screening Level[5] (pg/L)	Potential ARARs Screening Levels[6] (pg/L)	Project Quantitation Limit[1] (pg/L)	Analytical Method (DLM02.0 or 1616B)		Achievable Laboratory Limits Quantitation Limits (QLs)	
					MDLs (pg/L)	QLs (pg/L)[3]	MDLs (pg/L)	QLs (pg/L)[4]
2,3,7,8-TCDD	1746-01-6			10	Note 2	10	NA	10
1,2,3,7,8-PeCDD	40321-76-4			50	Note 2	50	NA	50
1,2,3,6,7,8-HxCDD	57653-85-7			50	Note 2	50	NA	50
1,2,3,4,7,8-HxCDD	39227-28-6			50	Note 2	50	NA	50
1,2,3,7,8,9-HxCDD	19408-74-3			50	Note 2	50	NA	50
1,2,3,4,6,7,8-HpCDD	35822-46-9			50	Note 2	50	NA	50
OCDD	3268-87-9			100	Note 2	100	NA	100
2,3,7,8-TCDF	51207-31-9			10	Note 2	10	NA	10
1,2,3,7,8-PeCDF	57117-41-6			50	Note 2	50	NA	50
2,3,4,7,8-PeCDF	57117-31-4			50	Note 2	50	NA	50
1,2,3,6,7,8-HxCDF	57117-44-9			50	Note 2	50	NA	50
1,2,3,7,8,9-HxCDF	72918-21-9			50	Note 2	50	NA	50
1,2,3,4,7,8-HxCDF	70648-26-9			50	Note 2	50	NA	50
2,3,4,6,7,8-HxCDF	60851-34-5			50	Note 2	50	NA	50
1,2,3,4,6,7,8-HpCDF	67562-39-4			50	Note 2	50	NA	50
1,2,3,4,7,8,9-HpCDF	55673-89-7			50	Note 2	50	NA	50
OCDF	39001-02-0			100	Note 2	100	NA	100
Total 2,3,7,8-TCDD TEQ		0.52 – 52 (ca)	10					

1. The PQLs are based upon the CLP CRQLs. Specific MDLs for aqueous samples are not given in USEPA DLM02.0 or Method 1613B, but the QLs listed are the minimum levels published in table 2 of USEPA 1613B and the CRQLs listed in DLM02.0. The lab sample specific

detection limits may be different.

2. The actual detection limits are usually dependent on the level of interference rather than instrument limitations.
3. The assigned laboratory must have determined MDLs annually meeting the requirements in USEPA CLP SOW DLM01.2.
4. The achievable Laboratory Limits listed as based upon USEPA CLP CRQLs for trace water samples. The actual achievable QLs based upon the assigned laboratory MDL studies should be equivalent or lower.
5. Refer Table 3-1 in the work plan.
   Risk-Based Screening Levels are the USEPA Region 3 Regional Screening Levels for tapwater (12SEP2008) accessed online at: www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm. For non-carcinogens (nc), the range represents screening levels for target hazard quotients of 0.1 and 1. For carcinogens (ca), the range represents screening levels for target cancer risks of 10^{-6} and 10^{-4}.
6. Refer to Table 3-1 in the work Plan. Potential ARARs, selected as the lowest of the Federal MCLs (40 CFR 141), State MCLs (N.J.A.C. 7:10-5.2 & 7.2), and the NJ Groundwater Quality Criteria (N.J.A.C. 7:9-6).
7. Bold – indicates Preliminary COPCs, refer to Table 3-1 in the work plan.

QAPP Worksheet #15
(UFP-QAPP Manual Section 2.8.1)

Reference Limits and Evaluation Table

Matrix: Aqueous (Groundwater)

Analytical Group: TCL Volatiles (SOM01.2 Trace Water)

Concentration Level: Low

Analyte	CAS Number	Risk-Based Screening Level [5] (µg/L)	Potential ARARs Screening Levels[6] (µg/L)	Project Quantitation Limit [1] (ug/L)	Analytical Method		Achievable Laboratory Limits Based upon CLP CRQLs	
					MDLs	Method QLs(ug/L)	MDLs (ug/L)	QLs (ug/L) [4]
Dichlorodifluoromethane	75-71-8	39 - 390	1,000	0.50	Note 2	0.50	Note 4	0.50
Chloromethane	74-87-3	1.8 - 180 (ca)	N/A	0.50	Note 2	0.50	Note 4	0.50
Vinyl Chloride	75-01-4	0.016 - 16 (ca)	1	0.50	Note 2	0.50	Note 4	0.50
Bromomethane	74-83-9	0.87 – 8.7	10	0.50	Note 2	0.50	Note 4	0.50
Chloroethane	75-00-3	2,100 – 21,000	5	0.50	Note 2	0.50	Note 4	0.50
Trichlorofluoromethane	75-69-4	130 – 1,300	2,000	0.50	Note 2	0.50	Note 4	0.50
1,1-Dichloroethene	75-35-4	34 – 340	1	0.50	Note 2	0.50	Note 4	0.50
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	5,900 – 59,000	N/A	0.50	Note 2	0.50	Note 4	0.50
Acetone	67-64-1	2,200 – 22,000	6,000	5.0	Note 2	5.0	Note 4	5.0
Carbon Disulfide	75-15-0	100 – 1,000	700	0.50	Note 2	0.50	Note 4	0.50
Methyl Acetate	79-20-9	3,700 – 37,000	7,000	0.50	Note 2	0.50	Note 4	0.50
Methylene Chloride	75-09-2	4.8 - 480 (ca)	3	0.50	Note 2	0.50	Note 4	0.50
trans-1,2-Dichloroethene	156-60-5	11 - 110	100	0.50	Note 2	0.50	Note 4	0.50
Methyl tert-Butyl Ether	1634-04-4	12 – 1,200 (ca)	70	0.50	Note 2	0.50	Note 4	0.50
1,1-Dichloroethane	75-34-3	2.4 - 240 (ca)	50	0.50	Note 2	0.50	Note 4	0.50
cis-1,2-Dichloroethene	156-59-2	□□ - 370	70	0.50	Note 2	0.50	Note 4	0.50
2-Butanone	78-93-3	710 – 7,100	300	5.0	Note 2	5.0	Note 3	5.0
Bromochloromethane	74-97-5	N/A	N/A	0.50	Note 2	0.50	Note 3	0.50
Chloroform	67-66-3	0.19 - 19 (ca)	70	0.50	Note 2	0.50	Note 3	0.50

Reference Limits and Evaluation Table

Matrix: Aqueous (Groundwater)

Analytical Group: TCL Volatiles (SOM01.2 Trace Water)

Concentration Level: Low

Analyte	CAS Number	Risk-Based Screening Level [5] (µg/L)	Potential ARARs Screening Levels[6] (µg/L)	Project Quantitation Limit [1] (ug/L)	Analytical Method		Achievable Laboratory Limits Based upon CLP CRQLs	
					MDLs	Method QLs(ug/L)	MDLs (ug/L)	QLs (ug/L) [4]
1,1,1-Trichloroethane	71-55-6	910 – 9,100	30	0.50	Note 2	0.50	Note 3	0.50
Cyclohexane	110-82-7	1,300 – 13,000	N/A	0.50	Note 2	0.50	Note 3	0.50
Carbon Tetrachloride	56-23-5	0.2 – 20 (ca)	1	0.50	Note 2	0.50	Note 3	0.50
Benzene	71-43-2	0.41 - 41 (ca)	1	0.50	Note 2	0.50	Note 3	0.50
1,2-Dichloroethane	107-06-2	0.15 - 15 (ca)	2	0.50	Note 2	0.50	Note 3	0.50
Trichloroethene	79-01-6	1.7 - 170 (ca)	1	0.50	Note 2	0.50	Note 3	0.50
Methylcyclohexane	108-87-2	630 – 6,300	N/A	0.50	Note 2	0.50	Note 3	0.50
1,2-Dichloropropane	78-87-5	0.39 - 39 (ca)	1	0.50	Note 2	0.50	Note 3	0.50
Bromodichloromethane	75-27-4	1.1 - 110 (ca)	1	0.50	Note 2	0.50	Note 3	0.50
cis-1,3-Dichloropropene	10061-01-5	N/A	N/A	0.50	Note 2	0.50	Note 3	0.50
4-Methyl-2-pentanone	108-10-1	200 – 2,000	400	5.0	Note 2	5.0	Note 3	5.0
Toluene	108-88-3	230 – 2,300	600	0.50	Note 2	0.50	Note 3	0.50
trans-1,3-Dichloropropene	10061-02-6	N/A	N/A	0.50	Note 2	0.50	Note 3	0.50
1,1,2-Trichloroethane	79-00-5	0.24 - 24 (ca)	3	0.50	Note 2	0.50	Note 3	0.50
Tetrachloroethene	127-18-4	0.11 - 11 (ca)	1	0.50	Note 2	0.50	Note 3	0.50
2-Hexanone	591-78-6	N/A	300	5.0	Note 2	5.0	Note 3	5.0
Dibromochloromethane	124-48-1	0.8 - 80 (ca)	1	0.50	Note 2	0.50	Note 3	0.50
1,2-Dibromoethane	106-93-4	0.0065 – 0.65 (ca)	0.03	0.50	Note 2	0.50	Note 3	0.05 (SIM)
Chlorobenzene	108-90-7	9.1 - 91	50	0.50	Note 2	0.50	Note 3	0.50
Ethylbenzene	100-41-4	1.5 - 150 (ca)	700	0.50	Note 2	0.50	Note 3	0.50
o-Xylene	95-47-6	20 (for total xylene)	1,000 (for total xylene)	0.50	Note 2	0.50	Note 3	0.50
m,p-Xylenes	N/A			0.50	Note 2	0.50	Note 3	0.50
Styrene	100-42-5	160 – 1,600	100	0.50	Note 2	0.50	Note 3	0.50
Bromoform	75-25-2	8.5 - 850 (ca)	4	0.50	Note 2	0.50	Note 3	0.50
Isopropylbenzene	98-82-8	68 - 680	700	0.50	Note 2	0.50	Note 3	0.50

Reference Limits and Evaluation Table

Matrix: Aqueous (Groundwater)

Analytical Group: TCL Volatiles (SOM01.2 Trace Water)

Concentration Level: Low

Analyte	CAS Number	Risk-Based Screening Level [5] (µg/L)	Potential ARARs Screening Levels[6] (µg/L)	Project Quantitation Limit [1] (ug/L)	Analytical Method		Achievable Laboratory Limits Based upon CLP CRQLs	
					MDLs	Method QLs(ug/L)	MDLs (ug/L)	QLs (ug/L) [4]
1,1,2,2-Tetrachloroethane	79-34-5	0.067 – 6.7 (ca)	1	0.50	Note 2	0.50	Note 3	0.50
1,3-Dichlorobenzene	541-73-1	N/A	600	0.50	Note 2	0.50	Note 3	0.50
1,4-Dichlorobenzene	106-46-7	0.43 – 43 (ca)	75	0.50	Note 2	0.50	Note 3	0.50
1,2-Dichlorobenzene	95-50-1	37 - 370	600		Note 2		Note 3	
1,2-Dibromo-3-chloropropane	96-12-8	0.00032 – 0.032 (ca)	0.02	0.50	Note 2	0.50	Note 3	0.05 (SIM)
1,2,4-Trichlorobenzene	120-82-1	0.82 - 82 (ca)	9	0.50	Note 2	0.50	Note 3	0.50
1,2,3-Trichlorobenzene	87-61-6	N/A	N/A	0.50	Note 2	0.50	Note 3	0.50

1. The project quantitation limits are based upon USEPA CLP contract required Quantitation limits (CRQLs) given in CLP SOW SOM01.2.
2. The method detection limits (MDLs) should meet the criteria given in USEPA CLP SOW SOM01.2.
3. The assigned laboratory must have determined MDLs annually meeting the requirements in USEPA CLP SOW SOM01.2.
4. The achievable Laboratory Limits listed as based upon USEPA CLP CRQLs for trace water samples. The actual achievable QLs based upon the assigned laboratory MDL studies should be equivalent or lower.
5. Refer Table 3-1 in the work plan.
   Risk-Based Screening Levels are the USEPA Region 3 Regional Screening Levels for tapwater (12SEP2008) accessed online at: www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm. For non-carcinogens (nc), the range represents screening levels for target hazard quotients of 0.1 and 1. For carcinogens (ca), the range represents screening levels for target cancer risks of 10^{-6} and 10^{-4}.
6. Refer to Table 3-1 in the work Plan. Potential ARARs, selected as the lowest of the Federal MCLs (40 CFR 141), State MCLs (N.J.A.C. 7:10-5.2 & 7.2), and the NJ Groundwater Quality Criteria (N.J.A.C. 7:9-6).
7. Bold – indicates Preliminary COPCs, refer to Table 3-1 in the work plan.

QAPP Worksheet #15
(UFP-QAPP Manual Section 2.8.1)

Reference Limits and Evaluation Table

Matrix: Groundwater

Analytical Group: TCL Semi-Volatiles (SOM01.2 Low Water)

Concentration Level: Low

Analyte	CAS Number	Risk-Based Screening Level [4] (µg/L)	Potential ARARs Screening Levels[5] (µg/L)	Project Quantitation Limit [1] (ug/L)	Analytical Method		Achievable Laboratory Limits (Based upon CLP CRQLs)	
					MDLs	Method QLs	MDLs	QLs (ug/L)[4]
Benzaldehyde	100-52-7	370 – 3,700	N/A	5.0	Note 2	5.0	Note 3	5.0
Phenol	108-95-2	1,100 – 11,000	2,000	5.0	Note 2	5.0	Note 3	5.0
bis(2-Chloroethyl) ether	111-44-4	0.012 -1.2 (ca)	7	5.0	Note 2	5.0	Note 3	5.0
2-Chlorophenol	95-57-8	18 - 180	40	5.0	Note 2	5.0	Note 3	5.0
2-Methylphenol	95-48-7	180 – 1,800	N/A	5.0	Note 2	5.0	Note 3	5.0
2,2'-oxybis(1-Chloropropane)	108-60-1	0.32 - 32 (ca)	N/A	5.0	Note 2	5.0	Note 3	5.0
Acetaphenone	98-86-2	370 – 3,700	N/A	5.0	Note 2	5.0	Note 3	5.0
4-Methylphenol	106-44-5	18 - 180	N/A	5.0	Note 2	5.0	Note 3	5.0
N-Nitroso-di-n-propylamine	621-64-7	0.0096 – 0.96 (ca)	10	5.0	Note 2	5.0	Note 3	5.0
Hexachloroethane	67-72-1	4.8 - 480 (ca)	7	5.0	Note 2	5.0	Note 3	5.0
Nitrobenzene	98-95-3	0.34 – 3.4	6	5.0	Note 2	5.0	Note 3	5.0
Isophorone	78-59-1	71 – 7,100 (ca)	40	5.0	Note 2	5.0	Note 3	5.0
2-Nitrophenol	88-75-5	N/A	N/A	5.0	Note 2	5.0	Note 3	5.0
2,4-Dimethylphenol	105-67-9	73 – 7,300	100	5.0	Note 2	5.0	Note 3	5.0
bis(2-Chloroethoxy) methane	111-91-1	11 – 110	N/A	5.0	Note 2	5.0	Note 3	5.0
2,4-Dichlorophenol	120-83-2	11 - 110	20	5.0	Note 2	5.0	Note 3	5.0
Naphthalene	91-20-3	0.14 – 1.4	300	5.0	Note 2	5.0	Note 3	0.1 (SIM)
1,4-Dioxane	123-91-1	6.1 - 610 (ca)	10	2.0	Note 2	2.0	Note 3	2.0
4-Chloroaniline	106-47-8	15 - 150	30	5.0	Note 2	5.0	Note 3	5.0
Hexachlorobutadiene	87-68-3	0.86 - 86 (ca)	1	5.0	Note 2	5.0	Note 3	5.0
Caprolactam	105-60-2	1,800 – 18,000	5,000	5.0	Note 2	5.0	Note 3	5.0
4-Chloro-3-methyphenol	59-50-7	N/A	N/A	5.0	Note 2	5.0	Note 3	5.0

Reference Limits and Evaluation Table

Matrix: Groundwater

Analytical Group: TCL Semi-Volatiles (SOM01.2 Low Water)

Concentration Level: Low

Analyte	CAS Number	Risk-Based Screening Level [4] (µg/L)	Potential ARARs Screening Levels[5] (µg/L)	Project Quantitation Limit [1] (ug/L)	Analytical Method		Achievable Laboratory Limits (Based upon CLP CRQLs)	
					MDLs	Method QLs	MDLs	QLs (ug/L)[4]
2-Methylnaphthalene	91-57-6	15 - 150	30	5.0	Note 2	5.0	Note 3	5.0
Hexachlorocyclopentadiene	77-47-4	22 - 220	40	5.0	Note 2	5.0	Note 3	5.0
2,4,6-Trichlorophenol	88-06-2	6.1 - 610 (ca)	20	5.0	Note 2	5.0	Note 3	5.0
2,4,5-Trichlorophenol	95-95-4	370- 3,700	700	5.0	Note 2	5.0	Note 3	5.0
1,1'-Biphenyl	92-52-4	180 – 1,800	N/A	5.0	Note 2	5.0	Note 3	5.0
2-Chloronapthalene	91-58-7	290 – 2,900	600	5.0	Note 2	5.0	Note 3	5.0
2-Nitroaniline	88-74-4	N/A	N/A	10	Note 2	10	Note 3	10
Dimethylphthalate	131-11-3	N/A	N/A	5.0	Note 2	5.0	Note 3	5.0
2,6-Dinitrotoluene	606-20-2	3.7 - 37	N/A	5.0	Note 2	5.0	Note 3	5.0
Acenaphthylene	208-96-8	N/A	N/A	5.0	Note 2	5.0	Note 3	5.0
3-Nitroaniline	99-09-2	N/A	N/A	10	Note 2	10	Note 3	10
Acenaphthene	83-32-9	220 – 2,200	400	5.0	Note 2	5.0	Note 3	5.0
2,4-Dinitrophenol	51-28-5	7.3 - 73	40	10	Note 2	10	Note 3	10
4-Nitrophenol	100-02-7	N/A	N/A	10	Note 2	10	Note 3	10
Dibenzofuran	132-64-9	N/A	N/A	5.0	Note 2	5.0	Note 3	5.0
2,4-Dinitrotoluene	121-14-2	7.3 - 73	N/A	5.0	Note 2	5.0	Note 3	5.0
Diethylphthalate	84-66-2	2,900 – 29,000	6,000	5.0	Note 2	5.0	Note 3	5.0
4-Chlorophenyl-phenylether	7005-72-3	N/A	N/A	5.0	Note 2	5.0	Note 3	5.0
Fluorene	86-73-7	150 – 1,500	300	5.0	Note 2	5.0	Note 3	5.0
4-Nitroaniline	100-01-6	N/A	N/A	10	Note 2	10	Note 3	10
4,6-Dinitro-2-methylphenol	534-52-1	N/A	N/A	10	Note 2	10	Note 3	10
N-Nitrosodiphenylamine	86-30-6	14 – 1,400 (ca)	10	5.0	Note 2	5.0	Note 3	5.0
1,2,4,5-Tetrachlorobenzene	95-94-3	1.1 - 11	N/A	5.0	Note 2	5.0	Note 3	5.0
4-Bromophenyl-phenylether	101-55-3	N/A	N/A	5.0	Note 2	5.0	Note 3	5.0
Hexachlorobenzene	118-74-1	0.042 – 0.42	0.02	5.0	Note 2	5.0	Note 3	5.0
Atrazine	1912-24-9	0.29 - 29 (ca)	3	5.0	Note 2	5.0	Note 3	5.0

Reference Limits and Evaluation Table

Matrix: Groundwater

Analytical Group: TCL Semi-Volatiles (SOM01.2 Low Water)

Concentration Level: Low

Analyte	CAS Number	Risk-Based Screening Level [4] (µg/L)	Potential ARARs Screening Levels[5] (µg/L)	Project Quantitation Limit [1] (ug/L)	Analytical Method		Achievable Laboratory Limits (Based upon CLP CRQLs)	
					MDLs	Method QLs	MDLs	QLs (ug/L)[4]
Pentachlorophenol	87-86-5	0.56 - 56 (ca)	0.3	10	Note 2	10	Note 3	0.2 (SIM)
Phenathrene	85-01-8	N/A	N/A	5.0	Note 2	5.0	Note 3	5.0
Anthracene	120-12-7	1,100 – 11,000	2,000	5.0	Note 2	5.0	Note 3	5.0
Carbazole	86-74-8	3.4 - 340 (ca)	N/A	5.0	Note 2	5.0	Note 3	5.0
Di-n-butylphthalate	84-74-2	370 – 3,700	700	5.0	Note 2	5.0	Note 3	5.0
Fluoranthene	206-44-0	150 – 1,500	300	5.0	Note 2	5.0	Note 3	5.0
Pyrene	129-00-0	110 – 1,100	200	5.0	Note 2	5.0	Note 3	5.0
Butylbenzylphthalate	85-68-7	730 – 7,300	100	5.0	Note 2	5.0	Note 3	5.0
3,3'-Dichlorobenzidene	91-94-1	0.15 – 15 (ca)	30	5.0	Note 2	5.0	Note 3	5.0
Benzo(a)anthracene	56-55-3	0.029 – 2.9 (ca)	0.1	5.0	Note 2	5.0	Note 3	0.1 (SIM)
Chrysene	218-01-9	2.9 - 290 (ca)	5	5.0	Note 2	5.0	Note 3	0.1 (SIM)
bis-(2-Ethylhexyl)phthalate	117-81-7	4.8 - 480 (ca)	3	5.0	Note 2	5.0	Note 3	5.0
Di-n-octylphthalate	117-84-0	N/A	100	5.0	Note 2	5.0	Note 3	5.0
Benzo(b)fluoranthene	205-99-2	0.029 – 2.9 (ca)	0.2	5.0	Note 2	5.0	Note 3	0.1 (SIM)
Benzo(k)fluoranthene	207-08-9	0.29 - 29 (ca)	0.5	5.0	Note 2	5.0	Note 3	0.1 (SIM)
Benzo(a)pyrene	50-32-8	0.0029 – 0.29 (ca)	0.1	5.0	Note 2	5.0	Note 3	0.1 (SIM)
Indeno(1,2,3-cd)pyrene	193-39-5	0.029 – 2.9 (ca)	0.2	5.0	Note 2	5.0	Note 3	0.1 (SIM)
Dibenzo(a,h)anthracene	53-70-3	0.0029 – 0.29 (ca)	0.3	5.0	Note 2	5.0	Note 3	0.1 (SIM)
Benzo(g,h,I)perylene	191-24-2	N/A	N/A	5.0	Note 2	5.0	Note 3	5.0
2,3,4,6-Tetrachlorphenol	58-90-2	110 – 1,100	N/A	5.0	Note 2	5.0	Note 3	5.0

1. The project quantitation limits are based upon USEPA CLP contract required Quantitation limits (CRQLs) given in CLP SOW SOM01.2. The project quantitation limit can increase depending upon percent moisture.
2. The method detection limits (MDLs) should meet the criteria given in USEPA CLP SOW SOM01.2.
3. The assigned laboratory must have determined MDLs annually meeting the requirements in USEPA CLP SOW SOM01.2.

4. The achievable Laboratory Limits listed as based upon USEPA CLP CRQLs for low water samples. The actual achievable QLs based upon the assigned laboratory MDL studies should be similar.
5. Refer Table 3-1 in the work plan.
   Risk-Based Screening Levels are the USEPA Region 3 Regional Screening Levels for tapwater (12SEP2008) accessed online at: www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm. For non-carcinogens (nc), the range represents screening levels for target hazard quotients of 0.1 and 1. For carcinogens (ca), the range represents screening levels for target cancer risks of 10^{-6} and 10^{-4}.
6. Refer to Table 3-1 in the work Plan. Potential ARARs, selected as the lowest of the Federal MCLs (40 CFR 141), State MCLs (N.J.A.C. 7:10-5.2 & 7.2), and the NJ Groundwater Quality Criteria (N.J.A.C. 7:9-6).
7. Bold – indicates Preliminary COPCs, refer to Table 3-1 in the work plan.

QAPP Worksheet #15
(UFP-QAPP Manual Section 2.8.1)

Reference Limits and Evaluation Table

Matrix: Aqueous (Groundwater)

Analytical Group: TCL Pesticides (SOM01.2 with modifications to lower the CRQLs)

Concentration Level: Low

Analyte	CAS Number	Risk-Based Screening Level[5] (µg/L)	Potential ARARs Screening Levels[6] (µg/L)	Project Quantitation Limit [1] (ug/L)	Analytical Method		Achievable Laboratory Limits (Based upon CLP CRQLs)	
					MDLs	Method QLs	MDLs	QLs (ug/L)[4]
alpha-BHC	319-84-6	0.011 – 1.1 (ca)	0.02	0.05	Note 2	0.05	Note 3	0.05
beta-BHC	319-85-7	0.037 – 3.7 (ca)	0.04	0.05	Note 2	0.05	Note 3	0.05
delta-BHC	319-86-8	N/A	100	0.05	Note 2	0.05	Note 3	0.05
gamma-BHC (Lindane)	58-89-9	0.061 – 6.1 (ca)	0.03	0.05	Note 2	0.05	Note 3	0.05
Heptachlor	76-44-8	0.015 – 1.5 (ca)	0.05	0.05	Note 2	0.05	Note 3	0.05
Aldrin	309-00-2	0.004 – 0.4 (ca)	0.04	0.05	Note 2	0.05	Note 3	0.05
Heptachlor epoxide	1024-57-3	0.0074 – 0.74 (ca)	0.2	0.05	Note 2	0.05	Note 3	0.05
Endosulfan I	959-98-8	N/A	40	0.05	Note 2	0.05	Note 3	0.05
Dieldrin	60-57-1	0.0042 – 0.42 (ca)	0.03	0.1	Note 2	0.1	Note 3	0.1
4,4'-DDE	*72-55-9*	*0.2 – 20 (ca)*	*0.1*	*0.1*	*Note 2*	*0.1*	*Note 3*	*0.1*
Endrin	72-20-8	1.1 - 11	2	0.1	Note 2	0.1	Note 3	0.1
Endosulfan II	33213-65-9	N/A	N/A	0.1	Note 2	0.1	Note 3	0.1
4,4'-DDD	72-54-8	0.28 - 28 (ca)	0.1	0.1	Note 2	0.1	Note 3	0.1
Endosulfan sulfate	1031-07-8	N/A	40	0.1	Note 2	0.1	Note 3	0.1
4,4'-DDT	50-29-3	0.2 - 20 (ca)	0.1	0.1	Note 2	0.1	Note 3	0.1
Methoxychlor	72-43-5	18 - 180	40	0.5	Note 2	0.5	Note 3	0.5
Endrin ketone	53494-70-5	N/A	N/A	0.1	Note 2	0.1	Note 3	0.1
Endrin aldehyde	7421-93-4	N/A	N/A	0.1	Note 2	0.1	Note 3	0.1
alpha-Chlordane	5103-71-9	N/A	N/A	0.05	Note 2	0.05	Note 3	0.05
gamma-Chlordane	5103-74-2	N/A	N/A	0.05	Note 2	0.05	Note 3	0.05
Toxaphene	8001-35-2	0.061 – 6.1 (ca)	2	5	Note 2	5	Note 3	5

1. The project quantitation limits are based upon USEPA CLP contract required Quantitation limits (CRQLs) given in CLP SOW SOM01.2.
2. The method detection limits (MDLs) should meet the criteria given in USEPA CLP SOW SOM01.2.
3. The assigned laboratory must have determined MDLs annually meeting the requirements in USEPA CLP SOW SOM01.2.
4. The achievable Laboratory Limits listed as based upon USEPA CLP CRQLs for low water samples. The actual achievable QLs based upon the assigned laboratory MDL studies should be similar.
5. Refer Table 3-1 in the work plan.
   Risk-Based Screening Levels are the USEPA Region 3 Regional Screening Levels for tapwater (12SEP2008) accessed online at: www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm. For non-carcinogens (nc), the range represents screening levels for target hazard quotients of 0.1 and 1. For carcinogens (ca), the range represents screening levels for target cancer risks of 10^{-6} and 10^{-4}.
6. Refer to Table 3-1 in the work Plan. Potential ARARs, selected as the lowest of the Federal MCLs (40 CFR 141), State MCLs (N.J.A.C. 7:10-5.2 & 7.2), and the NJ Groundwater Quality Criteria (N.J.A.C. 7:9-6).
7. Bold – indicates Preliminary COPCs, refer to Table 3-1 in the work plan.

QAPP Worksheet #15
(UFP-QAPP Manual Section 2.8.1)

Reference Limits and Evaluation Table

Matrix: Aqueous (Groundwater)

Analytical Group: TCL PCB Aroclors (SOM01.2 with modifications to low CRQLs)

Concentration Level: Low

Analyte	CAS Number	Risk-Based Screening Level[5] (µg/L)	Potential ARARs Screening Levels[6] (µg/L)	Project Quantitation Limit [1] (ug/L)	Analytical Method		Achievable Laboratory Limits (Based upon required CRQLs) [4]	
					MDLs	Method QLs	MDLs [4]	QLs (ug/L)[4]
Aroclor-1016	12674-11-2	0.96 – 96 (ca)	N/A	0.1	Note 2	0.1	Note 3	0.1
Aroclor-1221	11104-28-2	0.0068 – 0.68 (ca)	N/A	0.1	Note 2	0.1	Note 3	0.1
Aroclor-1232	11141-16-5	0.0068 – 0.68 (ca)	N/A	0.1	Note 2	0.1	Note 3	0.1
Aroclor-1242	53469-21-9	0.034 – 3.4 (ca)	N/A	0.1	Note 2	0.1	Note 3	0.1
Aroclor-1248	12672-29-6	0.034 – 3.4 (ca)	N/A	0.1	Note 2	0.1	Note 3	0.1
Aroclor-1254	11097-69-1	0.034 – 3.4 (ca)	N/A	0.1	Note 2	0.1	Note 3	0.1
Aroclor-1260	11096-82-5	0.034 – 3.4 (ca)	N/A	0.1	Note 2	0.1	Note 3	0.1
Aroclor-1262	37324-23-5	N/A	N/A	0.1	Note 2	0.1	Note 3	0.1
Aroclor-1268	11100-14-4	N/A	N/A	0.1	Note 2	0.1	Note 3	0.1
Total PCBs		0.0068 – 0.68 (ca)	0.5					

1. The project quantitation limits are based upon USEPA CLP contract required Quantitation limits (CRQLs) given in CLP SOW SOM01.2.
2. The method detection limits (MDLs) should meet the criteria given in USEPA CLP SOW SOM01.2.
3. The assigned laboratory must have determined MDLs annually meeting the requirements in USEPA CLP SOW SOM01.2.
4. The achievable Laboratory Limits listed as based upon USEPA CLP CRQLs for low water samples assuming the extract is concentrated a by a factor of 10 (to 1 mL). The actual achievable QLs based upon the assigned laboratory MDL studies should be similar.
5. Refer Table 3-1 in the work plan.
   Risk-Based Screening Levels are the USEPA Region 3 Regional Screening Levels for tapwater (12SEP2008) accessed online at: www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm. For non-carcinogens (nc), the range represents screening levels for target hazard quotients of 0.1 and 1. For carcinogens (ca), the range represents screening levels for target cancer risks of 10^{-6} and 10^{-4}.

6. Refer to Table 3-1 in the work Plan. Potential ARARs, selected as the lowest of the Federal MCLs (40 CFR 141), State MCLs (N.J.A.C. 7:10-5.2 & 7.2), and the NJ Groundwater Quality Criteria (N.J.A.C. 7:9-6).
7. Bold – indicates Preliminary COPCs, refer to Table 3-1 in the work plan.

QAPP Worksheet #15
(UFP-QAPP Manual Section 2.8.1)

Reference Limits and Evaluation Table

Matrix: Aqueous (Groundwater)

Analytical Group: TAL Metals, Mercury and Cyanide (ILM05.4)

Concentration Level: Low

Analyte	CAS Number	Risk-Based Screening Level[5] (µg/L)	Potential ARARs Screening Levels[6] (µg/L)	Project Quantitation Limit [1] (ug/L)	Analytical Method		Achievable Laboratory Limits (Based upon CLP CRQLs) [4]	
					MDLs	Method QLs	MDLs [4]	QLs (ug/L)[4]
Aluminum	7429-90-5	3,700 – 37,000	50	200	Note 2	200	Note 3	200
Antimony	7440-36-0	1.5 - 15	6	2	Note 2	2	Note 3	2
Arsenic	7440-38-2	0.045 – 4.5 (ca)	3	1	Note 2	1	Note 3	1
Barium	7440-39-3	730 – 7,300	2,000	10	Note 2	10	Note 3	10
Beryllium	7440-41-7	7.3 - 73	1	1	Note 2	1	Note 3	1
Cadmium	7440-43-9	1.8 - 18	4	1	Note 2	1	Note 3	1
Calcium	7440-70-2	N/A	N/A	5000	Note 2	5000	Note 3	5000
Chromium	7440-47-3	N/A	70	2	Note 2	2	Note 3	2
Cobalt	7440-48-4	1.1 - 11	100	1	Note 2	1	Note 3	1
Copper	7440-50-8	150 – 1,500	1,300	2	Note 2	2	Note 3	2
Iron	7439-89-6	2,600 – 26,000	300	100	Note 2	100	Note 3	100
Lead	7439-92-1	N/A	5	1	Note 2	1	Note 3	1
Magnesium	7439-95-4	N/A	N/A	5000	Note 2	5000	Note 3	5000
Manganese	7439-96-5	88 - 880	50	1	Note 2	1	Note 3	1
Mercury	7439-97-6	0.063 – 0.63	2	0.2	Note 2	0.2	Note 3	0.2
Nickel	7440-02-0	73 - 730	100	1	Note 2	1	Note 3	1
Potassium	2023695	N/A	N/A	5000	Note 2	5000	Note 3	5000
Selenium	7782-49-2	18 - 180	40	5	Note 2	5	Note 3	5
Silver	7440-22-4	18 - 180	40	1	Note 2	1	Note 3	1
Sodium	7440-23-5	N/A	50,000	5000	Note 2	5000	Note 3	5000

Reference Limits and Evaluation Table

Matrix: Aqueous (Groundwater)

Analytical Group: TAL Metals, Mercury and Cyanide (ILM05.4)

Concentration Level: Low

Analyte	CAS Number	Risk-Based Screening Level[5] (µg/L)	Potential ARARs Screening Levels[6] (µg/L)	Project Quantitation Limit[1] (ug/L)	Analytical Method		Achievable Laboratory Limits (Based upon CLP CRQLs)[4]	
					MDLs	Method QLs	MDLs[4]	QLs (ug/L)[4]
Thallium	7440-28-0	0.24 – 2.4	2	1	Note 2	1	Note 3	1
Vanadium	7440-62-2	26 - 260	N/A	5	Note 2	5	Note 3	5
Zinc	7440-66-6	1,100 – 11,000	2,000	2	Note 2	2	Note 3	2
Cyanide	57-12-5	73 - 730	100	10	Note 2	10	Note 3	10

1. The project quantitation limits are based upon USEPA CLP contract required Quantitation limits (CRQLs) given in CLP SOW ILM05.4.
2. The method detection limits (MDLs) should meet the criteria given in USEPA CLP SOW ILM05.4.
3. The assigned laboratory must have determined MDLs annually meeting the requirements in USEPA CLP SOW ILM05.4.
4. The achievable Laboratory Limits listed as based upon USEPA CLP CRQLs for low water samples using both ICP-AES and IPC-MS for the metals. The actual achievable QLs based upon the assigned laboratory MDL studies should be similar.
5. Refer Table 3-1 in the work plan.
   Risk-Based Screening Levels are the USEPA Region 3 Regional Screening Levels for tapwater (12SEP2008) accessed online at: www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm. For non-carcinogens (nc), the range represents screening levels for target hazard quotients of 0.1 and 1. For carcinogens (ca), the range represents screening levels for target cancer risks of 10^{-6} and 10^{-4}.
6. Refer to Table 3-1 in the work Plan. Potential ARARs, selected as the lowest of the Federal MCLs (40 CFR 141), State MCLs (N.J.A.C. 7:10-5.2 & 7.2), and the NJ Groundwater Quality Criteria (N.J.A.C. 7:9-6).
7. Bold – indicates Preliminary COPCs, refer to Table 3-1 in the work plan.

QAPP Worksheet #15
(UFP-QAPP Manual Section 2.8.1)

Reference Limits and Evaluation Table

Matrix: Groundwater

Analytical Group: TOC and DOC

Concentration Level: Low

Analyte	CAS Number	Project Action Limit	Project Quantitation Limit	Analytical Method		Achievable Laboratory Limit based upon Typical Lab Reporting Limit	
				MDLs (mg/L)	Method QLs	MDLs (mg/L) [3]	QLs (mg/L) [3]
Total Organic Carbon	NA	Note 1	NA	Note 2	Note 2	0.311	1
Dissolved Organic Carbon	NA	Note 1	NA	Note 2	Note 2	0.311	1

1. This is a screening parameter, which is an indicator of biodegradation. There is no regulatory action level for this parameter. TOC above 20 mg/L helps to drive dechlorination.
2. Reference SW-846-9060A.
3. The MDLs and QLs are based upon Accutest MDL studies. The detection limits may differ slightly if the USEPA DESA lab or a backup subcontract lab performs the analysis.

QAPP Worksheet #15
(UFP-QAPP Manual Section 2.8.1)

Reference Limits and Evaluation Table

Matrix: Groundwater

Analytical Group: Nitrate

Concentration Level: Low

Analyte	CAS Number	Project Action Limit[1] (mg/L)	Project Quantitation Limit (mg/L)	Analytical Method		Achievable Laboratory Limits based upon Typical Lab Reporting Limits	
				MDLs (mg/L)	Method QLs	MDLs (mg/L)[3]	QLs (mg/L)[3]
Nitrate	14797-55-8	Note 1	NA	0.008	0.10	0.008	0.10

1. This is a screening parameter. There is no project action level for this project for this parameter.
2. Reference EPA 353.2, EPA300.0/SW-846-9056 or equivalent methods or the DESA method utilized by the assigned lab.
3. The limits are based upon Accutest EPA 353.2 MDL. The limits may differ slightly if a backup lab performs the analysis.

QAPP Worksheet #15
(UFP-QAPP Manual Section 2.8.1)

Reference Limits and Evaluation Table

Matrix: Groundwater

Analytical Group: Anions: Sulfate, Chloride

Concentration Level: Low

Analyte	CAS Number	Project Action Limit[1] (mg/L)	Project Quantitation Limit (mg/L)	Analytical Method		Achievable Laboratory Limits based upon Typical Lab Reporting Limits	
				MDLs (mg/L)	Method QLs	MDLs (mg/L)[3]	QLs (mg/L)[3]
Sulfate	14808-79-8	Note 1	NA	Note 2	Note 2	0.5	0.118
Chloride	16887-00-6	Note 1	NA	Note 2	Note 2	0.5	0.124

1. These are screening parameters, which are indicators of biodegradation. There are no regulatory action levels.
2. See reference Table 1A of EPA Methods 300.0/SW-846-9056. The DESA lab method is also acceptable. Optionally Nitrate can also be determined these ion chromatography methods.
3. The MDLs and QLs are based upon Accutest MDL studies. The detection limits may differ slightly if a backup lab performs the analysis actual reporting limits may differ slightly.

QAPP Worksheet #15

(UFP-QAPP Manual Section 2.8.1)

Reference Limits and Evaluation Table

Matrix: Groundwater

Analytical Group: Ferrous Iron

Concentration Level: Low

Analyte	CAS Number	Project Action Limit[1] (mg/L)	Project Quantitation Limit[2] (mg/L)	Analytical Method		Estimated Achievable Test Limits	
				MDLs	Method QLs	MDLs [2,3]	QLs (mg/L) [2,3]
Ferrous Iron	1345-25-1	Note 1	NA	Note 2	Note 2	0.03	0.1

1. There is no regulatory action level for this parameter, but Ferrous Iron over 1 mg/L may be an indication of biodegradation.
2. See the estimated achievable MDL and QL are estimated based SM 3500 Fe-D.
3. The MDL and QL listed are based upon Accutest MDL studies. The limits may differ if a different lab performs the analysis.

QAPP Worksheet #15
(UFP-QAPP Manual Section 2.8.1)

Reference Limits and Evaluation Table

Matrix: Groundwater

Analytical Group: Hardness (As CaCO3)

Concentration Level: Low

Analyte	CAS Number	Project Action Limit[1] (mg/L)	Project Quantitation Limit[2] (mg/L)	Analytical Method		Achievable Laboratory Limit based upon Typical Lab Reporting Limits	
				MDLs	Method QLs	MDLs [3]	QLs (mg/L) [3]
Hardness	NA	Note 1	NA	Note 2	Note 2	2.3	4

1. This is groundwater screening parameters. There is no regulatory action level for this parameter.
2. See the test method for Hardness SM 2340B.
3. The MDLs and QLs are based upon Accutest detection limits. Reporting limits will be similar, but will differ if a backup lab is employed.

QAPP Worksheet #15

(UFP-QAPP Manual Section 2.8.1)

Reference Limits and Evaluation Table

Matrix: Groundwater

Analytical Group: Methane, Ethane and Ethene

Concentration Level: Low

Analyte	CAS Number	Project Action Limit (ug/L)	Project Quantitation Limit (ug/L)	Analytical Method		Achievable Laboratory Limit based upon Typical Lab Reporting Limits	
				MDLs (ug/L)	Method QLs	MDLs (ug/L)[3]	QLs (ug/L)[3]
Methane	14808-79-8	Note 1	NA	Note 2	Note 2	0.0220	0.1000
Ethane	14797-55-8	Note 1	NA	Note 2	Note 2	0.013	0.050
Ethene	14797-65-0	Note 1	NA	Note 2	Note 2	0.0015	0.0050

1. These are ground water natural attenuation screening parameters. There are no specific project action levels have been set, but these dissolved gases are the ultimate daughter products of chlorinated solvents and their presence are a good indicator of reducing conditions.
2. See methods SW-846 8015B and EPA RSK-175.
3. The MDLs and QLs are based upon Accutest detection limits, if a backup lab performs the analysis they may differ slightly.

QAPP Worksheet #15
(UFP-QAPP Manual Section 2.8.1)

Reference Limits and Evaluation Table

Matrix: Solids (Bedrock)

Analytical Group: VOCs

Concentration Level: Low

Analyte	CAS Number	Project Action Limit (ug/kg) [2]	Project Quantitation Limit (ug/kg)	Analytical Method		Achievable Laboratory Limits	
				MDLs (ug/kg) [3]	Method QLs (ug/kg) [3]	MDLs (ug/kg) [3]	QLs (ug/kg) [3]
1,1,-Dichloroethene	75-35-4	5.2	10	0.6	10	0.6	10
Trans-1,2-Dichloroethene	156-60-5	400	10	1.2	10	1.2	10
Cis-1,2-Dichloroethene [1]	**156-59-2**	220	10	0.3	10	0.3	10
Trichloroethene [1]	**79-01-6**	7.0	1.0	0.2	1.0	0.2	1.0
Tetrachloroethene [1]	**127-18-4**	2.9	1.0	0.2	1.0	0.2	1.0
Trichlorotrifluoroethane	75-69-4	22,000	1.0	0.2	1.0	0.2	1.0
Chloroform	67-66-3	230	1.0	0.7	1.0	0.7	1.0
1,1,1-Trichloroethane	71-55-6	180	1.0	0.1	1.0	0.1	1.0
Carbon Tetrachloride	56-23-5	3.5	1.0	0.1	1.0	0.1	1.0

1. The bolded chemicals are compounds of potential concern (COPCs) previously identified in bedrock groundwater samples per Table 3-1 in the Work Plan.
2. The preliminary remediation goals for bedrock have not been established for the study area. Appropriate applicable action levels are not available for the bedrock matrix, but the values listed are based upon 2008 New Jersey Health-Based Soil Impact to Ground Water criteria.
3. Based upon Stone Environmental detection limits by SOP SEI-10.18.0.

QAPP Worksheet #15
(UFP-QAPP Manual Section 2.8.1)

Reference Limits and Evaluation Table

Matrix: Solids (Bedrock)

Analytical Group: PCBs

Concentration Level: Low

Analyte	CAS Number	Project Action Limit (ug/kg)	Project Quantitation Limit (ug/kg)[2]	Analytical Method		Achievable Laboratory Limits (Based upon Accutest detection limits)	
				MDLs	QLs	MDLs	QLs(ug/kg)[3,4]
Aroclor 1016	12674-11-2	Note 1	33	NA	NA	1.28	6.8
Aroclor 1221	11104-28-2	Note 1	33	NA	NA	4.0	6.8
Aroclor 1232	11141-16-5	Note 1	33	NA	NA	3.6	6.8
Aroclor 1242	53469-21-9	Note 1	33	NA	NA	2.0	6.8
Aroclor 1248	12672-29-6	Note 1	33	NA	NA	2.4	6.8
Aroclor 1254	11097-69-1	Note 1	33	NA	NA	3.2	6.8
Aroclor 1260	11096-82-5	Note 1	33	NA	NA	1.36	6.8
Aroclor 1262	37324-23-5	Note 1	33	NA	NA	1.23	6.8
Aroclor 1268	11100-14-4	Note 1	33	NA	NA	2.95	6.8
Total PCB (Sum of all the Aroclors)		160 (Note 1)					

1. Applicable action levels have not been set for PCB Aroclors in bedrock, but the 2008 New Jersey Health-Based Soil Impact to Ground Water criteria for PCBs is 1.6 E-01 mg/kg or 160 ug/kg, while the NJ Residential Direct Contact Soil Remediation Standard for total PCBs is 0.2 ppm or 200 ug/kg.
2. The project quantitation limits are based upon the CLP CRQLs.
3. The MDLs and QLs are based upon Accutest detection limits assuming that the sample extract can be concentrated by a factor of 5 to achieve lower detection limits.
4. If the DESA lab, an assigned CLP lab or a backup laboratory is employed the detection limits may differ, but should be low enough to support the project quantitation limits.

QAPP Worksheet #15
(UFP-QAPP Manual Section 2.8.1)

Reference Limits and Evaluation Table

Matrix: Solids (Bedrock)

Analytical Group: Total Organic Carbon

Concentration Level: Low

Analyte	CAS Number	Project Action Limit[1] (mg/L)	Project Quantitation Limit (mg/L)	Analytical Method		Achievable Laboratory Limits	
				MDLs (mg/L)	Method QLs	MDLs (mg/L)[3]	QLs (mg/L)[3]
Total Organic Carbon	NA	Note 1	NA	Note 2	Note 2	40.221	100.0

1. This is a screening parameter. There is no project action level for this project for this parameter.
2. Reference USEPA Region 2 Lloyd Kahn or equivalent method or DESA method utilized by lab.
3. The limits are based upon Accutest MDL studies. The detection limits may differ slightly if a backup lab performs the analysis.

QAPP Worksheet #16 (UFP-QAPP Manual Section 2.8.2)

Project Schedule Timeline Table

Activities	Organization	Dates		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
Planning and Development of DQOs	Malcolm Pirnie	June 2008	July 2008	DQOs	July 2008
Development of the Sampling Rationale, FSP and associated sampling SOPs	Malcolm Pirnie	June 2008	October 2008	Final FSP	October 2008
Preparation of the QAPP and the testing QA requirements	Malcolm Pirnie	June 2008	November 2008	Final QAPP	November 2008
Preparation of SSHP	Malcolm Pirnie	June 2008	November 2008	Final SSHP	November 2008
Submit DESA/CLP request forms to Jennifer Feranda	Malcolm Pirnie	December 2008	January 2009	Submitted forms	One month before sampling begins
Collection of Bedrock Samples and Submission for Analysis	Malcolm Pirnie	January 2009	March 2009	Samples collected per Final QAPP/FSP	Spring 2009
Collection of Ground Water Samples Round 1	Malcolm Pirnie	May 2009	May 2009	Samples collected per Final QAPP/FSP	May 2009
Collection of Ground Water Samples Round 2	Malcolm Pirnie	August 2009	August 2009	Samples collected per Final QAPP/FSP	August 2009
Collection of Ground Water Samples Round 3	Malcolm Pirnie	November 2009	November 2009	Samples collected per Final QAPP/FSP	November 2009
Submit Trip COC Reports	Malcolm Pirnie	At time of sample collection	May/August/November 2009	Included with samples	When samples are collected

Project Schedule Timeline Table

Activities	Organization	Dates		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
Laboratory Analyses	Laboratories assigned by USEPA-CLP, DESA and subcontract labs	2009	CLP Analyses should be completed within 21 days of receipt of samples. Subcontract lab analysis within 35 days of receipt of samples	Analytical data to USEPA or Malcolm Pirnie assigned data validators	One month after sample are analyzed
Validation, Review and Verification of Data	USEPA Region 2 and Malcolm Pirnie	2009	EPA Validation should be completed within 21 days after the laboratory provides the data packages. Malcolm Pirnie validations of subcontract lab data 35 days after data packages are received.	Validated data reported	Two month after all the lab data packages are available

Note: A more detailed project schedule is maintained by the USACE and Malcolm Pirnie Project Manager. An up-to-date copy of the Project Schedule will be available to the project team members.

QAPP Worksheet #17
(UFP-QAPP Section 3.1.1)

Sampling Design and Rationale

See the FSP Section 3 and table 3-1 in QAPP Appendix A for the details of the sampling design and rationale.

QAPP Worksheet #18
(UFP-QAPP Manual Section 3.1.1)

Sampling Locations and Methods/SOP Requirements Table (Bedrock Sampling Event)

Sampling Location/ID Number	Matrix	Depth (Ft.)	Analytical Group	Concentration Level	Est. Number of Samples (identify field duplicates)	Sampling SOP Reference	Rationale for Sampling Location
Wells MW 14, 16, 19, and 20 per FSP table 3-2	Bedrock	Every 2 ft. to 250 ft.	Selected Chlorinated Volatiles	Low	480 plus duplicates 1 per 20 samples	See Worksheet 21	See FSP in Appendix A
Wells MW 14, 16, 19, and 20 per FSP table 3-2	Bedrock	Every 20 ft.	Physical Property Tests: Density, Specific Gravity, Porosity, Moisture plus TOC	Low	60 samples plus duplicates 1 per 20 samples	See Worksheet 21	See FSP in Appendix A
Wells MW 14, 16, per FSP table 3-2	Bedrock	Every 2 ft. to 250 ft.	PCB Aroclors	Low	240 plus duplicates 1 per 20 samples	See Worksheet 21	See FSP in Appendix A
Wells MW 14, 16, per FSP table 3-2	Bedrock	Every 125 ft.	Rock Oxidant Demand	Low	4 plus 1 duplicate	See Worksheet 21	See FSP in Appendix A

QAPP Worksheet #18 (Continued)
(UFP-QAPP Manual Section 3.1.1)

Sampling Locations and Methods/SOP Requirements Table (Groundwater Event No. 1)

Sampling Location/ID Number	Matrix	Depth (inches)	Analytical Group	Concentration Level	Number of Samples (identify field duplicates)	Sampling SOP Reference	Rationale for Sampling Location
140 samples points per FSP Table 3-3 in Appendix A	Groundwater	See FSP Table 3-3	Chemical Parameters: VOCs, SVOCs, PCB Aroclors, Pesticides, TAL Metals and Cyanide	Trace to low	140 samples plus duplicates 1 per 20	See Worksheet 21	See FSP Table 3-1 in Appendix A
58 samples points per FSP Table 3-4 in Appendix A	Groundwater	See FSP Table 3-4	Geochemical Parameters: TOC/DOC, Ferrous Iron, Anions, Dissolved Gases, Alkalinity, Hardness	low	58 samples plus duplicates 1 per 20	See Worksheet 21	See FSP Table 3-1 in Appendix A
20 samples points per FSP Table 3-4 in Appendix A	Groundwater	See FSP Tables 3-4	Microbiological Microcosm (Presence Absence test)	low	20 samples plus 1 duplicate	See Worksheet 21	See FSP Table 3-1 in Appendix A

QAPP Worksheet #18 (Continued)
(UFP-QAPP Manual Section 3.1.1)

Sampling Locations and Methods/SOP Requirements Table (Groundwater Event No. 2)

Sampling Location/ID Number	Matrix	Depth (inches)	Analytical Group	Concentration Level	Number of Samples (identify field duplicates)	Sampling SOP Reference	Rationale for Sampling Location
140 samples points per FSP Table 3-3 in Appendix A	Groundwater	See FSP Table 3-3	Chemical Parameters: VOCs, SVOCs, PCB Aroclors, Pesticides, TAL Metals and Cyanide	Trace to low	140 samples plus duplicates 1 per 20	See Worksheet 21	See FSP Table 3-1 in Appendix A
58 samples points per FSP Table 3-4 in Appendix A	Groundwater	See FSP Table 3-4	Geochemical Parameters: TOC/DOC, Ferrous Iron, Anions, Dissolved Gases, Alkalinity, Hardness	low	58 samples plus duplicates 1 per 20	See Worksheet 21	See FSP Table 3-1 in Appendix A
20 samples points per FSP Table 3-4 in Appendix A	Groundwater	See FSP Tables 3-4	Microbiological Microcosm (Presence Absence test)	low	20 samples plus 1 duplicate	See Worksheet 21	See FSP Table 3-1 in Appendix A
Per FSP the number of sample points will be based upon locations in Event 1 where PCB Aroclors were detected	Groundwater	See FSP Table 3-3	PCB Congeners And Dioxins/Furans	Trace to Low	To be determined based number of samples plus 1 duplicate per 20 samples	See Worksheet 21	See FSP Table 3-1 in Appendix A

QAPP Worksheet #18 (Continued)
(UFP-QAPP Manual Section 3.1.1)

Sampling Locations and Methods/SOP Requirements Table (Groundwater Event No. 3)

Sampling Location/ID Number	Matrix	Depth (inches)	Analytical Group	Concentration Level	Number of Samples (identify field duplicates)	Sampling SOP Reference	Rationale for Sampling Location
Per FSP the number of sample points will based upon locations in Event 1 where PCB Aroclors were detected	Groundwater	See FSP Table 3-3	PCB Congeners And Dioxins/Furans	Trace to Low	To be determined based number of samples plus 1 duplicate per 20 samples	See Worksheet 21	See FSP Table 3-1 in Appendix A

QAPP Worksheet #19
(UFP-QAPP Manual Section 3.1.1)

Analytical SOP Requirements Table

Matrix	Analytical Group	Concentration Level	Analytical and Preparation Method/SOP Reference	Sample Volume/Mass per Analysis	Containers[1] (number, size, and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/ analysis)[2]
Aqueous	Trace Concentration Volatile Organics [CLP]	Low	SOM01.2	120 ml	(3) 40 ml VOA vials w/Teflon lined septum	1:1 HCl to pH<2; cool to 4°C	10 days
Aqueous	Semi-Volatile Organics [CLP]	Low	SOM01.2	1000 ml	(2) 1L amber glass bottles w/Teflon lined cap	Cool to 4°C	5 days extract, 40 days analyze
Aqueous	Pesticide Compounds [CLP]	Low	SOM01.2	1000 ml	(2) 1L amber glass bottle w/Teflon lined cap	Cool to 4°C	5 days extract, 40 days analyze
Aqueous	PCB Compounds [CLP]	Low	SOM01.2	1000 ml	Included with Pesticides	Cool to 4°C	5 days extract, 40 days analyze
Aqueous	TAL Metals [CLP]	Low	ILM05.4	250 ml	(1) 1 L Plastic	HNO_3 to pH<2; cool to 4°C	6 months (Hg-26 days)
Aqueous	PCB Congeners	Low	CBC01.0 or 1668A	1000 mL	(2) 1L amber glass bottles w/Teflon lined cap	Cool to 4°C	One year

Analytical SOP Requirements Table

Matrix	Analytical Group	Concentration Level	Analytical and Preparation Method/SOP Reference	Sample Volume/Mass per Analysis	Containers[1] (number, size, and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/ analysis)[2]
Aqueous	Dioxins/Furans	Low	DLM02.0 or 1613B	1000 mL	(2) 1L amber glass bottles w/Teflon lined cap	Cool to 4°C	One year
Aqueous	TOC	Low	SW-846-9060A or EPA 415.1or DESA method	250 mL	250 mL Plastic	H2SO4 to pH >2, Cool to 4°C	28 days
Aqueous	DOC	Low	SW-846-9060A or EPA 415.1 or DESA method	250 mL	250 mL Plastic	Cool to 4°C And preserved with H2SO4 to pH >2	28 days
Aqueous	Nitrate, Sulfate and Chloride		SM method SW-846-9056, EPA 300.0 or equivalent DESA, (EPA 353.2 can be used for nitrate)	500 mL	500 mL Glass or Plastic	Cool to 4°C	28 days
Groundwater	Alkalinity	NA	SM2320B or equivalent or DESA method	250 mL	250 mL Plastic or Glass	Cool to 4 °C or (2 to 6° C)	14 days
Groundwater	Ferrous Iron	Low	SM -3500 Fe D or equivalent such as a DESA method	100 mL	100 mL Plastic or Glass	Test as soon as possible. (If the sample is turbidity may cause interference)	Lab must test sample as soon as possible

Analytical SOP Requirements Table

Matrix	Analytical Group	Concentration Level	Analytical and Preparation Method/SOP Reference	Sample Volume/Mass per Analysis	Containers[1] (number, size, and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/ analysis)[2]
Groundwater	Hardness	NA	SM2340C, EPA 130.1 or DESA method	250 mL	250 mL Plastic	HNO3 to pH >2, Cool to 4°C	28 days
Groundwater	Dissolved Gases including Ethane, Methane and Ethene	Low	SW-846-8015 or RSK-175 or DESA method	40 mL VOC vials	5 x 40 mL Glass vials with Teflon-lined septa	2 drops of 1:1 HCL to pH>2, no bubbles or headspace, Cool to 4 °C or (2 to 6° C)	14 days
Groundwater	De-Chlorination Bacteria	NA	BCI Microcosm Presence-Absence Test	One Liter	One Liter bottle provided by the lab	Cool to 4°C	Initiate test as soon as possible
Bedrock	Chlorinated Volatiles	Low	Stone Environmental mobile lab microwave extraction direct injection and GC-ECD method (SEI-10.17.0 and SEI-10.18.0)	Core segment approx. 1 to 2 inch long	Zip lock bag	Core is crushed as soon as possible at drilling site and approx. 15 gram transferred to 40 mL vial with 20 mL methanol (plus vial for moisture)	Test sample as soon as possible in mobile lab
Bedrock	PCB Aroclors	Low	EPA SW-846 8082 or CLP SOM01.2 or DESA method	At least 30 grams (crushed pea size rock)	8 oz jar	Cool to 4°C	14 days to extraction, 40 days to analysis

Analytical SOP Requirements Table

Matrix	Analytical Group	Concentration Level	Analytical and Preparation Method/SOP Reference	Sample Volume/Mass per Analysis	Containers[1] (number, size, and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation/ analysis)[2]
Bedrock (Core)	Physical Property Tests: % Moisture, Bulk Density (Wet and Dry), Specific Gravity (Porosity calculated value)	Low	ASTM D2216 ASTM D4531 ASTM D854	Core section of undisturbed rock weighing at least 400 g	Core section (Wrapped twice in Al foil and sealed in plastic wrap)	Room temperature	NA
Bedrock	Total Organic Carbon	Low	EPA Lloyd Kahn, ASTM D2794, or equivalent	10 grams	Crushed portion taken from Core Section in 4 oz jar	Cool to 4°C	14 days
Bedrock	Chemical Oxidation Demand	NA	ASTM D7262	Approx. 600 grams of crushed rock	Crushed portion of rock in large one quart jar or other suitable clean container	Room temperature	Test as soon as possible

1. The size and type of the sample container may differ depending upon the requirements of the assigned laboratory.
2. Requested laboratory turn-around times (TATs) for the non-CLP test methods for the majority of the requested analyses will be within 35 days of receipt of the sample.

QAPP Worksheet #20

(UFP-QAPP Manual Section 3.1.1)

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

Field Quality Control Sample Summary Table

Matrix	Analytical Group	Conc. Level	Analytical and Preparation SOP Reference[1]	Approximate No. of Sampling Locations	No. of Field Duplicate Pairs	No. of MS	No. of Trip Blanks	No. of Equip. Blanks	No. of PT Samples	Approximate Total No. of Samples to Lab
Ground-water	Trace Concentration Volatile Organics [CLP]	Low	SOM01.2	280	14 (1 per 20)	14 (1 per 20)	Estimated 40	14 (1 per 20)	0	368
Ground-water	Semi-Volatile Organics [CLP]	Low	SOM01.2	280	14 (1 per 20)	14 (1 per 20)	0	14 (1 per 20)	0	322
Ground-water	Pesticide Compounds [CLP]	Low	SOM01.2	280	14 (1 per 20)	14 (1 per 20)	0	14 (1 per 20)	0	322
Ground-water	PCB Aroclors [CLP]	Low	SOM01.2	280	14 (1 per 20)	14 (1 per 20)	0	14 (1 per 20)	1	323
Ground-water	Inorganic (TAL Metals and cyanide) [CLP]	Low	ILM05.4	280	14 (1 per 20)	14 (1 per 20)	0	14 (1 per 20)	0	322
Ground-water	PCB Congeners	Low	CBC01.0 or 1668A	Note 3	1 per 20	0	0	1 per 20	1	Estimated 70 (Note 3)

Field Quality Control Sample Summary Table

Matrix	Analytical Group	Conc. Level	Analytical and Preparation SOP Reference[1]	Approximate No. of Sampling Locations	No. of Field Duplicate Pairs	No. of MS	No. of Trip Blanks	No. of Equip. Blanks	No. of PT Samples	Approximate Total No. of Samples to Lab
Ground-water	Dioxins/ Furans	Low	DLM02.0 or 1613B	Note 3	1 per 20	0	0	1 per 20	0	Estimated 70 (Note 3)
Ground-water	TOC	Low	SW-846-9060A or DESA method	116	6 (1 per 20)	6 (1 per 20)	0	0	0	128
Ground-water	DOC	Low	SW-846-9060A or DESA method	116	6 (1 per 20)	6 (1 per 20)	0	0	0	128
Ground-water	Sulfate and Chloride Nitrate	Low	SW-846-9056, EPA 300.0 or DESA method (EPA 353.2 for nitrate)	116	6 (1 per 20)	6 (1 per 20)	0	0	0	128
Ground-water	Alkalinity	NA	SM2320B or equivalent	116	6 (1 per 20)	0	0	0	0	122
Ground-water	Ferrous Iron	Low	SM -3500 Fe D or equivalent	116	6 (1 per 20)	0	0	0	0	122
Ground-water	Hardness	Low	SM2340C or DESA method	116	6 (1 per 20)	0	0	0	0	122
Ground-water	Dissolved Gases	Low	SW-846-8015 or RSK-175	116	6 (1 per 20)	6 (1 per 20)	0	0	0	128

Field Quality Control Sample Summary Table

Matrix	Analytical Group	Conc. Level	Analytical and Preparation SOP Reference[1]	Approximate No. of Sampling Locations	No. of Field Duplicate Pairs	No. of MS	No. of Trip Blanks	No. of Equip. Blanks	No. of PT Samples	Approximate Total No. of Samples to Lab
Ground-water	Microcosm Presence-Absence	NA	BCI SOP #14 Microcosm Presence-Absence	20	1	0	0	0	0	21
Bedrock	Chlorinated Volatiles	Low	Mobile lab microwave extraction Stone Environmental GC-ECD Method (SEI-10.18.0 and SEI-10.17.0)	480	24 (1 per 20)	24 (1 per 20)	0	24 (1 per 20, wipe)	0	552
Bedrock	PCB Aroclors	Low	EPA SW-846 8082 or SOM01.2 or equivalent	240	12 (1 per 20)	0	NA	12 (1 per 20, wipe)	1	265
Bedrock	Moisture	Low	ASTM D2216	60	3 (1 per 20)	0	NA	0	0	63
Bedrock	Bulk Density (Wet and Dry)	NA	ASTM D4531	60	3 (1 per 20)	0	NA	0	0	63
Bedrock	Specific Gravity	NA	ASTM D 854	60	3 (1 per 20)	0	NA	0	0	63
Bedrock	Organic Carbon	Low	EPA Lloyd Kahn, ASTM D2794 or equivalent	60	3 (1 per 20)	0	NA	0	0	63
Bedrock	Chemical Oxidation Demand	NA	ASTM D7262	4	1	0	NA	0	0	5

1. The analytical laboratories have not been selected at this time. Methods considered as equivalent such as DESA methods will be also considered as acceptable.
2. Performance Testing (PT) samples are performed quarterly by CLP laboratories per USEPA-CLP SOW requirements. PT samples for PCB Aroclors and PCB Congeners are also planned if arrangements can be made to obtain these materials through the USEPA.
3. To be determined. The number of samples collected for PCB Congeners and Dioxin/Furans in groundwater Event 3 will be determined by the number of samples in Event 1 with the detections of PCB Aroclors.

QAPP Worksheet #21
(UFP-QAPP Manual Section 3.1.2)

Project Sampling SOP References Table

Reference Number	Title, Revision Date and/or Number	Originating Organization	Equipment Type	Modified for Project Work?	Comments
SOP 1	Procedure to Conduct Sample Management for CLP and Non-CLP Samples, August 2006, Revision No. 0	Malcolm Pirnie	Note book, personal computer, safety glasses, gloves, sample coolers, ice, sample labels etc,	No	Attachment to QAPP
SOP 2	Procedure to Conduct Sample Preservation, August 2006, Revision No. 0	Malcolm Pirnie	Ice and other sample preservatives described in the SOP etc.	No	Attachment to QAPP
SOP 3	Procedure to Conduct a Technical System Field Audit, August 2006, Revision No. 0	Malcolm Pirnie	Check list and camera	No	Attachment to QAPP
SEI-6.37.0	Field Methods for Retrieval, Collection, Handling, and Preservation of Rock Samples to be Analyzed for VOCs and Physical Properties	Stone Environmental Inc	Sample collection, processing and analysis, decontamination procedures.	No	Attachment to QAPP
SOP 5	Procedure for Collection of Equipment Blanks, August 2006, Revision No. 0	Malcolm Pirnie	Sample processing equipemt, sample bottles, preservation chemicals, ice, coolers etc.	No	Attachment to QAPP
SOP 8	Procedure for Calibration, Operation and Maintenance of the Photoinonization Detector (PID), September 2006, Revision No. 0	Malcolm Pirnie	PID Instrument	No	Attachment to FSP
SOP 9	Procedures for Decontamination	Malcolm Pirnie	Decontamination of Sampling Equipment	No	Attachment to FSP

Project Sampling SOP References Table

Reference Number	Title, Revision Date and/or Number	Originating Organization	Equipment Type	Modified for Project Work?	Comments
QAM	Quality Assurance Manual, with attachments ('SOP LCP' series) July 2008, Revision 0.	Malcolm Pirnie	Collection and analysis of groundwater 'analyze-immediately' inorganic (field) parameters	No	Attachment to FSP
SOP LCP 003	Minirae 2000 Photoionization meter	Malcolm Pirnie	PID instrumant	No	Attachment to FSP
SOP LCP 005	On-Site Turbidity Measurement of an Aqueous Sample using a Portable Turbidity Meter.	Malcolm Pirnie	Turbidity, Hach model 2100P.	No	Attachment to FSP
SOP LCP 006	On-Site pH Measurement of an Aqueous Sample using a Portable pH Meter.	Malcolm Pirnie	Oakton model 11, pH, temperature, ORP.	No	Attachment to FSP
SOP LCP 007	On-Site Oxidation-Reduction Potential Measurement of an Aqueous Sample using a Portable pH/mV Meter.	Malcolm Pirnie	Oakton model 11, pH, temperature, ORP.	No	Attachment to FSP
SOP LCP 008	On-Site Conductivity Measurement of an Aqueous Sample using a Portable Conductivity Meter.	Malcolm Pirnie	YSI model 30, temperature, salinity, conductivity.	No	Attachment to FSP
SOP LCP 009	On-Site Dissolved Oxygen Measurement of an Aqueous Sample using a Portable DO Meter.	Malcolm Pirnie	YSI model 55, dissolved oxygen.	No	Attachment to FSP
SOP LCP 010	On-Site Dissolved Oxygen Measurement of an Aqueous Sample using a Portable Multi-Parameter Meter.	Malcolm Pirnie	Horiba model U-10, pH, temperature, conductivity, turbidity, dissolved oxygen, flow through cell.	No	Attachment to FSP

Project Sampling SOP References Table

Reference Number	Title, Revision Date and/or Number	*Originating* Organization	Equipment Type	*Modified for* Project Work?	Comments
BCI SOP # 14	Microcosm Studies and Presence-Absence Tests. October 21, 2008.	Bioremediation Consultants, Inc.	Groundwater sample collection, bottles, shipping, analytical procedures.	No	Attachment to QAPP
BCI SOP # 14 Supplement	BCI Functional Test for D. ethenogenes (Presence/Absence Test). October 20, 2008	Bioremediation Consultants, Inc.	Supplement to groundwater sample collection, bottles, shipping, analytical procedures.	No	Attachment to QAPP
NJDEP GW Sampling	Groundwater Sampling Procedure Low Stress (Low Flow) Purging and Sampling, Final, October 2004	NJDEP	Low Flow Sampling Procedures	No	Attachment to FSP
OSWER 9240.0-44 USEPA540-R-07-06	Contract Laboratory Program Guidance for Field Samplers, OSWER 9240.0-44, USEPA540-R-07-06, July 2007	USEPA	Notebook, personal computer, safety glasses, gloves, sample coolers, ice, sample labels and other materials described in the document.	No	To be used by the field team as a guide for collecting and preparing CLP samples. Referenced in SOP 1
Form II Lite User's Guide	Forms II Lite, Version 5.1 User's Guide, Version 5.1	USEPA	Personal computer, printer, Forms II Lite software etc	No	Forms II Lite is required for creating chain of custody forms for USEPA CLP samples.

Project Sampling SOP References Table

Reference Number	Title, Revision Date and/or Number	Originating Organization	Equipment Type	Modified for Project Work?	Comments
Flute Guidance Documents	Sampling Guidelines For Water Flute Systems, Revised April 2007 Guide For the Use of Flute Hydraulic Conductivity Profile Results Installation Procedure For NAPL Blank Liner In Open Borehole Below the Water Table Use of Pressure Transducer With Water Flute System Subsurface Vault to Accomodate The Water Flute Surface Hardware	Flute	Flute liner	No	Attachment to FSP
D6032	ASTM D6032-02, Determining Rock Quality Designation of Rock core. ASTM D6032-02	ASTM	NA	No	Available from ASTM
D2113	Standard Practice for Rock Core Drilling and Sampling of Rock for Site Investigation. ASTM D2113-08	ASTM	Drilling Equipment	No	Available from ASTM
D5876	Use of Direct Rotary Wireline Casing advancement Drilling Methods for Geoenvironmental exploration and Installation of Subsurface Water-Quality Monitoring Devices. ASTM D5876-95	ASTM	Direct Rotary Casing	No	Available from ASTM

Project Sampling SOP References Table

Reference Number	Title, Revision Date and/or Number	Originating Organization	Equipment Type	Modified for Project Work?	Comments
D5783	Use of Direct Rotary Drilling with Water-Based Drilling Fluid for Geoenvironmental Exploration and the Installation of Subsurface Water-Quality Monitoring Devices. ASTM D5783-95	ASTM	Drilling equipment	No	Available from ASTM
Flute Guidance Documents	Sampling Guidelines For Water Flute Systesm, Revised April 2007 Guide For the Use of Flute Hydraulic Conductivity Profile Results Installation Procedure For NAPL Blank Liner In Open Borehole Below The Water Table Use of Pressure Transducer With Water Flute System Subsurface Vault to Accomodate The Water Flute Surface Hardware	Flute	Flute liner	No	Attachment to FSP
D6032	ASTM D6032-02, Determining Rock Quality Designation of Rock core. ASTM D6032-02	ASTM	NA	No	Available from ASTM
D2113	Standard Practice for Rock Core Drilling and Sampling of Rock for Site Investigation. ASTM D2113-08	ASTM	Drilling Equipement		Available from ASTM

D5876	Use of Direct Rotary Wireline Casing advancement Drilling Methods for Geoenvironmental exploration and Installation of Subsurface Water-Quality Monitoring Devices. ASTM D5876-95	ASTM	Direct Rotary Casing	No	Available from ASTM
D5783	Use of Direct Rotary Drilling with Water-Based Drilling Fluid for Geoenvironmental Exploration and the Installation of Subsurface Water-Quality Monitoring Devices. ASTM D5783-95	ASTM	Drilling equipment	No	Available from ASTM

QAPP Worksheet #22
(UFP-QAPP Manual Section 3.1.2.4)

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
Photoionization Detector (PID)	See SOP 8 in Appendix X of FSP, Procedure for Calibration, Operation and Maintenance of the Photo Inonization Detector (PID), September 2006, Revision No. 0	See SOP 8	See SOP 8	See SOP 8	See SOP 8	See SOP 8	See SOP 8	Field Team Leader	See FSP
Portable turbidity meter (Turbidity, Hach model 2100P)	See SOP LCP005	See SOP LCP005	See SOP LCP005	See SOP LCP005	See SOP LCP005	See SOP LCP005	See SOP LCP005	Field Team Leader	See FSP
Portable pH meter (Oakton model 11)	See SOP LCP006	See SOP LCP006	See SOP LCP006	See SOP LCP006	See SOP LCP006	See SOP LCP006	See SOP LCP006	Field Team Leader	See FSP
Portable pH/mV meter (Oakton model 11)	See SOP LCP007	See SOP LCP007	See SOP LCP007	See SOP LCP007	See SOP LCP007	See SOP LCP007	See SOP LCP007	Field Team Leader	See FSP

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
Portable conductivity meter (YSI model 30)	See SOP LCP008	See SOP LCP008	See SOP LCP008	See SOP LCP008	See SOP LCP008	See SOP LCP008	See SOP LCP008	Field Team Leader	See FSP
Portable dissolved oxygen meter (YSI model 55)	See SOP LCP009	See SOP LCP009	See SOP LCP009	See SOP LCP009	See SOP LCP009	See SOP LCP009	See SOP LCP009	Field Team Leader	See FSP
Horiba U-10 Field Instrument	Horiba U-10 Multi-Parameter Water Quality Monitoring System Operation Manual	Horiba U-10 Multi-Parameter Water Quality Monitoring System Operation Manual	Horiba U-10 Multi-Parameter Water Quality Monitoring System Operation Manual	Horiba U-10 Multi-Parameter Water Quality Monitoring System Operation Manual	Horiba U-10 Multi-Parameter Water Quality Monitoring System Operation Manual	Horiba U-10 Multi-Parameter Water Quality Monitoring System Operation Manual	Horiba U-10 Multi-Parameter Water Quality Monitoring System Operation Manual	Field Team Leader	See FSP

Field Instrumentation: The field team leader will be responsible for insuring that these instruments are calibrated before each field sampling event. Field equipment must be inspected and calibrated before use according to the criteria given in the Field Sampling Plan and the SOPs. If problems occur with field instruments or equipment which cannot be resolved by the field team personnel they should contact the field team leader. If field equipment fails inspection it is the field team leader's responsibility to investigate and resolve the problem. The Malcolm Pirnie equipment facility manager can also be contacted by the field crew or the field team leader to help resolve problems with field equipment and supply or obtain any spare or replacement parts or equipment.

QAPP Worksheet #23
(UFP-QAPP Manual Section 3.2.1)

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?
SOM01.2	USEPA Contract Laboratory Program Statement of Work for Multi-Media, Multi-Concentration Organic Analysis, October 2006	Definitive	Target Compound List Volatile Organics	GC/MS	Lab to be assigned - USEPA DESA or CLP lab	No
SOM01.2	USEPA Contract Laboratory Program Statement of Work for Multi-Media, Multi-Concentration Organic Analysis, October 2006	Definitive	Target Compound List Semi-Volatile Organics	GC/MS	Lab to be assigned - USEPA DESA or CLP lab	No
SOM01.2	USEPA Contract Laboratory Program Statement of Work for Multi-Media, Multi-Concentration Organic Analysis, October 2006	Definitive	Target Compound List Pesticides	GC/ECD	Lab to be assigned - USEPA DESA or CLP lab	No
SOM01.2	USEPA Contract Laboratory Program Statement of Work for Multi-Media, Multi-Concentration Organic Analysis, October 2006	Definitive	Target Compound List PCBs	GC/ECD	Lab to be assigned - USEPA DESA or CLP lab	No
ILM05.4	USEPA Contract Laboratory Program Statement of Work for Multi-Media, Multi-Concentration Inorganic Analysis, December 2006	Definitive	Target Analyte List Metals and Cyanide	ICP-AES / ICP-MS/ CVAA and Colorimetric	Lab to be assigned - USEPA DESA or CLP lab	No
DLM02.0	USEPA Contract Laboratory Program Statement of Work for Multi-Media, Multi-Concentration Dioxin and Furan Analysis or USEPA 1613B	Definitive	TCL Dioxins/Furans	GC-HRMS	Non-RAS CLP lab or subcontract lab	No
CBC01.0	USEPA Contract Laboratory Program Statement of Work for Multi-Media, Multi-Concentration Chlorinated Biphenyls Congeners Analysis or USEPA 1668A	Definitive	PCB Congeners	GC-HRMS	Non-RAS CLP lab, DESA or subcontract lab	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?
SW-846- 9060	USEPA SW-846 Method 9060, Total Organic Carbon (DOC determined by measuring TOC after filtration though a 0.45 micron filter)	Definitive	TOC/DOC	Combustion	Accutest or DESA	No
SW-846-9056 or EPA 300.0	SW-846 9056, Determination of Inorganic Anions By Ion Chromatography	Definitive	Sulfate , Nitrate and Chloride	Ion chromatograph	Accutest or DESA	No
EPA 353.2 (Alternate for Nitrate)	EPA 353.2, Nitrogen, Nitrate-Nitrite (Colorimetric, Automated, Cadmium Reduction)	Definitive	Nitrate	Colorimetric	Accutest or DESA	No
SM2320B	SM2320B Alkalinity by Titration	Definitive	Alkalinity	Titrimeter	Accutest or DESA	No
SM3500 Fe D	SM3500 Fe D for Ferrous Ion	Definative	Ferrous Iron	Spectrophotometer	Accutest or DESA	
Hardness	SM 2340C Total Hardness by Spectrophotometer	Definitive	Hardness	Spectrophotometer	Accutest or DESA	No
SW-846-8015A/ USEPA RSK-175	SW-846-8015A, Non –halogenated Organics by GC/FID *Or equivalent method similar to the RSK-175, based upon US EPA Region 1, Technical Guidance for the Natural Attenuation Indicators: Methane, Ethane, and Ethene, Revision 1, 2/21/02,*	Screening	Ethane, Methane and Ethene	Gas chromatograph-flame Ionization Detector (GC/FID)	Accutest or DESA	No
Microcosm Presence-Absence Test	Bioemediation Consulting, Inc., Standard Operating Procedure #14 Microcosm Studies and Presence-Absence tests	Screening	Microbial Presence-Absence Test	Microcosm bottles and associates test equipment	Bioemediation Consulting, Inc.	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?
SEI-10.18.0 and SEI-10.17.0	The determination of Volatile Organic Compounds By Gas Chromatography/Dual ECD Detectors In Rock Samples (Using Cool On column Injection and split Method Injection) Microwave Assisted Extraction of Volatile Organic Compounds From Rock	Definitive	Chlorinated Volatiles	GC-ECD	Stone Environmental Inc	No
SOM01.2 or SW-845-8082	SW-846-8082 or equivalent method such as USEPA Contract Laboratory Program Statement of Work for Multi-Media, Multi-Concentration Organic Analysis, October 2006	Definitive	PCB Aroclors	GC/ECD	DESA and or assigned CLP lab or Accutest	Not for Groundwater, but rock matrix may require additional crushing
D7263	ASTM D7263, Standard test Method for Estimating of the Permanganate Natural Oxidant Demand of Soil and Aquifer Solids	Screening	Soil Oxidant Demand	Colorimetric	Redox Tech or other assigned lab	No
Bedrock Physical Properties:						
TOC (Bedrock)	USEPA Lloyd Kahn, ASTM D2974 or equivalent	Screening	Organic Carbon Content	Combustion	Accutest, DESA or orther assigned subcontract lab	No
% Moisture	ASTM D2216, Standard Test method for Laboratory Determination of Water Content of Soil and Rock by Mass	Screening	Moisture	Drying oven and balance	Golder Associates or other assigned lab	No
Specific Gravity	ASTM D854 Standard Test Method for Specific Gravity of Soil Solid by Water Pycnometer (Note 1)	Screening	Specific Gravity	Water Pycnometer	Golder Associates or other assigned lab	No
Bulk Density	ASTM D4531, Standard Test Methods for Bulk Density	Screening	Bulk Density	Paraffin-wax displacement	Golder Associates or other assigned lab	No
Porosity	The Geotech lab will calculate from the unit weight, gravity and water content obtained for the above tests	Screening	Porosity	NA	Golder Associates or other assigned lab	Calculated value

Analytical Methods for Groundwater

We anticipate for this project that a large number of the chemical analyses will be performed by a USEPA CLP Lab and the USEPA Region 2 DESA laboratory and that subcontract commercial laboratories such as Stone Environmental and Accutest will also be utilized. If a subcontract laboratory is used for chemical analyses, it must be qualified in the analytical methods and, where applicable, certified through the programs such as: National Environmental Laboratory Accreditation Program (NELAP), USEPA CLP or self declared as compliment with the applicable requirements of the Department of Defense (DoD) Quality Systems Manual (QSM) For Environmental Laboratories.

Any subcontract laboratory utilized for the project will undergo an evaluation to determine if their experience and capability in the requested analytical methods are appropriate for the project. When possible the test methods selected must either be USEPA methods or national consensus methods, such as those published by ASTM.

QAPP Worksheet #24
(UFP-QAPP Manual Section 3.2.2)

Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
Gas Chromatograph with Electron Capture Detector (GC-ECD)	See USEPA CLP SOW SOM01.2 or SW-846 method or Stone Environmental SOP SEI-10.18.0	See USEPA CLP SOW or SW-846 method or Stone Environmental SOP SEI-10.18.0	See USEPA CLP SOW or SW-846 method or Stone Environmental SOP SEI-10.18.0	See USEPA CLP SOW or SW-846 method or Stone Environmental SOP SEI-10.18.0	Assigned Lab personnel or SW-846 method or Stone Environmental SOP SEI-10.18.0	SOM01.2 or SW-846 method 8082 or Stone Environmental SOP SEI-10.18.0
GC-MS	See USEPA CLP SOW SOM01.2	See USEPA CLP SOW	See USEPA CLP SOW	See USEPA CLP SOW	Assigned CLP Lab personnel	SOM01.2
ICP-AES, ICP-MS and Cold Vapor AA	See USEPA CLP SOW ILM05.4	See USEPA CLP SOW	See USEPA CLP SOW	See USEPA CLP SOW	Assigned CLP Lab personnel	ILM05.4
GC-High Resolution Mass Spectrometry	See USEPA CLP SOWs DLM02.0 USEPA 1613B) and CBC01.0 (USEPA 1668A) and assigned lab QA manuals	See USEPA CLP SOW	See USEPA CLP SOW	See USEPA CLP SOW	Assigned CLP Lab personnel	DLM02.0 (USEPA 1613B) and CBC01.0 (USEPA 1668A)
Ion chromatograph	Per SW-846-9056 or EPA 300.0	Daily	Per SW-846-9056 /EPA 300.0	Correct and Re-calibrate	Assigned Lab personnel	Per SW-846-9056, EPA 300.0
GC-ECD (Mobile Lab-Chlorinated volatiles screening)	Per Stone Environmental SEI-10.18.0	Prior to analysis	Per Stone Environmental SEI-10.18.0	Correct and Re-calibrate	Mobile Lab Personnel	Per Stone Environmental SEI-10.18.0
Spectrometer	Per EPA and SM methods and assigned lab SOPs	Check Daily	Per EPA method	Correct and Re-calibrate	Assigned Lab personnel	Per EPA and SM method
Titration Equipment	Per EPA and SM methods and assigned lab SOPs	Check Daily	Per EPA method	Correct and Re-calibrate	Assigned Lab personnel	Per EPA and SM method

QAPP Worksheet #25
(UFP-QAPP Manual Section 3.2.3)

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
Gas Chromatograph with Electron Capture Detector (GC-ECD)	Per instrument manual and USEPA CLP SOM01.2[ab]	See USEPA CLP SOM01.2	See USEPA CLP SOM01.2	See USEPA CLP SOM01.2	See USEPA CLP SOM01.2	See USEPA CLP SOM01.2	Assigned lab personnel	See USEPA CLP SOM01.2
GC-MS	Per instrument manual and USEPA CLP SOM01.2[ab]	See USEPA CLP SOM01.2	See USEPA CLP SOM01.2	See USEPA CLP SOM01.2	See USEPA CLP SOM01.2	See USEPA CLP SOM01.2	Assigned lab personnel	See USEPA CLP SOM01.2
ICP-AES, ICP-MS and Cold Vapor AA	Per instrument manual and USEPA CLP ILM05.4[ab]	See USEPA CLP ILM05.4	See USEPA CLP ILM05.4	See USEPA CLP ILM05.4	See USEPA CLP ILM05.4	See USEPA CLP ILM05.4	Assigned lab personnel	See USEPA CLP ILM05.4
GC-High Resolution Mass Spectrometry	Per instrument manual and USEPA CLP DLM02.0 and CBC01.0[ab]	See USEPA DLM02.0 and CBC01.0	See USEPA DLM02.0 and CBC01.0	See USEPA DLM02.0 and CBC01.0	See USEPA DLM02.0 and CBC01.0	See USEPA DLM02.0 and CBC01.0	Assigned lab personnel	See USEPA DLM02.0 and CBC01.0
Ion chromatograph	Per instrument manual, USEPA or SM method and assigned lab SOP	See EPA or SM method per Worksheet 24	See EPA or SM method per Worksheet 24	See EPA or SM method per Worksheet 24	See EPA or SM method per Worksheet 24	Investigate and re-calibrate if necessary	Assigned lab personnel	EPA or SM method
GC-ECD (Mobile Lab)	Per instrument manual and assigned lab SOP	Stone Environmental SOP SEI 10.18.0	Calibration verification sample	Prior to analysis and Every 40 samples	Calibration verification +/- 15%	Investigate and re-calibrate if necessary	Assigned mobile lab personnel	SEI 10.18.0

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
Spectrometer	Per instrument manual and assigned lab SOP	See EPA or SM method per Worksheet 24	See EPA or SM method per Worksheet 24	See EPA or SM method per Worksheet 24	See EPA or SM method per Worksheet 24	Investigate and re-calibrate if necessary	Assigned lab personnel	EPA or SM method per Worksheet 24
Titration Equipment	Per instrument manual and assigned lab SOP	See EPA or SM method per Worksheet 24	See EPA or SM method per Worksheet 24	See EPA or SM method per Worksheet 24	See EPA or SM method per Worksheet 24	Investigate and re-calibrate if necessary	Assigned lab personnel	EPA or SM method per Worksheet 24

a. The maintenance of the analytical instruments including the testing activity, inspection activity, frequency, acceptance criteria, responsible person and SOP reference must be documented in the Laboratory's Quality Control Manual. It is anticipated that the samples will be analyzed through the USEPA CLP program, which has developed detailed Statements of Work referenced in Worksheet #23. There is also possibility that some of the analyses will be assigned by USEPA Region 2 for analysis by the USEPA Region 2 DESA Lab. If the DESA lab or a contract lab performs analyses, the associated instrumentation must be maintained according to their SOPs and Laboratory Quality Manual.
b. Spare parts and maintenance of laboratory analytical instrumentation are the responsibility of the assigned USEPA CLP laboratory, the DESA laboratory or the subcontract laboratory.

QAPP Worksheet #26
(UFP-QAPP Manual Appendix A)

Sample Handling System

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): Malcolm Pirnie Field Team supervised by the Field Team Leader will collect the samples.
Sample Packaging (Personnel/Organization): Malcolm Pirnie Field Team
Coordination of Shipment (Personnel/Organization): Malcolm Pirnie Sample Management Officer
Type of Shipment/Carrier: Federal Express for Overnight Delivery or courier to the laboratory
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Assigned USEPA CLP laboratory, the USEPA Region 2 DESA laboratory or subcontract lab personnel
Sample Custody and Storage (Personnel/Organization): Assigned USEPA CLP laboratory, the USEPA Region 2 DESA laboratory or subcontract lab personnel
Sample Preparation (Personnel/Organization): Assigned USEPA CLP laboratory, the USEPA Region 2 DESA laboratory or subcontract lab personnel
Sample Determinative Analysis (Personnel/Organization): Assigned USEPA CLP laboratory, the USEPA Region 2 DESA laboratory or subcontract lab personnel
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): Samples will not be stored in the field, but will be shipped within 24 hours of collection. If due to an emergency they are stored in the field, they will be kept in a cooler or transferred to a refrigerator kept at 4 degrees C.
Sample Extract/Digestate Storage (No. of days from extraction/digestion): Sample extraction and digestion will be conducted according to the USEPA-CLP SOWs and the requirements given in Worksheet 19.
Biological Sample Storage (No. of days from sample collection): NA
SAMPLE DISPOSAL
Personnel/Organization: Assigned USEPA DESA or CLP Lab or Subcontract Lab Sample Custodians
Number of Days from Analysis: At least 60 days

Sample Handling System

Sample handling and custody procedures ensure the timely, correct, and complete analysis of each sample for all parameters requested. A sample is considered to be in someone's custody if it:

- Is in his/her possession.
- Is in his/her view, after being in his/her possession.
- Is in his/her possession and has been placed in a secure location.
- Is in a designated secure area.

Sample custody documentation provides a written record of sample collection and analysis. The sample custody procedures provide for specific identification of samples associated with an exact location, the recording of pertinent information associated with the sample, including time of sample collection and any preservation techniques, and a Chain of Custody (COC) record which serves as physical evidence of sample custody. Custody procedures will be similar to the procedures outlined in the USACE's Requirements for the Preparation of Sampling and Analysis Plans (USACE, 2001) and the USEPA's Contract Laboratory Program Guidance for Field Samplers (USEPA, 2007). The COC documentation system provides the means to individually identify, track, and monitor each sample from the time of collection through final data reporting. Sample custody procedures are developed in three areas: sample collection, laboratory analysis, and final evidence files, which are described below. See Attachment 9 for a copy of SOP No. HW-32, Implementing the National Strategy for Procuring Analytical Services for All OSWER Programs. It includes the Region 2 requirements for obtaining analytical services for Superfund projects and outlines the arrangements that must be made through the Region 2 Regional Sample Control Coordinator (RSCC) and gives example forms that must be submitted.

Field Sample Handling and Custody

Field records provide a means of recording information for each field activity performed at the Site. COC procedures document pertinent sampling data and all transfers of custody until the samples reach the analytical laboratory. The sample packaging and shipment procedures summarized below will ensure that the samples arrive at the laboratory with the COC intact. Refer to SOP No. 1

in Attachment 2 for sample management information. Worksheet 19 lists the specific sample preservation requirements for each test method.

Field Procedures

The general responsibilities of the field team are listed below:

- The field sampler is personally responsible for the care and custody of the samples until they are transferred to the Sample Management Officer (SMO) or until they are properly dispatched. As few people as possible should handle the samples.
- The Field Team Leader, or designee, is responsible for entering the proper information in the field logbook, including all pertinent information such as sample identification number, date and time of sample collection, type of analysis, and description of sample location. The information entered into the field logbook will be used to generate a COC.
- All sample containers will be labeled with the project identification, sample number, matrix, type of analysis required, and preservation requirements.
- The samples will be properly preserved, bagged, and packed into coolers. The original COC form will be placed into the lead cooler and will be shipped to the laboratory.
- The SMO or designee will review all field activities to determine whether proper custody procedures were followed during the field work and if additional samples are required.

Field Records

The field logbook will provide the means of recording data collection activities. Entries will be described in as much detail as possible so that persons going to the Site can reconstruct a particular situation without reliance on memory. At the beginning of each day, the date, start time, weather, and names of all sampling team members present will be entered. The names of visitors to the Site and the purpose of their visit will also be recorded. All field measurements, as well as the instrument(s), will be noted.

Samples will be collected following the sampling procedures documented in the FSP. Observations such as sampling conditions or any problems will also be recorded. Sample identification numbers will be assigned at the time the data are entered in the logbook. Field duplicate samples for soil samples, which will receive a unique sample identification number, are "blind" to the laboratory and will be identified under the sample description so that they can be associated with their respective samples by project staff. Matrix Spike/Matrix Spike Duplicate (MS/MSD) samples for soils will not be noted in the logbook or COC forms as they will be collected after homogenization of the soil samples in the analytical laboratory. In the case of dust samples, at least one sample collected per day should have a mass of 30 grams or more to allow for analysis of one MS and one MSD per analytical batch. If necessary, the sampling area will be expanded to collect additional material. It will be noted in the field log book where a larger area has been sampled and the amount of material obtained appears to be sufficient for analysis of MS/MSD samples.

Sample Identification System

All samples collected from the Site must be identified with a sample label in addition to an entry on a COC record. Indelible ink will be used to complete sample labels and handwritten COC records. COCs for all CLP samples must be generated using Forms II Lite software. Forms II Lite can also be used to create COCs for non-CLP samples. CLP Samples will be assigned a CLP sample number by the Forms II Lite software. In addition, each groundwater and bedrock sample shall be identified by a unique sample number assigned by the field team as described in the FSP. The unique sample number will include a sequential sample number, the Well location ID, and the type of sample and the depth of the sample if it is a bedrock sample.

Sample Labels/Tags

Sample labels will require the field team to complete the following information for each sample container:

1. Sample Number.
2. In the case of CLP samples, the label must also list the CLP Number and the CLP Case Number.
3. Sample Matrix.
4. Parameters to be analyzed.

5. Date of Collection.
6. Time of Collection.
7. Preservation Technique Employed (if applicable).
8. Sampler's Name.

QAPP Worksheet #27
(UFP-QAPP Manual Section 3.3.3)

Sample Custody Requirements

Field Sample Custody Procedures (sample collection, packaging, shipment, and delivery to laboratory): Samples will be collected per the procedures described in the FSP (given in Appendix A). The field sample custody procedures including sample packing, shipment, and delivery requirements are discussed in the text in Worksheets 17 and 26. Also refer to SOP No. 1 in Attachment 2 and the CLP Guidance for Field Samplers for sample management information.
Laboratory Sample Custody Procedures (receipt of samples, archiving, disposal): Each laboratory will have a sample custodian who accepts custody of the samples and verifies that the information on the sample labels matches the information on the COC. The sample custodian will document any discrepancies and will sign and date all appropriate receiving documents. The sample custodian will also document the condition of the samples upon receipt at the laboratory. The laboratory sample custody procedures ware discussed further in the following text.
Sample Identification Procedures: The sample identification scheme that will be employed is described in the FSP. Sample labeling procedures are discussed in the text in Worksheet 26.
Chain-of-Custody (COC) Procedures: A COC record will accompany the samples from the time of sampling through all transfers of custody. Example COC forms are presented in Attachments 3.1 and 3.2. The COC procedures are detailed in the following text and in SOP No. 1 in Attachment 2, and for CLP samples, in the USEPA CLP Guidance for Field Samplers.

Sample Custody Requirements:

Chain of Custody Procedure

When samples are being submitted to an USEPA-CLP laboratory, the COC must be generated using FORMS II Lite software. Each CLP sample will have a CLP number assigned in addition to the field sample identification number. The following information is typically recorded on manual COC forms. All COC forms must be signed in ink:

- Project name and/or project number.
- Signature of SMO or designee.
- Sampling station number.
- Date and time of collection.

- Grab or composite sample designation.
- Sample matrix.
- Sampling location description.
- Field identification number.
- Analyses required.
- Preservation technique.
- Signatures and dates for transfers of custody.
- Air express/shipper's bill of lading identification numbers.

The COC form serves as an official communication to the laboratory detailing the particular analyses required for each sample. The COC record will accompany the samples from the time of sampling through all transfers of custody. It will be kept on file at the laboratory where samples are analyzed and archived. Three copies of the COC form are created; one copy is retained by the Field Team Leader and two are sent to the laboratory. An electronic copy of each COC should be also made and kept in the project directory. The SMO or designee completes a COC record to accompany each shipment from the field to the laboratory. In the case of CLP samples a copy of the COC created by Forms II Lite must be sent to the USEPA CLP Region 2 coordinator.

The completed COC is put in a zip-lock bag and taped to the inside cover of the sample shipping container. If there is more than one container in a shipment, copies of the COC form will be placed in each container. Each container is then sealed with custody seals and custody is transferred to the laboratory.

Transfer of Custody and Shipment

The custody of samples must be maintained from the time of sampling through shipment and relinquishment to the laboratory. Instructions for transferring custody are given below:

- All samples are accompanied by a COC. When transferring custody of samples, the individuals relinquishing and receiving will sign, date, and note the time on the COC. This form documents sample custody transfer from the SMO or designee, through the shipper, to the analytical laboratory. Since a common carrier will usually not accept responsibility for handling COC forms, the name of the carrier is entered under "Received by," the bill-of-lading number is recorded in the comments section, and the COC form is placed in a zip-lock plastic bag and taped to the inside lid of the lead shipping cooler. Copies of the COC form will be placed in each additional cooler in a shipment.

- Samples will be packaged for shipment and either picked up at the former CDE facility by the laboratory or dispatched to the appropriate laboratory via overnight delivery service. SOP No. 1 in Attachment 2 contains the proper sample packaging techniques. A separate COC record must accompany each shipment. Shipping containers will be sealed for shipment to the laboratory. Two custody seals will be applied to each cooler to document that the container was properly sealed and to determine if the container was tampered with during shipment. The custody seals will be placed on the coolers in such a manner that the custody seal would be broken if the cooler were opened (*i.e.*, diagonally opposite corners of the cooler lid).

- The original COC (and a copy for CLP laboratories) will accompany the shipment. A copy will be retained by the Field Team Leader.

- If the samples are sent by common carrier or air freight, proper documentation must be maintained. For example, the bill of lading must be retained by the Field Team Leader.

Laboratory Custody Procedures

The laboratory custody procedures will be equivalent to those described in the latest edition of the CLP SOW. The following will be addressed in the laboratory custody SOPs:

- A designated sample custodian accepts custody of the samples and verifies that the information on the sample labels matches the information on the COC. The sample custodian will document any discrepancies and will sign and date all appropriate

receiving documents. The sample custodian will also document the condition of the samples upon receipt at the laboratory. The CLP laboratories will send a copy of the sample receipt checklist to USEPA's RSCC, while the subcontract laboratories will complete the form and return it electronically.

- Once the samples have been accepted by the laboratory, checked and logged in, they must be maintained in accordance with laboratory custody and security requirements.
- To ensure traceability of samples while in the possession of the laboratory, a method for sample identification that has been documented in a laboratory SOP will be used to assign sample numbers.
- The following stages of analysis must be documented by the laboratory:
    - Sample Extraction/Preparation.
    - Sample Analysis.
    - Data Reduction.
    - Data Reporting.
- Laboratory personnel are responsible for the custody of samples until they are returned to the sample custodian.
- When sample analyses and QA checks have been completed in the laboratory, the used portion of the sample must be stored or disposed of in accordance with the protocols specified in the CLP SOW or the subcontract agreement. Identifying labels, data sheets, COCs, and laboratory records will be retained until analyses and QA checks are completed in accordance with the protocols specified in the CLP SOW or the subcontract agreement.

Final Evidence Files

This is the final phase of sample custody. The COC records and sample analysis request form copies are archived in their respective project files. Laboratory custody forms, sample preparation and analysis logbooks, and data packages will become part of the laboratory final evidence file. Other relevant documentation including records, reports, and correspondence, logs, pictures, and data

review reports will be archived by Malcolm Pirnie.

Sample Holding Times

Information on sample holding times and required preservation for each test method are provided in Worksheet 19.

Sample Packaging and Shipping Requirements

- Custody of samples must be maintained through the shipment of samples to the selected laboratory. All samples will be packaged and shipped at the end of each day unless other arrangements are made with the laboratory. See SOP 1 in Attachment 2 for a description of the Sample Management and Custody Procedures, as well as sample packing. Also refer to the USEPA CLP Guidance for Field Samplers for more details regarding shipment of CLP samples.

QAPP Worksheet #28
(UFP-QAPP Manual Section 3.4)

QC Samples Table

Matrix	Groundwater
Analytical Group	TCL Dioxins/Furans
Concentration Level	Low
Sampling SOP	See Worksheet 20 and FSP Appendix A
Analytical Method/ SOP Reference	DLM02.0 or EPA 1613B
Sampler's Name	Malcolm Pirnie Field Sampling Crew
Field Sampling Organization	Malcolm Pirnie Inc
Analytical Organization	Assigned EPA CLP Lab
No. of Sample Locations	See Worksheet 18 and FSP Tables

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QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Equipment Blank	At least once a week but no more than once a day	QAPP	Investigate source of contamination	Malcolm Pirnie Field Team Leader	Sensitivity	<QL
Field Duplicates	1 per 20 field samples per matrix	FSP and QAPP	If the limits exceed limits for the field replicate, this will be addressed by the Malcolm Pirnie Data Reviewer	Malcolm Pirnie Field Team Leader and Laboratory	Precision	RPD 35% for duplicate values greater than or equal to 5 times the CRQL
Initial Calibration	At initial set up or when corrective is taken which may change calibration per DLM02.2	Per DLM02.0 or EPA 1613B	Per DLM02.0 or EPA 1613B	Assigned lab	Accuracy	Per DLM02.0 or EPA 1613B

QAPP Worksheet #28
(UFP-QAPP Manual Section 3.4)

QC Samples Table

Matrix	Groundwater
Analytical Group	TCL Dioxins/Furans
Concentration Level	Low
Sampling SOP	See Worksheet 20 and FSP Appendix A
Analytical Method/ SOP Reference	DLM02.0 or EPA 1613B
Sampler's Name	Malcolm Pirnie Field Sampling Crew
Field Sampling Organization	Malcolm Pirnie Inc
Analytical Organization	Assigned EPA CLP Lab
No. of Sample Locations	See Worksheet 18 and FSP Tables

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Calibration Verification	Prior to every 12 hr. period, but following Column Performance Solution and at end of 12 hr. period per DLM02.0	Per DLM02.0 or EPA 1613B	Per DLM02.0 or EPA 1613B	Assigned lab	Accuracy	Per DLM02.0 or EPA 1613B
Laboratory Control Sample	Each group of 20 samples or less prior or analysis of samples	Per DLM02.0 or EPA 1613B	3 compounds may fail. Frequent failures must be investigated per DLM02.2	Assigned lab	Accuracy	Acceptance Limits in Table 6 ofDLM02.0
Method Blanks	Each time samples are extracted	Per DLM02.0 or EPA 1613B	Per DLM02.0 or EPA 1613B	Assigned lab	Sensitivity	Per DLM02.0 or EPA 1613B or EPA 1613B
Method Detection Limits	Annually	Per DLM02.0 or EPA 1613B	Per DLM02.0 or EPA 1613B	Assigned lab	Sensitivity	Per DLM02.0 or EPA 1613B
The assigned laboratory also must perform and meet all the measurement performance criteria which assess the analytical DQIs specified in EPA CLP SOW DLM02.0 (based upon EPA 1613B) including initial and ongoing studies to demonstrate precision and accuracy, and calibration verification, internal standards added to all extracts, cleanup standards and laboratory control standards for accuracy, and blanks and method detection limits for sensitivity. The lab personnel must follow all the corrective actions required by the EPA CLP SOW or EPA 1613B .						

Note: The laboratory must meet all the QA requirements in CLP SOW DLM02.0 and EPA 1613B.

QAPP Worksheet #28
(UFP-QAPP Manual Section 3.4)

QC Samples Table

Matrix	Groundwater
Analytical Group	PCB Congeners
Concentration Level	Low
Sampling SOP	See Worksheet 20 and FSP Appendix A
Analytical Method/ SOP Reference	CBC01.0 or EPA 1668A
Sampler's Name	Malcolm Pirnie Field Sampling Crew
Field Sampling Organization	Malcolm Pirnie Inc
Analytical Organization	Assigned EPA CLP Lab
No. of Sample Locations	See Worksheet 18 and FSP Tables

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Equipment Blank	At least once a week but no more than once a day	QAPP	Investigate source of contamination	Malcolm Pirnie Field Team Leader	Sensitivity	<QL
Field Duplicates	1 per 20 field samples per matrix	FSP and QAPP	If the limits exceed limits for the field replicate, this will be addressed by the Malcolm Pirnie Data Reviewer	Malcolm Pirnie Field Team Leader and Laboratory	Precision	RPD 35% for duplicate values greater than or equal to 5 times the CRQL
Initial Calibration	At initial set up or when corrective is taken which may change calibration per CBC01.0 or EPA 1668A	Per CBC01.0 or EPA 1668A	Per CBC01.0 or EPA 1668A	Assigned lab	Accuracy	Per CBC01.0 or EPA 1668A
Calibration Verification	Prior to every 12 hr. period, but following Column Performance Solution and at end of 12 hr. period per CBC01.0 or EPA 1668A	Per CBC01.0 or EPA 1668A	Per CBC01.0 or EPA 1668A	Assigned lab	Accuracy	Per CBC01.0 or EPA 1668A

QAPP Worksheet #28
(UFP-QAPP Manual Section 3.4)

QC Samples Table

Matrix	Groundwater
Analytical Group	PCB Congeners
Concentration Level	Low
Sampling SOP	See Worksheet 20 and FSP Appendix A
Analytical Method/ SOP Reference	CBC01.0 or EPA 1668A
Sampler's Name	Malcolm Pirnie Field Sampling Crew
Field Sampling Organization	Malcolm Pirnie Inc
Analytical Organization	Assigned EPA CLP Lab
No. of Sample Locations	See Worksheet 18 and FSP Tables

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Laboratory Control Sample	Each group of 20 samples or less prior or analysis of samples	Per CBC01.0 or EPA 1668A	3 compounds may fail. Frequent failures must be investigated per CBC01.0 or EPA 1668A	Assigned lab	Accuracy	Acceptance Limits in Table 6 of CBC01.0 or EPA 1668A
Method Blanks	Each time samples are extracted	Per CBC01.0 or EPA 1668A	Per CBC01.0 or EPA 1668A	Assigned lab	Sensitivity	Per CBC01.0 or EPA 1668A
Method Detection Limits	Annually	Per CBC01.0 or EPA 1668A	Per CBC01.0 or EPA 1668A	Assigned lab	Sensitivity	Per CBC01.0 or EPA 1668A
The assigned laboratory also must perform and meet all the measurement performance criteria which assess the analytical DQIs specified in EPA CLP SOW CBC01.0 or EPA 1668A including initial and ongoing studies to demonstrate precision and accuracy, and calibration verification, internal standards added to all extracts, cleanup standards and laboratory control standards for accuracy, and blanks and method detection limits for sensitivity. The lab personnel must follow all the corrective actions required by the EPA CLP SOW.						

Note: The laboratory must meet all the QA requirements in CLP SOW CBC01.0 or EPA 1668A.

QAPP Worksheet #28
(UFP-QAPP Manual Section 3.4)

QC Samples Table

Matrix	Groundwater
Analytical Group	TCL Volatiles
Concentration Level	Trace
Sampling SOP	See Worksheet 20 and FSP Appendix A
Analytical Method/ SOP Reference	SOM01.2
Sampler's Name	Malcolm Pirnie Field Sampling Crew
Field Sampling Organization	Malcolm Pirnie Inc
Analytical Organization	Assigned EPA CLP Lab
No. of Sample Locations	See Worksheet 18 and FSP Tables

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Equipment Blank	At least once a week but no more than once a day	QAPP	Investigate source of contamination	Malcolm Pirnie Field Team Leader	Sensitivity	< QL
Field Duplicates	1 per 20 field samples	QAPP	If the limits exceed limits for the field replicate, this will be addressed by the Malcolm Pirnie Data Reviewer	Malcolm Pirnie Field Team Leader and or Laboratory	Precision	RPD < 30% for duplicate and 405 for soilds values greater than or equal to 5 times the CRQL
Matrix Spike/Matrix Spike Duplicates (MS/MSD)	Each group of field samples in an SDG, or each SDG, whichever is most frequent per SOM01.2	Per SOM01.2	Per SOM01.2, reanalyze samples	Assigned lab	Accuracy/Bias and Precision	Recoveries and %RPD per SOM01.2, Exhibit D Table 6 for criteria
Deuterated Monitoring Compounds	Each sample, standard, blank	Recoveries per SOM01.2	Per SOM01.2, correct, re-prepare and re-analyze	Assigned lab	Accuracy/Bias	Recoveries per SOM01.2, Exhibit D, Table 5 for criteria

QC Samples Table

Matrix	Groundwater
Analytical Group	TCL Volatiles
Concentration Level	Trace
Sampling SOP	See Worksheet 20 and FSP Appendix A
Analytical Method/ SOP Reference	SOM01.2
Sampler's Name	Malcolm Pirnie Field Sampling Crew
Field Sampling Organization	Malcolm Pirnie Inc
Analytical Organization	Assigned EPA CLP Lab
No. of Sample Locations	See Worksheet 18 and FSP Tables

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	Once every 12 hour for each instrument per SOM01.2	Per SOM01.2	Per SOM01.2, correct problems and reanalyze	Assigned lab	Sensitivity	less the CRQLs Reference USEPA Region 2 SOP No. 34/Trace VOA – Blank Type Criteria Table
Method Detection Limits	Annual	Per SOM01.2	Per SOM01.2, correct problem and determine detection limits	Assigned lab	Sensitivity	Per SOM01.2, Low enough to support CRQLs
The assigned laboratory also must perform the QC/QA sample analyses and meet all the measurement performance criteria which assess the analytical DQIs specified in EPA CLP SOW SOM01.2 such as laboratory duplicates and matrix spike duplicates for precision, matrix spikes, Deuterated monitoring compounds for accuracy, and blanks and method detection limits for sensitivity. The lab personnel must follow all the corrective actions required by the EPA CLP SOW.						

QAPP Worksheet #28
(UFP-QAPP Manual Section 3.4)

QC Samples Table

Matrix	Bedrock
Analytical Group	Chlorinated Volatiles
Concentration Level	Low
Sampling SOP	See FSP and Stone Environmental Inc.SEI-6.36.0
Analytical Method/ SOP Reference	Stone Environmental Inc. SOPs SEI-10.18.0 and SEI-10.17.0
Sampler's Name	Malcolm Pirnie and Stone Enviornmental Field Crew
Field Sampling Organization	Malcolm Pirnie and Stone Environmental Inc.
Analytical Organization	Stone Environmental Inc. (Mobile Laboraotry)
No. of Sample Locations	See Worksheet 18 and FSP Tables

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Equipment Blank	Daily	QAPP		Analyst	Accuracy	Less than QLs
Fortified Blank	Every 40 samples	SEI-10.18.0	Investigate problem and correct	Analyst	Accuracy	Less than QLs
Laboratory Duplicate	Every 20 samples	SEI-10.18.0	Investigate problem and correct	Analyst	Precision	RPD 15%
Initial multiple point calibration	Prior to analyzing samples	SEI-10.18.0	Investigate and Re-calibrate	Analyst	Accuracy	Correlation coefficient of 0.995 or grater
Initial Calibration Verification	Immediately follwing calibration curves	SEI-10.18.0	Investigate and Re-calibrate	Analyst	Accuracy	Recovery ±20%

QC Samples Table

Matrix	Bedrock
Analytical Group	Chlorinated Volatiles
Concentration Level	Low
Sampling SOP	See FSP and Stone Environmental Inc.SEI-6.36.0
Analytical Method/ SOP Reference	Stone Environmental Inc. SOPs SEI-10.18.0 and SEI-10.17.0
Sampler's Name	Malcolm Pirnie and Stone Enviornmental Field Crew
Field Sampling Organization	Malcolm Pirnie and Stone Environmental Inc.
Analytical Organization	Stone Environmental Inc. (Mobile Laboraotry)
No. of Sample Locations	See Worksheet 18 and FSP Tables

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Laboratory Control Standard	Every 40 samples	SEI-10.18.0	Investigate problem and correct	Analyst	Accuracy	Recovery 80-120%
Matrix Spike	Initially and at least every 40 samples	SEI-10.18.0	Investigate problem, report to Project Manager and plan corrective action	Analyst	Accuracy	Recovery 70-130%

QAPP Worksheet #28
(UFP-QAPP Manual Section 3.4)

QC Samples Table

Matrix	Groundwater
Analytical Group	TCL SVOC
Concentration Level	Low
Sampling SOP	See Worksheet 20 and FSP Appendix A
Analytical Method/ SOP Reference	SOM01.2
Sampler's Name	Malcolm Pirnie Field Sampling Crew
Field Sampling Organization	Malcolm Pirnie Inc
Analytical Organization	Assigned EPA CLP Lab
No. of Sample Locations	See Worksheet 18 and FSP Tables

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Equipment Blank	At least once a week but no more than once a day	QAPP	Investigate source of contamination	Malcolm Pirnie Field Team Leader	Sensitivity	< QL
Field Duplicates	1 per 20 field samples	FSP Addendum No. 1 and QAPP	If the limits exceed limits for the field replicate, this will be addressed by the Malcolm Pirnie Data Reviewer	Malcolm Pirnie Field Team Leader and or Laboratory	Precision	RPD < 30% for duplicate values greater than or equal to 5 times the CRQL
Matrix Spike/Matrix Spike Duplicates (MS/MSD)	Each group of field samples in an SDG, or each SDG, whichever is most frequent per SOM01.2	Per SOM01.2	Per SOM01.2, reanalyze the samples	Assigned lab	Accuracy/Bias and Precision	Recoveries and RPD per SOM01.2, Exhibit D, Table 6 for Criteria
Deuterated Monitoring Compounds	Each sample, standard, blank	Recoveries per SOM01.2	Per SOM01.2, investigate and re-prepare and reanalyze the samples	Assigned lab	Accuracy/Bias	Recoveries per SOM01.2, Exhibit D, Table 5 for Criteria

QAPP Worksheet #28
(UFP-QAPP Manual Section 3.4)

QC Samples Table

Matrix	Groundwater
Analytical Group	TCL SVOC
Concentration Level	Low
Sampling SOP	See Worksheet 20 and FSP Appendix A
Analytical Method/ SOP Reference	SOM01.2
Sampler's Name	Malcolm Pirnie Field Sampling Crew
Field Sampling Organization	Malcolm Pirnie Inc
Analytical Organization	Assigned EPA CLP Lab
No. of Sample Locations	See Worksheet 18 and FSP Tables

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	Once every 12 hour for each instrument per SOM01.2	Per SOM01.2	Per SOM01.2, correct and reanalyze blank	Assigned lab	Sensitivity	Per SOM01.2, less than CRQLs Reference USEPA Region 2 SOP No. 33/Low/Medium VOA-Blank Type Criteria Table
Method Detection Limits	Annual	Per SOM01.2	Per SOM01.2, correct problem and determine detection limits	Assigned lab	Sensitivity	Per SOM01.2, low enough to support CRQLs
The assigned laboratory also must perform the QC/QA sample analyses and meet all the measurement performance criteria which assess the analytical DQIs specified in EPA CLP SOW SOM01.2 such as laboratory duplicates and matrix spike duplicates for precision, matrix spikes, Deuterated monitoring compounds for accuracy, and blanks and method detection limits for sensitivity. The lab personnel must follow all the corrective actions required by the EPA CLP SOW.						

QAPP Worksheet #28
(UFP-QAPP Manual Section 3.4)

QC Samples Table

Matrix	Groundwater and solids (Such as Bedrock)
Analytical Group	PCB Aroclors
Concentration Level	Low
Sampling SOP	See Worksheet 20 and FSP Appendix A
Analytical Method/ SOP Reference	SOM01.2
Sampler's Name	Malcolm Pirnie Field Sampling Team
Field Sampling Organization	Malcolm Pirnie
Analytical Organization	Assigned USEPA CLP Lab
No. of Sample Locations	See Worksheet 18 and FSP Tables

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Equipment and/or Field Blanks	At least once a week but no more than once a day	FSP and QAPP	Investigate source of contamination	Malcolm Pirnie Field Team Leader	Sensitivity	< QL
Field Duplicates	1 per 20 field samples per matrix	FSP and QAPP	If the results exceed limits for the field replicate, this will be addressed by the Malcolm Pirnie Data Reviewer	Malcolm Pirnie Field Team Leader and or Laboratory	Precision	RPD 30% for duplicate values on aqueous samples and 40% for solids greater than or equal to 5 times the CRQL
Matrix Spike/Matrix Spike Duplicates (MS/MSD)	Each group of field samples in an SDG, or each SDG, whichever is most frequent per SOM01.2	Per SOM01.2 Exhibit D, Section 12.0,	Reanalyze the samples	Assigned lab	Accuracy/Bias/ Precision	Per Recovery and RPD criteria in SOM01.2 Exhibit D, Table 1
Laboratory Control Samples (LCS)	Once every 20 samples of the sample matrix in an SDG	Per SOM01.1/1.2, Exhibit D, Section 12.0,	Check calculation. It may be necessary to re-calibrate to meet acceptance criteria	Assigned lab	Accuracy/Bias	Per Recovery criteria in SOM01.2 Exhibit D, Table 2

QC Samples Table

Matrix	Groundwater and solids (Such as Bedrock)
Analytical Group	PCB Aroclors
Concentration Level	Low
Sampling SOP	See Worksheet 20 and FSP Appendix A
Analytical Method/ SOP Reference	SOM01.2
Sampler's Name	Malcolm Pirnie Field Sampling Team
Field Sampling Organization	Malcolm Pirnie
Analytical Organization	Assigned USEPA CLP Lab
No. of Sample Locations	See Worksheet 18 and FSP Tables

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	Each group of 20 samples or less of the same matrix	Per SOM01.1/1.2, Exhibit D, Section 12.0,	Correct problem and reanalyze blank	Assigned lab	Sensitivity	No analyte > CRQLS Reference USEAP Region 2 SOP No. 37/Low/Medium Aroclor- Blank Type Criteria Table
Method Detection Limits	Annually	Per SOM01.1/1.2, Exhibit D, Section 12.0,	Correct the problem and determine method detection limits per SOM01.2 before analyzing samples	Assigned lab	Sensitivity	Must be less than CRQLs

The assigned laboratory also must perform all the QA/QC sample analyses and meet all the measurement performance criteria that assess the analytical DQIs specified in USEPA CLP SOW SOM01.2, such as laboratory duplicates and matrix spike duplicates for precision, matrix spikes, laboratory control standards for accuracy, and blanks and method detection limits for sensitivity. The lab personnel must follow all the corrective actions required by the USEPA CLP SOW.

QAPP Worksheet #28
(UFP-QAPP Manual Section 3.4)

QC Samples Table

Matrix	Groundwater
Analytical Group	Pesticides
Concentration Level	Low
Sampling SOP	See Worksheet 20 and FSP Appendix A
Analytical Method/ SOP Reference	SOM01.2
Sampler's Name	Malcolm Pirnie Field Sampling Team
Field Sampling Organization	Malcolm Pirnie
Analytical Organization	Assigned USEPA CLP Lab
No. of Sample Locations	See Worksheet 18 and FSP Tables

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Equipment and/or Field Blanks	At least once a week but no more than once a day	FSP and QAPP	Investigate source of contamination	Malcolm Pirnie Field Team Leader	Sensitivity	< QL
Field Duplicates	1 per 20 field samples per matrix	FSP and QAPP	If the results exceed limits for the field replicate, this will be addressed by the Malcolm Pirnie Data Reviewer	Malcolm Pirnie Field Team Leader and or Laboratory	Precision	RPD 30% for duplicate values on aqueous samples greater than or equal to 5 times the CRQL
Matrix Spike/Matrix Spike Duplicates (MS/MSD)	Each group of field samples in an SDG, or each SDG, whichever is most frequent per SOM01.2	Per SOM01.2 Exhibit D, Section 12.0,	Reanalyze the samples	Assigned lab	Accuracy/Bias/ Precision	Per Recovery and RPD criteria in SOM01.2 Exhibit D, Table 3
Laboratory Control Samples (LCS)	Once every 20 samples of the sample matrix in an SDG	Per SOM01.1/1.2, Exhibit D, Section 12.0,	Check calculation. It may be necessary to re-calibrate to meet acceptance criteria	Assigned lab	Accuracy/Bias	Per Recovery criteria in SOM01.2 Exhibit D, Table 2

QC Samples Table

Matrix	Groundwater
Analytical Group	Pesticides
Concentration Level	Low
Sampling SOP	See Worksheet 20 and FSP Appendix A
Analytical Method/ SOP Reference	SOM01.2
Sampler's Name	Malcolm Pirnie Field Sampling Team
Field Sampling Organization	Malcolm Pirnie
Analytical Organization	Assigned USEPA CLP Lab
No. of Sample Locations	See Worksheet 18 and FSP Tables

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	Each group of 20 samples or less of the same matrix	Per SOM01.2, Exhibit D	Correct problem and reanalyze blank	Assigned lab	Sensitivity	No analyte > CRQLS
Method Detection Limits	Annually	Per SOM0 1.2, Exhibit D	Correct the problem and determine method detection limits per SOM01.2 before analyzing samples	Assigned lab	Sensitivity	Must be less than CRQLs
The assigned laboratory also must perform all the QA/QC sample analyses and meet all the measurement performance criteria that assess the analytical DQIs specified in USEPA CLP SOW SOM01.2, such as laboratory duplicates and matrix spike duplicates for precision, matrix spikes, laboratory control standards for accuracy, and blanks and method detection limits for sensitivity. The lab personnel must follow all the corrective actions required by the USEPA CLP SOW.						

QAPP Worksheet #28

QC Samples Table

Matrix	Groundwater
Analytical Group	Target Analyte List Inorganics Metals
Concentration Level	Low/Medium (ug/L)
Sampling SOP(s)	See Worksheet 20 and FSP Appendix A
Analytical Method/SOP Reference	SOM01.2
Sampler's Name	Malcolm Pirnie Field Personnel
Field Sampling Organization	Malcolm Pirnie
Analytical Organization	USEPA CLP Laboratory
No. of Sample Locations	See Worksheet 18 and FSP Tables

Lab QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Field Duplicate	1 per 20 samples	QAPP	Will be considered by data validator	Data Validator	Precision	≤20% RPD
Preparation Blank	1 per ≤ 20 samples	No constituent > CRQL	Suspend analysis until source rectified; redigest and reanalyze affected samples	EPA CLP RAS Laboratory ICP-AES/ICP-MS Technician	Accuracy	No constituent > CRQL
Spike	1 per ≤ 20 samples	75-125%R*	Flag outliers	EPA CLP RAS Laboratory ICP-AES/ICP-MS Technician	Accuracy	75-125%R*
Duplicate	1 per ≤ 20 samples	± 20% RPD**	Flag outliers	EPA CLP RAS Laboratory ICP-AES/ICP-MS Technician	Precision	± 20% RPD**
Post-Digestion Spike	after any analyte (except Ag and Hg) fails spike %R	75-125%R	Flag outliers	EPA CLP RAS Laboratory ICP-AES/ICP-MS Technician	Accuracy	75-125%R
Interference Check Sample [ICP Analysis Only]	beginning, end and periodically (not less than once per 20 samples)	± 2 times CRQL of true value or ± 20% of true value, whichever is greater	Check calculations and instruments, reanalyze affected samples	EPA CLP RAS Laboratory ICP-AES/ICP-MS Technician	Sensitivity	± 2 times CRQL of true value or ± 20% of true value, whichever is greater

*except when the sample concentration is greater than 4 times the spike concentration, then disregard the recoveries; no data validation action taken
**Reference USEPA Region 2 SOP No. HW-2, Revision 13/Evaluation of Metals Data for CLP - (include absolute difference criteria)
**except when the sample and/or duplicate concentration is less than 5 times the CRQL, then ± CRQL.

QAPP Worksheet #28

QC Samples Table

Matrix	Groundwater
Analytical Group	Target Analyte List Inorganics Metals [cont'd]
Concentration Level	Low/Medium (ug/L)
Sampling SOP(s)	See Worksheet 20 and FSP Appendix A
Analytical Method/SOP Reference	ILM05.4
Sampler's Name	Malcolm Pirnie Field Personnel
Field Sampling Organization	Malcolm Pirnie
Analytical Organization	USEPA CLP Laboratory
No. of Sample Locations	See Worksheet 18 and FSP

Lab QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Laboratory Control Sample	1 per ≤ 20 samples	80-120%R (except Ag and Sb)	Suspend analysis until source rectified; redigest and reanalyze affected samples	EPA CLP RAS Laboratory ICP-AES/ICP-MS Technician	Accuracy	80-120%R (except Ag and Sb)

QAPP Worksheet #28

QC Samples Table

(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 limit exceed the measurement performance criteria, the data obtained may be unusable for making project decisions.

Matrix	Groundwater
Analytical Group	Target Analyte List Inorganics – Total Mercury
Concentration Level	Low/Medium (ug/L)
Sampling SOP(s)	See FSP Appendix A
Analytical Method/SOP Reference	ILM05.4 – Cold Vapor Atomic Absorption (CVAA)
Sampler's Name	Malcolm Pirnie Field Personnel
Field Sampling Organization	Malcolm Pirnie
Analytical Organization	USEPA CLP Laboratory
No. of Sample Locations	See Worksheet 18 and FSP Tables

Lab QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Field Duplicate	1 per 20 samples	QAPP	Will be considered by data validator	Data Validator	Precision	≤20% RPD
Preparation Blank (PB)	1 per ≤ 20 samples	No analyte > CRQL	Suspend analysis; redigest and reanalyze	EPA CLP RAS Laboratory Technician	Accuracy	No analyte > CRQL
Duplicate Sample	1 per ≤ 20 samples	± 20% RPD*	Flag outliers	EPA CLP RAS Laboratory Technician	Precision	± 20% RPD
Spike Sample	1 per ≤ 20 samples	75 – 125 %R	Flag outliers	EPA CLP RAS Laboratory Technician	Accuracy	75 – 125 %R

*Reference USEPA Region 2 SOP No. HW-2, Revision 13/Evaluation of Metals Data for CLP - (include absolute difference criteria)

QAPP Worksheet #28

QC Samples Table

(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 limit exceed the measurement performance criteria, the data obtained may be unusable for making project decisions.

Matrix	Groundwater
Analytical Group	Target Analyte List Inorganics - Total Cyanide
Concentration Level	Low/Medium (ug/L)
Sampling SOP(s)	See Worksheet 20 and FSP Appendix A
Analytical Method/SOP Reference	ILM05.4 – Colorimeter or Spectrophotometer
Sampler's Name	Malcolm Pirnie Field Personnel
Field Sampling Organization	Malcolm Pirnie
Analytical Organization	USEPA CLP Laboratory
No. of Sample Locations	See Worksheet 18 and FSP Tables

Lab QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Field Duplicate	1 per 20 samples	QAPP	Will be considered by data validator	Data Validator	Precision	≤20% RPD
Preparation Blank (PB)	1 per ≤ 20 samples	No analyte > CRQL	Suspend analysis; redistill and reanalyze	EPA CLP RAS Laboratory Technician	Accuracy	No analyte > CRQL
Duplicate Sample	1 per ≤ 20 samples	± 20% RPD*	Flag outliers	EPA CLP RAS Laboratory Technician	Precision	± 20% RPD
Spike Sample	1 per ≤ 20 samples	75 – 125 %R	Flag outliers	EPA CLP RAS Laboratory Technician	Accuracy	75 – 125 %R

*Reference USEPA Region 2 SOP No. HW-2, Revision 13/Evaluation of Metals Data for CLP - (include absolute difference criteria)

QAPP Worksheet #28

QC Samples Table

Matrix	Ground Water
Analytical Group	Total Organic Carbon Dissolved Carbon
Concentration Level	Low
Sampling SOP	See Worksheet 20 and FSP Appendix A
Analytical Method/ SOP Reference	SW 846 9060
Sampler's Name	Malcolm Pirnie Field Sampling Crew
Field Sampling Organization	Malcolm Pirnie Inc
Analytical Organization	Assigned Lab
No. of Sample Locations	See Worksheet 18 and FSP Tables

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 Duplicates	One per analytical batch of 20 or fewer samples	QAPP	Identify problem and correct	Assigned lab	Precision	RPD $\leq$50% for duplicate values
Matrix Spike and Matrix Spike Duplicate	One per analytical batch of 20 or fewer samples	Per Lab SOP	Investigate	Assigned lab	Precision and Accuracy	Per control limits in Lab SOP
Lab Duplicate	One per analytical batch of 20 or fewer samples	Per SOP	Identify problem and correct	Assigned lab	Precision	RPD $\leq$ 30% for duplicate values
Method Detection Limits	At instrument setup and at least annually and for a new operator	Per Lab SOP	Re-determine when necessary	Subcontract lab or DESA	Sensitivity	Low enough to support RLs and project QLs
Method Blank	One per analytical batch of 20 or fewer samples	Per Lab SOP	Investigate and correct	Subcontract lab or DESA	Sensitivity	Less than RLs
If the DESA performs the analysis they must comply with their own laboratory SOP.						

QAPP Worksheet #28

QC Samples Table

Matrix	Groundwater
Analytical Group	Anions – Sulfate, Chloride and Nitrate
Concentration Level	Low
Sampling SOP	See Worksheet 20 and FSP Appendix A
Analytical Method/ SOP Reference	SW-846 9056 or EPA 300.0 (alternately Nitrate can be measured by EPA 353.2)
Sampler's Name	Malcolm Pirnie Field Sampling Crew
Field Sampling Organization	Malcolm Pirnie Inc
Analytical Organization	Assigned Lab
No. of Sample Locations	See Worksheet 18 and FSP Tables

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 Duplicates	One per analytical batch of 20 or fewer samples	QAPP	Identify problem and correct	Assigned lab	Precision	RPD ≤30% for duplicate values
Calibration and Calibration Verification	Per DoD QSM Table B-10	Per Lab SOP	Per DoD QSM Table B	Assigned lab	Accuracy	Per DoD QSM Table B-10
Method Detection Limits	Annually	Per Lab SOP	Per DoD QSM Table B	Assigned lab	Sensitivity	Per DoD QSM Table B
Method Blank	One per preparatory batch	Per Lab SOP	Per DoD QSM Table B	Assigned lab	Sensitivity	Per DoD QSM Table B
Laboratory Control Standard	One per analytical batch of 20 or fewer samples	Per Lab SOP	Per DoD QSM Table B	Assigned lab	Accuracy	Per DoD QSM Table B

Matrix	Groundwater
Analytical Group	Anions – Sulfate, Chloride and Nitrate
Concentration Level	Low
Sampling SOP	See Worksheet 20 and FSP Appendix A
Analytical Method/ SOP Reference	SW-846 9056 or EPA 300.0 (alternately Nitrate can be measured by EPA 353.2)
Sampler's Name	Malcolm Pirnie Field Sampling Crew
Field Sampling Organization	Malcolm Pirnie Inc
Analytical Organization	Assigned Lab
No. of Sample Locations	See Worksheet 18 and FSP Tables

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 Duplicate	One per preparatory batch	Per lab SOP	Per DoD QSM Table B	Assigned lab	Precision	Per DoD QSM Table B
Matrix Spike/Matrix Spike Duplicate	One per analytical batch of 20 or fewer samples	Per SOP	Per DoD QSM Table B	Assigned lab	Precision and Accuracy	Per DoD QSM Table B
The assigned laboratory should also perform the QC/QA sample analyses and meet all the measurement performance criteria which assess the analytical DQIs specified in the EPA methods. If the DESA performs the analysis they must comply with their own laboratory SOP. Nitrate can be measured by an alternate method such as EPA 353.2, in which case the method QC requirements must be followed.						

QAPP Worksheet #28

QC Samples Table

Matrix	Ground Water
Analytical Group	Alkalinity
Concentration Level	NA
Sampling SOP	See Worksheet 20 and FSP Appendix A
Analytical Method/ SOP Reference	SM2320B or equivalent
Sampler's Name	Malcolm Pirnie Field Sampling Crew
Field Sampling Organization	Malcolm Pirnie Inc
Analytical Organization	Assigned Lab
No. of Sample Locations	See Worksheet 18 and FSP Tables

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 Duplicates	One per analytical batch of 20 or fewer samples	QAPP	Identify problem and correct	Assigned lab	Precision	RPD $\leq$30% for duplicate values
Lab Duplicates	One per analytical batch of 20 or fewer samples	Per SOP		Assigned lab	Precision	RPD $\leq$ 20% for duplicate values
Laboratory Control Samples	One per analytical batch	Per SOP	Identify problem and correct	Assigned lab	Precision	Per Lab SOP
If the DESA performs the analysis they must comply with their own laboratory SOP.						

QAPP Worksheet #28

QC Samples Table

Matrix	Ground Water
Analytical Group	Ferrous Iron
Concentration Level	Low
Sampling SOP	See Worksheet 20 and FSP Appendix A
Analytical Method/ SOP Reference	SM 3500 Fe D or equivalent
Sampler's Name	Malcolm Pirnie Field Sampling Crew
Field Sampling Organization	Malcolm Pirnie Inc
Analytical Organization	Assigned Lab
No. of Sample Locations	See Worksheet 18 and FSP Tables

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Blank	At the beginning of each event	SM 3550 Fe D	Correct for blank	Assigned lab	Sensitivity	Blank corrected to 0.00 mg/L Fe^{2+}
Accuracy Check Standard	At the beginning of each event	SM 3550 Fe D	Adjust calibration	Assigned lab	Accuracy	0.977 to 1.23 for a 1.0 mg/L standard
Field Duplicates	One per analytical batch of 20 or fewer samples	FSP and QAPP	Investigate problem and repeat analyses. Inform field team leader	Assigned lab	Precision	RPD $\leq$35% for duplicate greater than 5 times the RL
If samples are turbid the data will not be valid unless the samples are filtered. If the DESA performs the analysis they must comply with their own laboratory SOP.						

QAPP Worksheet #28

QC Samples Table

Matrix	Groundwater
Analytical Group	Ethane, Methane and Ethene
Concentration Level	Low
Sampling SOP	See Worksheet 20 and FSP Appendix A
Analytical Method/ SOP Reference	EPA RSK-175 or SW-846 8015B or equivalent
Sampler's Name	Malcolm Pirnie Field Sampling Crew
Field Sampling Organization	Malcolm Pirnie Inc
Analytical Organization	Assigned Lab
No. of Sample Locations	See Worksheet 18 and FSP Tables

QC Sample:	Frequency/Number	Method/SOP QC *Acceptance Limits*	Corrective Action	Person(s) Responsible *for Corrective* Action	Data Quality Indicator (DQI)	Measurement *Performance* Criteria
Initial Calibration (IC)	Prior to the analysis of any field samples	Per Lab SOP	Repeat, prepare new standards	Assigned lab	Accuracy	Regression coefficient (r2) greater than 0.995
Continuing Calibration Check (CCC)	Every 4 hours, every 25 samples or less, whichever is more frequent, and at the end of each sample batch	Per Lab SOP	Repeat, recalibrate, prepare new standards	Assigned lab	Accuracy	Difference from the calculated value <20%
Method Blank	One per analytical batch of 20 or fewer samples	Per Lab SOP	Investigate and correct	Assigned lab	Sensitivity	Less than RLs
Field Duplicates	One per analytical batch of 20 or fewer samples	Per Lab SOP	Identify problem and correct	Assigned lab	Precision	RPD ≤30 % for duplicate values greater than or equal to 5 times the RL

Matrix	Groundwater
Analytical Group	Ethane, Methane and Ethene
Concentration Level	Low
Sampling SOP	See Worksheet 20 and FSP Appendix A
Analytical Method/ SOP Reference	EPA RSK-175 or SW-846 8015B or equivalent
Sampler's Name	Malcolm Pirnie Field Sampling Crew
Field Sampling Organization	Malcolm Pirnie Inc
Analytical Organization	Assigned Lab
No. of Sample Locations	See Worksheet 18 and FSP Tables

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix Spike (MS)	One per analytical batch of 20 or fewer samples		Investigate and correct, dilute if necessary and repeat. Report in case narrative	Assigned lab	Accuracy	Recovery 30-150%
If the DESA performs the analysis they must comply with their own laboratory SOP.						

QAPP Worksheet #28

QC Samples Table

Matrix	Bedrock
Analytical Group	Fraction of Organic Carbon
Concentration Level	Low
Sampling SOP	See Worksheet 20 and FSP Appendix A
Analytical Method/ SOP Reference	EPA Region 2 Lloyd Kahn, ASTM 2794 or equivalent
Sampler's Name	Malcolm Pirnie Field Sampling Crew
Field Sampling Organization	Malcolm Pirnie Inc
Analytical Organization	Assigned Lab
No. of Sample Locations	See Worksheet 18 and FSP Tables

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 Duplicates	One per analytical batch of 20 or fewer samples	QAPP	Identify problem and correct	Assigned lab	Precision	RPD $\leq$50% for duplicate values
Matrix Spike and Matrix Spike Duplicate	One per analytical batch of 20 or fewer samples	Per Lab SOP	Investigate	Assigned lab	Precision and Accuracy	Per control limits in Lab SOP
Lab Duplicate	One per analytical batch of 20 or fewer samples	Per SOP	Identify problem and correct	Assigned lab	Precision	RPD $\leq$ 30% for duplicate values
Method Detection Limits	At instrument setup and at least annually and for a new operator	Per Lab SOP	Re-determine when necessary	Subcontract lab or DESA	Sensitivity	Low enough to support RLs and project QLs
Method Blank	One per analytical batch of 20 or fewer samples	Per Lab SOP	Investigate and correct	Subcontract lab or DESA	Sensitivity	Less than RLs

QAPP Worksheet #29

(UFP-QAPP Manual Section 3.5.1)

This section identifies 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- and off-site analysis, and data assessment.

Project Documents and Records Table

Sample Collection Documents and Records:

- Field Notes and or data sheets
- Boring logs
- Chain of Custody Forms including Form II Lite COCs and Trip Reports for CLP samples
- Air bills
- Analytical and Testing Sample Data Packages
- Data Validation Reports for any non-CLP data
- ANSETS forms are monthly records of the collected samples and the associated costs and must be submitted to USEPA Region 2 with copies to the USACE-KCD.

On-Site Activities Documents and Records:

- Sample collection and processing record and custody records.
- Sample custody records
- Air bills
- Custody records
- Copies of field notes

Off-Site Analysis Documents and Records

- Chain of Custody (COC) records will be made and stored in the project files
- Copies of Air Bills will be kept in project files
- Copies of all Analytical Data Deliverables stored in Lab and transferred to Project files, instrument calibration records, lab, raw data stored in electronically or in hardcopy. Laboratory electronic data deliverables (EDD) will be obtained in a USEPA Region 2 compliment format.

Data Assessment Documents and Records

- Project Records: Copies of all field notes must be send to Malcolm Pirnie Inc
- Project Records: Copies of CLP COC must be send to the CLP coordinator and to Malcolm Pirnie Inc
- Project Records: Copies of COC for non-CLP must be kept by Malcolm Pirnie Inc
- Field and/or lab inspection reports/checklists
- Corrective action documentation
- Data validation narratives
- QA Review sheet
- Copies of Form 1
- Final Report

Data Management

This section describes the project data management process, tracing the path of the data from their generation to their final use or storage. All project data and information must be documented in a format useable to the project personnel.

Project Document Control System

Project documents will be controlled by the Malcolm Pirnie Technical Project Manager and the Deputy Project Manager who will maintain and distribute the hardcopies and electronic copies of the project documents, including any amendments. Electronic copies of project information will be maintained in the project directory on the server at Malcolm Pirnie corporate headquarters, which is backed up at least once per day.

Data Recording

Data for this project will be collected by handwritten entries and will be recorded into field logbooks or on forms. Forms II Lite software will be the primary tool used to generate COC records and sample labels, or possibly COCs and labels will be created manually for non-CLP test parameters. Forms II Lite software has been designed to meet USEPA requirements. Computer-generated data associated with laboratory analyses will be managed under the control of the assigned USEPA-CLP or sub-contract laboratory's laboratory information management system (LIMS).

Laboratory Data Transmittal

Laboratory data are managed by the laboratory's LIMS system, beginning with sample check-in on the sample receiving data terminal. For non-CLP laboratories, full laboratory data reports will be delivered to Malcolm Pirnie and will include electronic data deliverables (EDDs). In addition, Region 2 requires that non-CLP laboratories adhere to the Analytical Services Tracking System (ANSETS) reporting requirements, which utilize the ANSETS Data Requirement form. Detailed instructions and procedures can be obtained by contacting the USEPA Region 2 Regional Sample Control Coordinator, Ms. Jennifer Feranda, at 732-321-6687 or feranda.jennifer@epa.gov, or Mr. Adly Michael at 732-906-6161 or Michael.adly@epa.gov. For CLP laboratories, third-party validated laboratory results will be received by Malcolm Pirnie through the USEPA RSCC, and will include EDDs.

Data Storage and Retrieval

Paper copies of the forms, electronic copies of files, and the photographic log will be transmitted regularly to the Malcolm Pirnie PM or designee. The completed forms and notebooks will be stored in the custody of the PM for the duration of the project. The full laboratory data reports submitted to Malcolm Pirnie will be stored in the custody of the Project Quality Manager. The Laboratory will maintain copies of documents and backups of all data associated with the analyses of samples. Raw data and electronic media of all field samples, including QC samples and blanks, will be archived from the date of generation and will be kept by the laboratory per the requirements of USEPA-CLP. Hard copies of project files will be archived at a secure facility and retained until the end of the contract; project closeout will be conducted in accordance with USEPA Close-out Guidelines. Data will be transferred to the USACE upon completion of the project. Retrieval of data by others will be at the discretion of the USACE and the USEPA. The length of time that records will be archived will be at the discretion of the USACE and USEPA.

Each laboratory shall archive, electronically, the sample analyses and submit the electronic data files along with the data deliverable package. Laboratory electronic data deliverables (EDD) will be obtained in a USEPA Region 2 compliment format (http://www.epa.gov/Region2/superfund/medd.htm).

QAPP Worksheet #30
(UFP-QAPP Manual Section 3.5.2.3)

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) [1]	Backup Laboratory/Organization (Name and Address, Contact Person and Telephone Number [2]
Groundwater	TCL VOCs	Low	See Worksheet 18 and FSP in Appendix A	SOM01.2	42 days	Assigned CLP lab or EPA Region 2 DESA lab Contracts: Adly Michael or Jennifer Feranda	A backup lab has not been assigned at this time.
Groundwater	TCL SVOCs	Low	See Worksheet 18 and FSP in Appendix A	SOM01.2	42 days	Assigned CLP lab or EPA Region 2 DESA lab	A backup lab has not been assigned at this time.
Groundwater	TCL Pesticides	Low	See Worksheet 18 and FSP in Appendix A	SOM01.2	42 days	Contracts: Adly Michael or Jennifer Feranda	A backup lab has not been assigned at this time.
Groundwater	PCB Aroclors	Low	See Worksheet 18 and FSP in Appendix A	SOM01.2	42 days	Assigned CLP lab or EPA Region 2 DESA lab	A backup lab has not been assigned at this time.
Groundwater	TAL Metals/Cyanide	Low	See Worksheet 18 and FSP in Appendix A	ILM05.4	42 days	Contracts: Adly Michael or Jennifer Feranda	A backup lab has not been assigned at this time.
Groundwater	TCL Dioxins/Furans	Low	See Worksheet 18 and FSP in Appendix A	DLM02.0 or USEPA1613B	Est. 60 days	Assigned CLP lab or EPA Region 2 DESA lab Contracts: Adly Michael or Jennifer Feranda	A backup lab has not been assigned at this time.
Groundwater	PCB Congeners	Low	See Worksheet 18 and FSP in Appendix A	CBC01.0 or USEPA 1668A or DESA method	Est. 60 days	Assigned CLP lab or EPA Region 2 DESA lab Contracts: Adly Michael or Jennifer Feranda	A backup lab has not been assigned at this time.
Groundwater	TOC/DOC	Low	See Worksheet 18 and FSP in Appendix A	SW-846-9060A or equivalent or DESA method	Est. 60 days	Accutest Laboratories 2235 Route 130 Dayton, NJ 08810 Contact: Nadine Yates 732-329-0200	A backup lab has not been assigned at this time.

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) [1]	Backup Laboratory/Organization (Name and Address, Contact Person and Telephone Number [2]
Groundwater	Sulfate, Chloride and Nitrate	Low	See Worksheet 18 and FSP in Appendix A	SW-846-9056, EPA 300 (EPA 353.2 for Nitrate) or DESA methods	Est. 60 days	Accutest Laboratories 2235 Route 130 Dayton, NJ 08810 Contact: Nadine Yates 732-329-0200	A backup lab has not been assigned at this time.
Groundwater	Alkalinity	NA	See Worksheet 18 and FSP in Appendix A	SM2340B or equivalent or DESA method	Est. 60 days	Accutest Laboratories 2235 Route 130 Dayton, NJ 08810 Contact: Nadine Yates 732-329-0200	A backup lab has not been assigned at this time.
Groundwater	Hardness	NA	See Worksheet 18 and FSP in Appendix A	SM2340C or equivalent or DESA method	Est. 60 days	Accutest Laboratories 2235 Route 130 Dayton, NJ 08810 Contact: Nadine Yates 732-329-0200	A backup lab has not been assigned at this time.
Groundwater	Ferrous Iron	Low	See Worksheet 18 and FSP in Appendix A	SM-3500 Fe D or DESA method	Est. 60 days	Accutest Laboratories 2235 Route 130 Dayton, NJ 08810 Contact: Nadine Yates 732-329-0200	A backup lab has not been assigned at this time.
Groundwater	Dissolved Gases	Low	See Worksheet 18 and FSP in Appendix A	RSK-175, SW-846-8015 or equivalent or DESA method	Est. 60 days	Accutest Laboratories 2235 Route 130 Dayton, NJ 08810 Contact: Nadine Yates 732-329-0200	A backup lab has not been assigned at this time.
Groundwater	De-Chlorination Bacteria	NA	See Worksheet 18 and FSP in Appendix A	Bioemediation Consulting, Inc., SOP #14 Microcosm Studies and Presence-Absence tests	Est. 1 month	Bioemediation Consulting, Inc.	A backup lab has not been assigned at this time.

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) [1]	Backup Laboratory/Organization (Name and Address, Contact Person and Telephone Number [2]
Bedrock	Chlorinated volatiles	Low	See Worksheet 18 and FSP in Appendix A	Stone Environmental SOPs SEI-6.37.0, SEI-10.17.0 and SEI-10.18.0	Sample processing and analysis will be performed in the field. Data should be available the same day.	Stone Environmental, Inc 535 Stone Cutters Way Montpelier, Vermont 05602 Michael Rossi, 802-229-2194 (Stone Environmental will collect and process (crush and extract) the bedrock samples in the field)	A backup lab has not been assigned at this time.
Bedrock	PCB Aroclors	Low	See Worksheet 18 and FSP in Appendix A	SOM01.2 or SW-846-8082 or DESA method	Sample processing (crushing) will be performed in the field. Estimate 60 days for data package	Assigned CLP lab or EPA Region 2 DESA lab (assuming an assigned EPA lab can extract crushed rock matrix) Contracts: Adly Michael or Jennifer Feranda	Accutest Laboratories 2235 Route 130 Dayton, NJ 08810 Contact: Nadine Yates 732-329-0200 (Could become the primary lab for PCBs in rock, if it is determined that EPA CLP /DESA cannot process the rock samples)
Bedrock	Geological Physical Property Tests: Moisture, Bulk Density, Specific Gravity and Porosity - (calculated from physical property data)	NA	See Worksheet 18 and FSP in Appendix A	ASTM D2215 ASTM D4531 ASTM D854	Estimate 35 days for data package	Golder Associates Ltd. 2390 Argentia Road, Mississauga, Ontario, Canada L5N 5Z7 (905) 567 4444 Marijana Manojlovic Laboratory Manager	A backup lab has not been assigned at this time

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) [1]	Backup Laboratory/Organization (Name and Address, Contact Person and Telephone Number [2]
Bedrock	Fraction of Organic Carbon	Low	See Worksheet 18 and FSP in Appendix A	USEPA Lloyd Kahn, ASTM D2974 or DESA method	Estimate 60 days for data package	Accutest Laboratories 2235 Route 130 Dayton, NJ 08810 Contact: Nadine Yates 732-329-0200	A backup lab has not been assigned at this time
Bedrock	Soil Oxidant Demand	NA	See Worksheet 18 and FSP in Appendix A	ASTM D7363	Estimate 35 days for data package	Redox Tech Subcontracted through Accutest Laboratories 2235 Route 130 Dayton, NJ 08810 Contact: Nadine Yates 732-329-0200	A backup lab has not been assigned at this time

1. Backup subcontractor laboratories may be selected at a future date pending procurement by Malcolm Pirnie and approval by the USACE.

QAPP Worksheet #31
(UFP-QAPP Manual Section 4.1.1)

Planned Project Assessments Table

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment (Title and Organizational Affiliation)	Person(s) Responsible for Responding to Assessment Findings (Title and Organizational Affiliation)	Person(s) Responsible for Identifying and Implementing Corrective Actions (CA) (Title and Organizational Affiliation)	Person(s) Responsible for Monitoring Effectiveness of CA (Title and Organizational Affiliation)
QC Reports of any non-conformance	Daily as required	Internal	Malcolm Pirnie	Field Team Members	Malcolm Pirnie Project Quality Officer	Malcolm Pirnie PM or designees	Malcolm Pirnie Project Quality Officer
Field Safety Audit	Initially within the first week of field work and then at least quarterly if necessary	Internal	Malcolm Pirnie	Malcolm Pirnie Project Safety Officer	Malcolm Pirnie PM	Malcolm Pirnie PM or designees	Malcolm Pirnie Project Safety Officer
Technical System Audit	Initially within the first week and then at least quarterly if necessary	Internal	Malcolm Pirnie	Malcolm Pirnie Project Quality Officer	Malcolm Pirnie PM	Malcolm Pirnie PM or designees	Malcolm Pirnie Project Quality Officer
Technical System Audit	Initially within the first week	External	USACE-KCS	Amy Darpinian/Chemist or designee/USACEKCD Project Manager	Malcolm Pirnie PM	Malcolm Pirnie PM or designees	Malcolm Pirnie Project Quality Officer

QAPP Worksheet #32
(UFP-QAPP Manual Section 4.1.2)

Assessment Findings and Corrective Action Responses

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (Name, Title, Organization)	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (Name, Title, Org.)	Timeframe for Response
Non-Conformance	See below	Project Quality Officer	As soon as possible	Complete non-conformance form	PM	As soon as possible
Technical System Field Audits (TSAs)	See Technical Systems Audit report SOP 6 in Attachment 5	Project Quality Officer, PM or designees, Malcolm Pirnie Inc	Within week	See below section on Field Corrective Actions	Field Team Leader and PM	Within week
Internal Laboratory Audits (This does not apply if the USEPA Region 2 DESA or a USEPA CLP Lab are assigned to perform the analyses)	Per Laboratory Quality Manual	Laboratory Management or designee	Annually	Per Laboratory Quality Manual	Laboratory Personnel	Per Laboratory Quality Manual

Non-Conformance/QC Reporting

A non-conformance is defined as an identified or suspected deficiency or discrepancy with regard to an approved document (*e.g.*, improper sampling procedures, improper instrument calibration, calculation, computer program); or an item where the quality of the end product itself or subsequent activities using the document or item would be affected by the deficiency; or an activity that is not

conducted in accordance with the established plans or procedures.

Any staff member engaged in project work that discovers or suspects a non-conformance is responsible for initiating a non-conformance report to the Project QC Officer. The Project QC Officer will evaluate each non-conformance report and the Project QC Officer will provide a disposition which describes the actions to be taken. An example Non-Conformance/QC report form is included in the Field Sampling Plan, Appendix A.

The Technical PM will verify that no further project work dependent on the nonconforming item or activity is performed until approval is obtained and the non-conformance is properly addressed. If the non-conformance is related to material, the Technical PM shall be responsible for marking or identifying, with the non-conformance report number, the nonconforming item (if practical) and indicating that it is nonconforming and is not to be used.
A copy of each non-conformance report will be included in the project file. Copies of all non-conformances shall be maintained by the Project QC Officer.

Assessment and Oversight

This element addresses assessment of the effectiveness of the project implementation and associated QA/QC activities.

Assessment and Response Actions

To monitor the capability and performance of the FSP activities, several types of audits will be performed. These audits will be conducted by the PQO or designee. Performance audits (PAs) of laboratories are conducted to measure the accuracy of the measurement systems. Data Quality Audits (DQAs) are conducted to determine if the data generated by the sampling and analysis satisfy the DQOs.

Technical System Audits (TSAs)

Field audits will be conducted on an ongoing basis during the project as field data are generated, reduced, and analyzed. Numerical manipulations, including manual calculations, will be documented. Records of numerical analyses will be legible, of reproduction-quality, and sufficiently complete to permit logical reconstruction by a qualified individual other than the originator. System audits of work activities will be accomplished by an inspection of field activities. During this audit, the auditor(s) will compare current field practices with standard procedures. The following elements will be evaluated during a TSA:

- Whether procedures and analyses are conducted according to procedures outlined in the FSP.
- Whether proper sample documentation is being recorded.
- If the working order of field instruments and equipment is being properly checked and recorded.
- The level of QC conducted per each field team.
- Decontamination procedures, where applicable.
- Sample packaging and shipment.

TSAs are conducted for each field team at the beginning of each field sampling task to determine if the system is capable of producing data that meet the DQOs. As long as the field team(s) demonstrate proficiency in the sampling procedures being audited, a follow-up audit will not be required. However, if the audit indicates the need for corrective action, a second TSA will be required. Following the initial audit, TSAs will be conducted on the following schedule:

- Whenever key personnel leave the project or new key personnel are added to the project.
- Whenever a significant amount of time (more than 6 months) has elapsed between TSAs for a particular field task.

Any minor deficiencies that are noted during the TSA will be corrected in the field as they occur. If major deficiencies are noted (*i.e.*, those that cannot be immediately corrected in the field), a Stop-Work Order will be issued until appropriate measures can be taken to correct the problem. A Stop-Work Order may be issued by the Project QC Officer, following notification to the PM. The conditions

and the need for a Stop-Work Order will be documented in sufficient detail to permit evaluation of the deficiency and determination of proper corrective action(s). Pertinent communications with the Project QC Officer, Field Team Leader, DPM, and PM that pertain to an evaluation of the problem along with potential solutions and their implementation will be attached to the Stop-Work Order. In order for work to resume following a Stop-Work Order, the Malcolm Pirnie PM and Project QC Officer must rescind it in writing. The Project QC Officer is responsible for tracking non-conforming conditions, evaluating the effectiveness of corrective measures, and assuring that the necessary steps have been taken to prevent recurrence of the original problem.

Regardless of whether major, minor, or no deficiencies were noted during the audit, a written report of the TSA will be prepared by the Project QC Officer and submitted to the Malcolm Pirnie, USEPA, and USACE PMs, as well as the Field Team Leader and the field team. This report will identify any deficiencies found and will outline the corrective actions that were recommended/implemented to address them. A copy of SOP No. 3 on conducting a TSA and an example of an audit form are found in Attachment 5. Note that the audit form contained in the SOP is for example purposes only; the Project QC Officer will tailor this form for each type of activity audited. Periodically during the audit, it may be determined that the program should be modified to increase data quality or efficiency. These modifications will be documented by the Malcolm Pirnie PM or Project QC Officer in a Field Modification Form. An example of this form can be found in Attachment 4.

Field Corrective Actions

At the end of each sampling day, the sampling team is to report any problems requiring corrective action that were encountered during the day. Corrective action will be undertaken when a non-conforming condition is identified. A non-conforming condition occurs when QA objectives for precision, accuracy, completeness, representativeness, or comparability are not met, or when procedural practices or other conditions are not acceptable. A report is to be filed that documents the problems encountered and the corrective action implemented. A Stop-Work Order may be issued by the Project QC Officer, following notification to the PM, if corrective action does not adequately address a problem, or if no resolution can be reached.

Performance Audits

A performance audit (PA) consists of sending a laboratory a performance evaluation (PE) sample for analysis. The PE sample is a sample of known concentration [established by an independent party such as the National Institute of Standards and Technology (NIST)] that is analyzed by the laboratory, and the analytical results are compared with the certified concentration. The results provide a measure of laboratory performance that is used along with other QA criteria to monitor laboratory capability. USPEA-CLP laboratories are required to perform quarterly analysis of PE samples. At the current time, there are no plans to conduct any additional PA using PE samples. Therefore, all chemical subcontract laboratories performing chemical tests procured for this project must be NJDEP or federally-certified and are subject to the performance audits required by those programs.

Internal Laboratory Audits

As part of its QA program, the Laboratory Quality Assurance Manager (QAM) will conduct periodic checks and audits of the analytical systems to ensure that the systems are working properly and personnel are adhering to established procedures and documenting the required information. These checks and audits will also assist in determining or detecting where problems are occurring.

In addition to conducting internal reviews and audits, as part of its established QA program the laboratory is required to take part in regularly scheduled Performance Evaluations and laboratory audits from State and Federal agencies for applicable tests. Each laboratory selected to support this program must maintain current NJDEP or Federal certifications, as appropriate. **This does not apply if the USEPA Region 2 DESA or a USEPA CLP Lab is assigned to perform the analyses. Malcolm Pirnie will only perform audits of commercial subcontract laboratories.**

Laboratory Corrective Actions

If a particular laboratory analysis is deemed "out of control," corrective action will be taken by the laboratory to maintain continued data quality. Each laboratory must adhere to their in-house corrective action policy. The coordinator of the laboratory's analytical

section will be responsible for initiating laboratory corrective action when necessary.

Data Quality Audits (DQAs)

DQAs are conducted to determine if the data are adequate to support the DQOs and to determine the cause of deficiencies in the event that the data quality is not adequate. This audit is conducted by the Project QC Officer or his designee after the data have been fully validated. The Project QC Officer will first determine to what extent the data can be used to support the decision making process. If the data are deficient, the Project QC Officer will identify the cause of the deficiency and will determine what modifications need to be made (*e.g.*, request that the laboratory analyze a larger volume sample or employ an alternate or modified method to lower the RLs so that subsequent data are acceptable.)

QAPP Worksheet #33
(UFP-QAPP Manual Section 4.2)

QA Management Reports Table

Type of Report	Frequency (daily, weekly monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (Title and Organizational Affiliation)	Report Recipient(s) (Title and Organizational Affiliation)
Progress Reports	Monthly	End of each following month	Malcolm Pirnie Administrative PM or Deputy PM	USEPA and USACE-KCD PMs
Technical System Field Internal Audit	Initally within the first two weeks of field work and with follow up audits if significant deficiencies are found	Month after field work begins	Project QC Officer or designee	USACE PM
Data Validation Report	After lab data is received	Within one month after receiving data	Data Validator	Project QC Officer and PM
Corrective Action Reports	When corrective action is required	When corrective action is implemented	Technical Project Manager or designee	Project Team and PM(s)

The USACE PM will receive several types of management reports. These will include the results of any corrective action reports and data validation reports. In addition, the progress report will contain a section on quality control reports. Problems or issues that arise between regular reporting periods may be identified to program management at any time. Information included in the progress report will include the following:

- Results of Technical System field audits conducted during the period.
- An assessment of any problems with the measurement data, including accuracy, precision, completeness, representativeness, and comparability.
- A listing of the non-conformance reports, including Stop-Work Orders issued during the period, related corrective actions undertaken, and an assessment of the results of these actions.
- Identification of significant quality assurance problems and recommended solutions, as necessary.

QAPP Worksheet #34
(UFP-QAPP Manual Section 5.2.1)

This worksheet and text section describes 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 Internal or *external* is in relation to the data generator.

Verification (Step I) Process Table

Verification Input	Description	Internal/ External	Responsible for Verification (Name, Organization)
Chain of custody (COC)	Form will be internally reviewed upon completion and verified against field logs and laboratory reports. Review will occur with the completetion of each report.	I	Malcolm Pirnie
Field report	Field reports will be verified with the field logbooks.	I	Malcolm Pirnie
Laboratory data packages	Laboratory data packages will be used to verify the reported results in the project report and against QAPP criteria.	I	Malcolm Pirnie

Data Verification

- The Field Team Leader or designee is required to review the logbook entries for errors or omissions. This information is transmitted to the Project QC Officer or designee for correction.
- In addition, the Project QC Officer or designee is responsible for reviewing field data for completeness and to verify that the field crew followed the QC requirements detailed in this QAPP (*e.g.*, the collection of QC samples at the required frequency, response checking the field instruments). If any problems with the information are found, the Project QC Officer or designee will document the problems.
- The Project QC Officer or designee reviews the field data.

QAPP Worksheet #35
(UFP-QAPP Manual Section 5.2.2)

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

Validation (Steps IIa and IIb) Process Table

Step IIa/IIb	Validation Input	Description	Responsible for Validation (Name, Organization)
IIa	Methods	Records support implementation of SOP in QAPP.	J. McCann, Malcolm Pirnie, Inc.
IIa	Chain of Custody	Examine traceability of data from sample collection to generation of project report	J. McCann, Malcolm Pirnie, Inc. (Based upon USEPA DESA lab reports or USEPA-CLP Data Validation Reports for CLP data.)
IIb	Deviations from SOP and project documents.	Determine impacts of any deviation from method and the project plan.	Technical Project Team led by Dan St. Germain, Malcolm Pirnie, Inc.

QAPP Worksheet #36
(UFP-QAPP Manual Section 5.2.2)

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

Validation (Steps IIa and IIb) Summary Table

Step IIa/IIb	Matrix	Analytical Group[1]	Concentration Level	Validation Criteria	Data Validator (title and organizational affiliation)[1]
IIa/IIb	Groundwater and Bedrock	Chemical Parameters	Low	USEPA Region 2 Validation Criteria	USEPA Data Validators assisted by subcontractor validators
IIa/IIb	Groundwater and Bedrock	Chemical/Geochemical Parameters	Low	USEPA Validation Guidance and Laboratory SOP Criteria	Malcolm Pirnie assigned data validators

1. We anticipate that the majority of the analyses will be performed by a USEPA CLP laboratory or the USEPA Region 2 DESA laboratory. If the DESA laboratory performs the analyses, the reported data will be considered valid. Analytical data on chemistry parameters produced by subcontract laboratories will be reviewed by a qualified data validator assigned by Malcolm Pirnie. Geotechnical physical property and oxidant demand test data will not be validated.

USEPA DESA Laboratory Data

Data generated by the USEPA Region 2 DESA laboratory in Edison, NJ undergo an internal laboratory QA review and are considered USEPA-validated and useable as reported. No third party data validation will be performed on DESA-generated data.

USEPA CLP Data

Validation of USEPA CLP data will be accomplished by comparing the contents of the data packages and QA/QC results to the

requirements contained in the applicable analytical methods (SOWs), laboratory SOPs, and validation guidelines. All data generated through the CLP will be validated by RSCC using the latest applicable USEPA Region 2 validation procedures in accordance to the following USEPA guidance documents or their most recent revisions:

- USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review, OPSWER 9240.1-46, USEPA-540-R-07-003, July 2007

- SOP NO. HW-37/Aroclor Validation of Data USEPA Contract Laboratory Program for Organic Analysis of Low/Medium Concentration of Aroclor Organic Compounds SOM01.1, Revision 0, April 2006.

Commercial Subcontractor Laboratory Data

Data generated by a commercial subcontractor laboratory will be validated by Malcolm Pirnie or a qualified Malcolm Pirnie subcontractor. Parameters will be validated in accordance with the QC requirements of the QAPP, laboratory SOPs, USEPA's National Functional Guidelines, and applicable Region 2 guidelines.

The validator will conduct a 100% validation of the first SDG received for each analytical parameter. This means that the validator will review the raw data and logbook sheets, and will recalculate at least 10 percent of the sample and QC sample results. If this validation indicates that the laboratory is producing acceptable data, the validation may be scaled back and subsequent data packages will have a less rigorous review. The validation will then be based on the information provided by the laboratory on their QC forms. If the laboratory QC on the report forms are within limits no further review will be conducted; however, if there are QA/QC aspects not meeting criteria, the validator may then review some or the entire full data package to determine the cause or data quality impact of the non-compliance. In addition, at least one of every five data packages will be subject to a full review.

Once data validation is completed, a data validation report will be generated. The report will contain information regarding the parameters that are qualified, the reason for the qualification, and the direction of the bias (only for parameters qualified as estimated), when possible. Based upon the quality assurance review of the analytical data, specific codes (data qualifiers or 'flags') will be placed next to results to provide an indication of the quantitative and qualitative reliability of the results. The data qualifier codes in the National Function Guidelines are proposed for this project. Qualifiers assigned by laboratories will be defined by each laboratory in their data package and will be superseded by the data validator's qualifiers.

Mobile Laboratory Chlorinated Volatiles Data

Since it will not be possible to fully validate the data in the field, a technical systems audit of the mobile lab is planned during the first few days of data collection to insure it is producing quality data. We will require that Stone Environmental have a second chemist in the Mobile lab responsible for checking the data quality and will also require the Stone Environmental provide a data quality summary report with the raw data and a case narrative within 1 month after producing the data.

Field Data Evaluation

Procedures to evaluate field data for this program include reviewing the data entered into the log books to insure that errors have not been made. The field data documented includes data generated during measurement of field parameters, observations, results of any quality control sample analyses, and field instrument calibrations. This task will be the responsibility of a Malcolm Pirnie Data Reviewer with oversight by the Project QC Officer or designee.

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

This worksheet describes 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.

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 USEPA will perform the validation of the USEPA-CLP laboratory data or the DESA laboratory. Data reported by assigned USEPA CLP laboratories will be validated by USEPA Region 2 validators and /or subcontract validators under contract to USEPA following Region 2 SOPs. DESA laboratory data undergoes an internal laboratory QA review and is considered to be validated by the USEPA. The Malcolm Pirnie data validator will validate the non-CLP chemical data in accordance with the protocols outlined on Worksheet 35. Data validation alone does not insure usability of the data. Other factors will be considered, including comparison of actual reporting limits achieved by the lab on the samples collected to the project action levels and data needs.
Describe the evaluative procedures used to assess overall measurement error associated with the project: As part of the data validation process, the validator identifies any qualifications, the bias (if known) of the data, applies qualifiers and comments on the usability of the data. Once the validation package is received from the validator it is reviewed by the Project Quality Officer or a designee. Any QA/QC problems with the validation will be discussed with the validator and laboratories. Data will be compared to the lowest of the New Jersey Groundwater Quality Criteria, NJ Maximum Contaminant Levels (MCLs), and Federal MCLs.

Identify the personnel responsible for performing the usability assessment:

The usability of the data is the responsibility of the project team. The Technical PM and Deputy PM will reconvene the project team after all data has been validated and reviewed. The data users performing the remediation design will participate in a usability assessment to determine if the data is sufficient to meet the data needs and the project DQOs, and will recommend if additional data is required. A data assessment report will be issued by the PM or his designee documenting the results of the usability assessment review performed by the project team. The report will be submitted to the USEPA and USACE for their approval and regulatory review.

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21. USEPA, 2007. Basic Manual for Electronic Data EPA Region 2, Version 1.1, November 2007.

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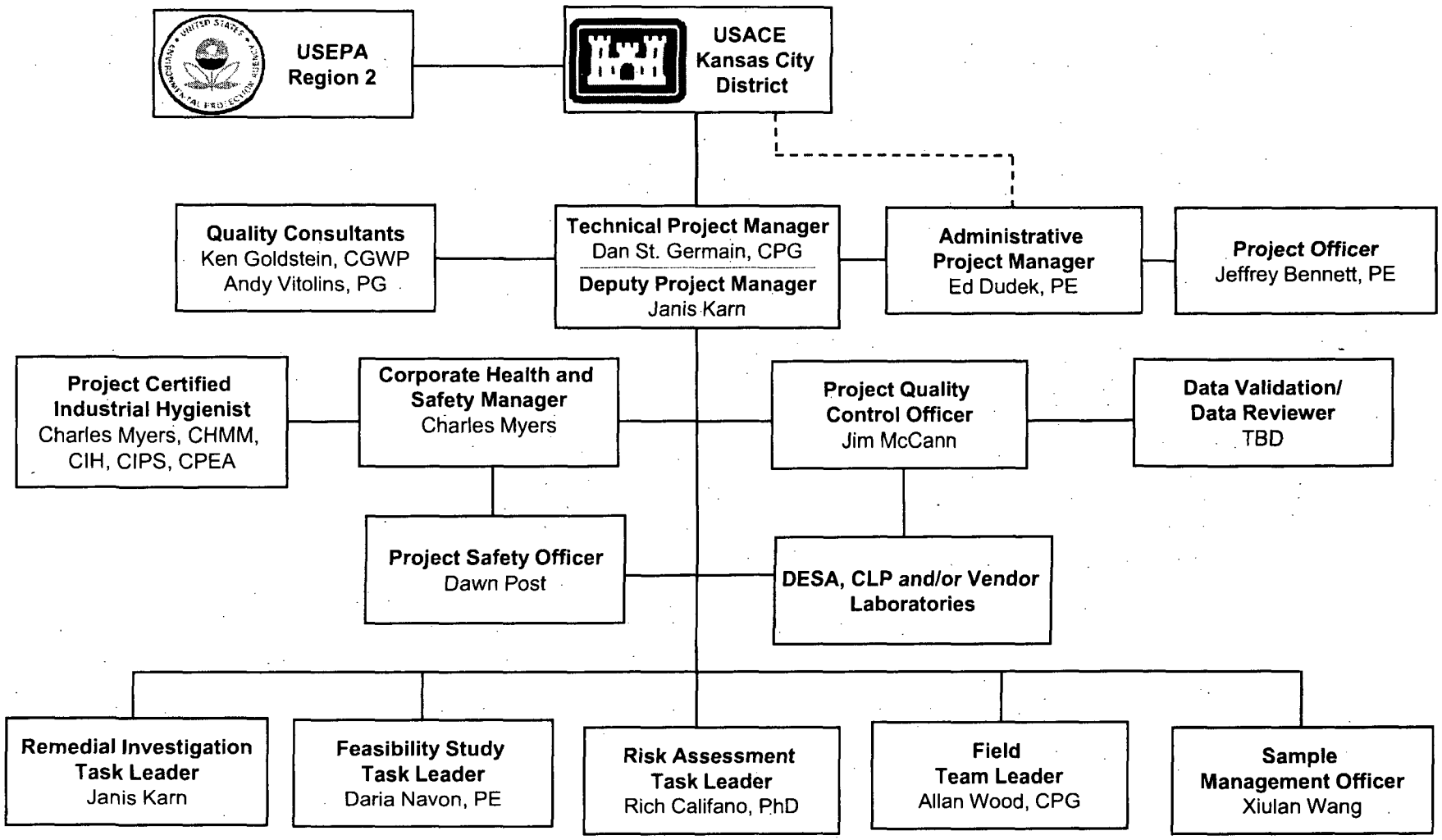
Attachments

Figure 1

Organization Chart



MALCOLM PIRNIE

Cornell-Dubilier Electronics Superfund Site OU-3 Project Team Organization Chart

FIGURE 1

Attachment 1.1

Data Quality Objectives

ATTACHMENT 1.1
DATA QUALITY OBJECTIVES FOR CORNELL-DUBILIER OU3 GROUNDWATER

Data Quality Objectives (DQOs) are used to help decision-makers collect data of the right type, quality, and quantity to support decisions. The approach to developing DQOs is an iterative one, designed to take decision makers through a strategic planning process from broad project goals through a number of refining steps toward generating environmental data that will be appropriate to making the decisions needed to reach the goals.

This document begins with a "project-level" statement of the DQOs that sets the framework for addressing the environmental problems of the study area. The project-level DQOs focus on the information that the decision-making team needs to produce a comprehensive plan for the Remedial Investigation and Feasibility Study (RI/FS) of the Cornell-Dubilier Electronics Superfund Site (Site) Operable Unit 3 (OU3) groundwater.

1.0 State the Problem

The Site is a Superfund Site and a RI/FS will be conducted in accordance with the Comprehensive Environmental Response, Compensation and Liability Act, as amended. This task is concerned with determining the nature and extent of the contamination in the groundwater of OU-3. The soils in the former Cornell-Dubilier Electronics facility are known to contain polychlorinated biphenyls (PCBs), volatile organic compounds (VOCs), semi-volatile organic compounds pesticides (SVOCs), dioxins/furans, metals and cyanide and other chemical contamination which potentially could impact the groundwater in the area. Previous analyses of groundwater samples collected from existing monitoring wells at the Site have also revealed the presence of chemical contamination such as VOCs, SVOCS, PCBs, and dioxin/furans. The objectives of the RI/FS are the following:

- Delineate the horizontal and vertical extent of groundwater contamination.
- Evaluate the lateral and vertical distribution and the amount of contaminant mass in the rock matrix by drilling rock core boreholes ('core borings'), collecting rock core samples and measuring the concentrations of VOCs and PCBs in the matrix pore water.
- Evaluate rock matrix moisture content, porosity, bulk density, solid-phase organic carbon (TOC), and rock oxidant demand by collecting rock core samples from the core borings.

- Measure the potential for presence of either light or dense non-aqueous phase liquid (LNAPL or DNAPL).
- Define the potential hydraulic connection between the water table aquifer in the Passaic Formation and Bound Brook.
- Identify water bearing zones and zones within the Passaic Formation that could be identified as confining units.
- Measure the Hydraulic Conductivity (K) of the Passaic Formation water bearing zones.
- Further evaluate exposure pathways and potential receptors to contaminants in groundwater underlying or migrating from the former CDE facility.
- Identify the level of human health risk associated with the chemical contamination in the groundwater.

2.0 Identify the Decision

To meet the objectives, the following fundamental questions will need to be answered during the investigation:

Fundamental Questions	Alternative Actions
What is the nature and extent of the groundwater contamination?	If groundwater is contaminated the nature and extent of the contamination will be evaluated when developing the remediation alternatives. The human health risk associated with the contamination detected will also be considered when considering the remedial alternatives. If groundwater is not contaminated no action may be taken at the location.
Are there sufficient data from previous investigations to make an informed determination on the nature and extent of contamination?	If the data is sufficient no further data collection may be necessary. If the data is insufficient more sampling and analyses will be performed to better characterize the nature and extent of the contamination.
Is contamination from the former Cornell-Dubilier Electronics facility	If contamination is migrating through the fractured bedrock, then the

migrating into the fractured bedrock downgradient of the former CDE facility?	nature and extent of downgradient contaminated groundwater will be characterized. If contamination is not migrating, then no further action will be necessary.
Is NAPL present?	If NAPL is present the amount will be estimated so the remedial alternatives can be evaluated. If NAPL is not present, it will not require action.

The following decisions will need to be addressed:

- Determine the nature and extent of potential chemicals of concern at the Site with respect to the groundwater of OU3.
- Determine if there is sufficient data from the previous investigations to determine the nature and extent of contamination.
- Identify the areas where there are significant data gaps in the previous groundwater data, which should be addressed.
- Determine the human health risk associated with the chemical contamination in the groundwater.

3.0 Identify the Inputs to the Decision

The following inputs are required to answer the fundamental questions identified in Step 2 above:

- Review the existing groundwater data for OU3
- Determine the sampling locations to further delineate contaminated groundwater.
- Analyze bedrock samples to determine the vertical extent of VOC and PCB contamination.
- Sample and analyze groundwater samples for Target Compound List VOCs, SVOCs, Pesticides, PCBs, Target Analyte List Metals, PCB Congeners, and Dioxins/Furans.
- Determine the optimal locations for new monitoring wells to be installed in the fractured bedrock.
- Determine what analytical methods and if field or laboratory analyses will be appropriate.

- Monitor the groundwater for VOCs, PCBs and other chemicals of potential concern to fill data gap(s), and to evaluate the extent and nature of contamination
- Perform tests to determine presence of NAPL.
- Evaluate the human health risk associated with the chemical contamination present and consider appropriate remediation options.

4.0 Define the Boundaries of the Study

The physical boundaries of the investigation have been defined by EPA as the entire area encompassing OU3. The Site is located in the Borough of South Plainfield, northern Middlesex County, in the central portion of New Jersey. The Site includes the former CDE facility located at 333 Hamilton Boulevard in South Plainfield, a fenced 26-acre facility that is bounded on the northeast by Bound Brook and the former Lehigh Valley Railroad, Perth Amboy Branch (presently Conrail); on the southeast by Bound Brook and a property used by the South Plainfield Department of Public Works; on the southwest, across Spicer Avenue, by single family residential properties; and to the northwest, across Hamilton Boulevard, by mixed residential and commercial properties. The surrounding area represents an urban environment with principally commercial and light industrial use to the northeast and east, principally residential development to the south and directly north, and mixed residential and commercial properties to the west. Most recently, the property contained numerous subdivided buildings, numbered 1 through 18. In 2007, EPA began implementing the OU2 ROD with the relocation of the tenants at the industrial park and demolition of the 18 buildings. Relocation of the tenants was completed in mid-2007, and demolition of buildings was completed in May 2008.

The CDE Superfund Site has been divided into four Operable Units (OUs) by the USEPA. Operable Unit 1 (OU1) addresses residential, commercial, and municipal properties in the vicinity of the former CDE facility. On September 30, 2003, the USEPA signed a Record of Decision (ROD) to address OU1. Operable Unit 2 (OU2) addresses contaminated soils and buildings at the former CDE facility. On September 30, 2004, the USEPA signed a ROD to address OU2. Operable Unit 3 (OU3) addresses contaminated groundwater and Operable Unit 4 (OU4) addresses the Bound Brook.

There are practical constraints and obstacles that may impede the sampling and interfere with the planned data collection. For example, the weather conditions at the site may prevent collecting samples as scheduled. Also the geological and physical conditions encountered at the sampling locations may interfere with the collection of a desired sample. If these conditions exist, it may be necessary to adjust the sampling schedule and possibly choose alternate sampling locations. Also laboratory data may not meet the quality assurance criteria or reporting limit criteria and it may be necessary to re-sample locations.

5.0 Develop a Decision Rule

The purpose of this step is to integrate the outputs from the previous steps into a statement that defines the conditions that would cause the decision-maker to choose among alternative actions. The following primary decision rules will be used to answer the fundamental questions:

- The nature and extent of the groundwater contamination will be evaluated against current federal and New Jersey standards.
- The levels of contamination, vertical and horizontal extent, impact on downgradient receptors and potential exposures will be considered when determining the risk to human health.

6.0 Specify Limits on Decision Errors

This step is to specify the decision-maker's acceptable limits on decision errors, which are used to establish appropriate performance goals for limiting uncertainty in environmental data. These acceptable limits on decision errors allow decision-makers to generate resource-effective sampling designs while limiting uncertainties in the collected data.

There are two types of decision errors applicable to estimating the true value of a population: 1) sampling design error, which occurs when the sampling design is unable to capture the complete state of natural variability over space and time; and 2) measurement error, which refers to a combination of random and systematic errors, known as the total error, can be controlled by hypothesis testing; that is, selecting the null hypothesis (H_o) and the alternative hypothesis (H_a) and testing to reject or accept H_o. The null hypothesis is the baseline condition that is presumed to be true in the absence of strong evidence to the contrary.

The null hypothesis and alternative hypothesis are as follows:

H_o: Groundwater from the site <u>does</u> contain chemical contamination with concentrations that pose a risk to human health that <u>will</u> need to be remediated.

H_a: Groundwater <u>does not</u> contain chemical contamination with concentrations that pose a risk to human health and <u>will not</u> need to be remediated.

There are two types of decision errors: 1) the false rejection decision error (false positive), or Type I error, which occurs when the null hypothesis is rejected when it is true; and 2) the false acceptance decision error (false negative), or Type II

error, which occurs when the null hypothesis is not rejected when it is false. In this case, the false rejection error is concluding that the groundwater does not contain chemical contamination with concentrations that exceeded the action levels when there are actually chemicals with concentrations that exceeded the action levels. And the false acceptance error is concluding that the groundwater does contain chemicals with concentrations that exceeded the action levels when there are actually no chemicals with concentrations that exceeded the action levels.

The consequence of the false acceptance decision error will be unnecessary expenditure of resources such as funding, personnel, and time. The consequence of the false rejection decision error is that the chemicals in the groundwater will not be remediated and will pose unacceptable risk to human health. Because of the possible severity of the false rejection decision error consequence, the false acceptance decision error is more tolerable than the false rejection decision error. The false acceptance decision error will occur when the analytical results are biased high, and the false rejection decision error will occur when the analytical results are biased low.

7.0 Optimize the Design for Obtaining Data

This step involves identifying the most resource-effective sampling and analysis design for generating data that are expected to satisfy project DQOs.

The consequence of the decision error will need to be balanced against the cost of limiting the possibility of these errors. These errors will be managed by the use of precise and accurate analytical methods and a relatively large number of samples along with duplicate samples. The large number of samples will need to be collected to minimize a false rejection decision error, and to minimize the risk of not remediating soils that have chemicals with concentrations greater than the action levels. The approach to overcome the large number of samples is to minimize duplication and unnecessary samples by evaluating the existing data, field visit observations, historical photographs, physical setting, documented past practice and process history. However, the approach to overcome the risk is to systematically perform sampling, even in areas where the chemicals may not be present.

The sampling design will consist of a nonprobabilistic sampling (judgmental sampling) methodology backed up with a probabilistic sampling (simple random sampling) methodology. In the judgmental sampling methodology, the sampling locations are based on the evaluations presented above. Typically, this is used to confirm or disprove the existence of contamination at specific locations. Judgmental samples can be used subjectively to provide information about specific areas of the site. However, to characterize areas that are not suspected of containing chemicals, a simple random sampling methodology will be performed on those areas. With simple random sampling, all areas that are not suspected of containing chemicals have an equal probably of being selected, and

each sampling point is selected independently from all other sample points. Sub-locations may also be sampled at equally spaced points depending on the size and homogeneity of the area.

To minimize unacceptable errors, laboratory analyses with a high degree of confidence and extensive QA/QC and documentation procedures will need to be utilized. Therefore, EPA's Contract laboratory Program resources will be utilized whenever possible or National Environmental Accreditation Conference (NELAC) certified subcontract laboratories will be utilized. For this Remedial Investigation, laboratory analysis will be conducted for parameters such as PCBs, dioxins and furans, volatile organics, semivolatile organics, pesticides, and metals.

Attachment 1.2

Data Needs Table

DATA NEEDS TABLE
Cornell-Dubilier Electronics Superfund Site
OU3 Groundwater RI/FS

Data Need	Data User	Parameter	Methods (Note 1)	QLs	Rationale for Analysis	Data Use
Groundwater	RI/FS Data Evalution Team including Engineers, Scientists and Risk Assessors	TCL Volatiles	SOM01.2, DESA method or SW-846 8260B	See Worksheet 15 for project QLs	Measure concentrations of volatile organics	Determine the nature and extent of groundwater contamination
		TAL Metals and Cyanide	EPA CLP SOW ILM05.4, DESA method or SW-846 methods	See Worksheet 15 for project QLs	Determine concentrations of metals	
		PCB Arolcors	EPA CLP SOW SOM01.2, DESA method or SW-846 8082	See Worksheet 15 for project QLs	Measure concentrations of PCB Aroclors	
		Chlorinated Organic Pesticides	SOM01.2, DESA method or SW-846 8081	See Worksheet 15 for project QLs	Measure concentrations of Chlorinated Organic Pesticides	
		TCL Semi-Volatiles	EPA CLP SOW SOM01.2, DESA method or SW-846 8270C	See Worksheet 15 for project QLs	Measure concentrations of semi-volatile organics	
		PCB Congeners (Note 2)	EPA 1668A or CLP SOW CBC01.1	See Worksheet 15 for project QLs	Measure concentrations of the World Health Orgnization (WHO) list toxic PCB congeners and their contribution to Toxic Equivalence (TEQ) as well as total PCBs as the sum of the PCB Congeners	
		Dioxins/Furans (Note 2)	EPA Method 1613B/8290 or CLP SOW DLM02.0	See Worksheet 15 for project QLs	Measure concentrations of Dioxin/Furan isomers and determine their contribution to TEQ	
		Total Organic Carbon (TOC) and Dissolved Organic Carbon (DOC)	SW-846-9060 (DOC on filtered sample) or equivalent	See Worksheet 15 for project QLs	Indicator of organic contamination	Determine baseline groundwater chemistry for use in remedial alternatives evaluation
		Anions: Nitrate, Sulfate and Chloride	SW-846-9056/EPA 300 and EPA 376.2 or equivalent tests	See Worksheet 15 for project QLs	Groundwater screening parameter	
		Alkalinity	SM2320B or equivalent	See Worksheet 15 for project QLs	Groundwater screening parameter	
		Ferrous Iron	SM3500Fe D	See Worksheet 15 for project QLs	Groundwater screening parameter	
		Hardness	SM2430C or equivalent	See Worksheet 15 for project QLs	Groundwater screening parameter	
		Dissolved Gases	USEPA RSK-175, SW 826 8015 or Equivalent	See Worksheet 15 for project QLs	To evaluate potential for natural attenuation	

DATA NEEDS TABLE

Cornell-Dubilier Electronics Superfund Site

OU3 Groundwater RI/FS

Data Need	Data User	Parameter	Methods (Note 1)	QLs		Data Use
Groundwater	RI/FS Evaluation Team	Test for Presence of De-Chlorination Bacteria	Microcosm Presence Absence Test	NA	To evaluate microbial activity	
		pH	Field meter	NA	Field screening parameters for low-flow sampling	Monitor groundwater sampling conditions
		Oxygen/reduction potential		NA		
		Dissolved Oxygen (DO)		NA		
		Conductivity		NA		
		Turbidity		NA		
		Temperature		NA		
Bedrock Coring (Note 3)	RI/FS Data Evaluaiton Team	Chlorinated Volatile Organics	Mobile Lab procedure for processing (crush and extracting samples) plus Vendor GC-ECD method	See Worksheet 15 for project QLs	Screen to determine the vertical and horizontial extent of volatile organic within the facility boundaries which may be migrating into bedrock	Determine the nature and extent of contamination in the rock matrix which potentially could migrate into groundwater.
		PCB Aroclors	SOM01.2, SW-846-8082 or equivalent with steps to process/crush bedrock	See Worksheet 15 for project QLs	PCBs in the bedrock could be a source of groundwater contamination.	
		Organic Carbon content	USEPA L. Kahn, ASTM D2974 or equivalent	NA	Indicator of potentail organic chemical contamination	Determine the physical and chemcial properies of the bedrock
		Chemical Oxidation Demand	ASTM D7262-07	NA	Chemical oxidant demand data is useful when considering chemcial treatment alternatives	
		Geological tests such as specific graivity, bulk denisty, % moisture and porosity (calculated)	ASTM methods	NA	Determine the physical nautre of the bedrock and the potential for adsorption and transport of contamination	

Note 1 - The actual test methods selected may vary dependent upon the capabiltiies of the assigned laboratory.

Note 2 - PCB Congeners and Dixon/Furan isomers should be analyzed from a subset of samples in which PCB Aroclors are present.

Note 3 - Final details of the methodologies for the test program are dependent upon subcontract arrangments. It has been proposed that the bedrock coring and testing be subcontracted to Stone Environmental.

Attachment 2

SOP No. 1:
Procedure to Conduct Sample Management for CLP and non-CLP Samples

Procedure to Conduct Sample Management for CLP and non-CLP Samples

I. Introduction

This guideline is to provide reference information on sample management procedures.

II. Definitions

Contract Laboratory Program (CLP) – The U.S. Environmental Protection Agency (USEPA) CLP was developed to retain laboratory services that will ensure that all environmental samples collected under the Superfund Program will be analyzed in accordance with recognized USEPA laboratory methods and quality assurance/quality control (QA/QC) procedures. See http://www.epa.gov/superfund/programs/clp/ for detailed information on the CLP.

Target Compound List (TCL) – This is a list of organic compounds typically analyzed for by the CLP. The list is broken into three subdivisions: volatiles, semi-volatiles, and pesticides/polychlorinated biphenyls (PCBs).

Target Analyte List (TAL) – This is a list of inorganic parameters typically analyzed for by the CLP. Parameters on this list include heavy metals and cyanide.

Routine Analytical Services (RAS) – Laboratory analysis for parameters included on the TCL and/or the TAL in solid and aqueous samples.

Non-RAS – Laboratory analysis for substances or parameters not included on the TCL or the TAL. Also encompasses analysis of non-soil/sediment, non-aqueous matrices, and analysis of RAS compounds using non-RAS protocols.

Trip Blanks – Trip blanks are used to check for sample contamination originating from sample transport and shipping, as well as from site conditions. Trip blanks are necessary when aqueous environmental samples are collected for volatile organic analysis.

Rinsate Blanks – Rinsate blanks, also known as field blanks, are used to check the efficacy of sampling equipment decontamination procedures. Rinsate blanks are collected for each type of non-dedicated sampling equipment used onsite. Demonstrated analyte-free water is poured over the equipment, collected in containers, and analyzed for the analytes of concern.

Field Duplicate – Field duplicates are two separate samples collected from the same sampling location. Field duplicates are used to evaluate field sampling precision and are collected at a set frequency for each analyte group. For soils, a sample aliquot is homogenized and split into two sampling containers.

Matrix Spike/Matrix Spike Duplicates (MS/MSD) – MS/MSD analysis is the process by which standard mixes of various organic TCL compounds are added to field samples prior to extraction. The sample is then split into duplicates and analyzed. The analysis is used to

evaluate the matrix effect of the sample upon the analytical methodology. Triple volume of aqueous samples for MS/MSD analysis is collected in the field, at a frequency of at least 5 percent per matrix/concentration. No extra volume is required for the soil samples.

III. Guidelines

The use of sample management procedures during sample collection is intended to ensure that samples required during the site investigation are accounted for when the project is completed. The sample management officer (SMO) is responsible for the implementation of sample management, and also for assuring that the proper QA/QC samples are collected. These goals can be achieved by adhering to the procedures and guidelines described in Sections IV, V, and VI below.

IV. Laboratory Coordination

Analysis of CLP Samples

For samples that will be analyzed through the CLP, a request must be made through the Regional Sample Control Center Coordinator (RSCC), prior to the initiation of sample collection activities, for assignment of a laboratory per USEPA Region 2 SOP No. HW-32: Standard Operating Procedure for Implementing the National Strategy for Procuring Analytical Services for All OSWER Programs, Revision 5, March 17, 2005. At this time, any requested modifications to the CLP SOWs must also be described [*e.g.*, lower detection limits, adding a parameter to the TCL or TAL list, requesting a quicker turnaround time (TAT)].

A description of how to request CLP services is included in the USEPA-issued CLP Guidance for Field Samplers, OSWER 9240.0-44, EPA 540-R-07-06, July 2007. This guidance document can be found online at the following URL:
http://www.epa.gov/superfund/programs/clp/guidance.htm.

A request for CLP services generally includes the following steps:

1. Contact the RSCC well before the sampling event to discuss the CLP sample submission requirements. For USEPA Region 2, the RSCC is Jennifer Feranda, who can be contacted at 732-321-6687.

2. Fill out the RSCC request forms.

3. RSCC will contact the originator of the request with the Case Number and assigned laboratories. At times, the EPA Region 2 Division of Environmental Science and Assessment (DESA) Laboratory may choose to perform all or part of the analyses requested.

4. For a long-term project, weekly contact should be maintained with the RSCC.

For samples submitted to a CLP laboratory, Forms II Lite software must be used by the samplers

to record the sample information and to create CLP sample numbers and the required COC forms. Assistance with Forms II Lite can be obtained by calling the help desk at 703-818-4200 or by visiting the Forms II Lite web site at http://dyncsdao1.fedcsc.com/itg/forms2lite/.

Analysis of non-CLP Samples

Subcontractor laboratory(ies) will be procured for the project to conduct analysis of non-CLP parameters, as required. Weekly contact should be maintained with these laboratories to keep them informed of the status of the sampling program.

V. Preparation of Sample Containers

When CLP and/or DESA laboratory(ies) are used, Malcolm Pirnie will purchase certified clean sample containers from an approved supplier. Copies of these certifications should be kept in the project files for future reference. When non-CLP laboratory(ies) are used, arrangements should be made with the subcontract laboratory to provide suitable, clean sample containers appropriate for each test.

It should be noted that non-CLP subcontract labs usually also provide coolers, but when samples are being sent to an assigned CLP lab or to the USEPA Region 2 DESA Laboratory, the samplers must make arrangements to obtain the coolers through the Malcolm Pirnie Equipment Facility prior to the sampling event. The Malcolm Pirnie Equipment Facility is located in the Tall Pines Industrial Park, 382 Route 59 Section 286, Monsey, NY 10952 (Contact Max Bateman at 845-357-0965).

VI. QA/QC Samples

Rinsate Blanks

Rinsate blanks (or Equipment Blanks) are collected for each type of equipment used to collect samples at the frequency specified in the Quality Assurance Project Plan (QAPP). It is recommended that rinsates be collected at a minimum frequency of one per week and at a maximum frequency of one rinsate per location per day. Decontaminated equipment must be properly stored in an area and in a manner that will prevent cross contamination. The analyte-free water required for equipment decontamination and rinsate blank collection will be purchased by Malcolm Pirnie from an approved supplier. Additional information on Rinsate Blanks is provided in SOP No. 5.

Where possible, composite rinsates will be collected from all equipment associated with a particular matrix for analysis of non-volatile parameters.

Rinsate blanks are collected using the following procedure:

1. Pour demonstrated analyte-free water over clean equipment.

2. Collect the water into sample containers.

3. Preserve, package, and ship to the analytical laboratory for analysis.

Field Duplicates

Samples for duplicate analysis are collected in the field for each matrix sampled, at the frequency described in QAPP. Sufficient quantity of matrix must be collected from the same sample location to fill a duplicate set of sample containers. The duplicate volume is shipped to the laboratory under a separate, "blind" sample ID (or CLP sample number).

For soil/sediment samples the volatile organic fraction is collected as co-located grab samples, while the non-volatile fraction is homogenized prior to collection. For dust and wipe samples field duplicate samples should be collected from an adjacent area of equal size.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) and Matrix Spike/Matrix Duplicate (MS/MD)

MS/MSD analysis for organics are typically conducted for 1 in 20 environmental samples per concentration/matrix. Three times the total volume is necessary for collection of aqueous MS/MSD samples. No extra volume is required for the soil samples. MS/MSDs are noted as such on the chain of custody (COC).

VII. Sample Documentation, Packaging, and Shipping Procedures

One or more members of the field team should be designated as SMOs. The SMO will bear the ultimate responsibility for the documentation, packaging, and shipping of the samples. These procedures are outlined in the sections below.

Pre-Field Activities

If samples are being submitted to non-CLP labs, no special arrangements are required prior to the initiation of fieldwork. If samples are being submitted to CLP-assigned labs, the USEPA RSCC must be contacted for information regarding the following items prior to going out into the field:

- CLP Sample Numbers.
- SMO-assigned Case Numbers.
- Traffic Report/COC (TR/COC) Records.
- Chain-of-custody seals.

Refer to the USEPA CLP Guidance for Field Samplers, Chapter 2 for detailed instructions regarding pre-field activities. This document can be found in QAPP Attachment 11.

Documentation and Chain of Custody

For documentation purposes, the field team will enter information about each sample into the field logbook as they collect the sample. The information recorded should include but not be limited to the following:

- The MPI assigned sample number (sample ID).
- The CLP Number and Case Number (for CLP samples, only);.

- Method of sample collection.
- Sample date.
- Time of collection.
- Preservative(s) used.
- Analyses required.
- Sample type.
- Associated rinsate(s).
- Sampler's initials.

The same information should be included on the sample bottle label(s).

Once all of the samples have been collected, they should be grouped for shipment by receiving laboratory and the COC filled out. How the COC is set up and filled out varies between CLP and non-CLP laboratories:

- When samples are being shipped to a non-CLP laboratory, COC forms provided by the laboratory can be used.

- When samples are being shipped to a CLP-assigned laboratory, the COC and sample labels should be created and printed using the FORMS II Lite software. The labels can be printed in the field using a laptop PC and printer, It is also possible to pre-print the labels using the FROMS II Lite prior to going to the field. Refer to the FORMS II Lite User's Guide for instructions on the use of FORMS II Lite software. FORMS II Lite can also be used to create COCs for non-CLP samples. If circumstances require that handwritten sample labels must be used for CLP samples, follow the requirements in checklist Appendix E-4 of the USEPA's CLP Guidance for Field Samplers.

Before placing a sample in a cooler, the sample label should be covered with clear tape. The sample labels should contain the following information:

- Malcolm Pirnie-designated sample number.

- For CLP samples only, the assigned CLP Sample Number and CLP Case Number must be recorded on each sample taken during a sampling event.

- The month, day, and year the sample was collected.

- The type of analysis requested.

- The type of preservation performed in the field.

CLP Documentation Requirements

A full description of the CLP documentation requirements are found in Sections 1.4 and 3.2 of USEPA's CLP Guidance for Field Samplers. Samplers must:

- Record the CLP Number and Case Number on each sample bottle.
- Complete the TR/COC Record using the Forms II Lite software, making sure to indicate on the TR/COC Record if samples require the use of a Modified Analysis.
- Complete and attached sample labels.
- Complete and attach tags to meet Regional requirements.
- Complete and attach custody seals to meet Regional requirements.
- Complete field operations records, as necessary.

Contact the RSCC before the sampling event for information regarding the assigned CLP Sample Numbers, SMO-assigned Case Numbers, TR/COC Records and chain-of custody seals for sampling events. Under no circumstances should the site name appear on any documentation that is sent to the laboratory when shipping CLP samples.

Packaging and Shipping Samples

Below are guidelines for packaging and shipping samples. A checklist for packing CLP samples for shipment can also be found in Appendix E-6 of the USEPA's CLP Guidance for Field Samplers.

1. Make sure the caps on the sample bottles are tightly sealed. Wipe down the outside of all of the sample bottles.
2. Preserve the samples according to requirements in the QAPP.
3. For CLP, apply one custody seal around the circumference of the container or over the cap and onto the sides of the container. The custody seal must applied to sample containers in such a manner as to reveal if the container was opened during transit.
4. Place containers in its individual zip-lock bag. Eliminate extra air space from the bag before sealing.
5. For CLP samples, place the associated sample tag into the zip-lock bag with the sample.
6. Prepare the shipping container (usually a cooler) so that no leakage can occur during shipping. Securely seal all valves using duct tape on both the inside and the outside of the cooler, and line with plastic or a larger garbage bag. Only coolers that conform to the general design requirements in 49 CFR 173.410 should be used for shipment.
7. Put 1-2 inches of packing material in the bottom of the coolers, and then place the samples into the garbage bag in the cooler.

8. Surround the sample bottles with bags of ice (only the samples that need to be cooled – refer to the QAPP requirements for sample preservation. Prior to placement in the cooler, remove the ice from the original bag and re-pack into doubled zip lock bags. Use enough ice to ensure that the proper temperature (2-6°C) is maintained during transport. Place a temperature blank (40-mL vial filled with DI water and labeled as "temperature blank") into the cooler.

9. Place packing material over and around the sample bottles. Sufficient packing material must be used so the bottles will not move or break during transport.

10. Once the samples are packed, close and securely tie or tape the plastic or garbage bag.

11. Fill in the "relinquished by" and "received by" sections of the COC prior to shipment. The SMO should fill in the name of the commercial shipper (courier) and add the airbill number, if applicable, as the courier generally will not sign the COC.

12. Make enough copies of the COC form(s) to allow for placement of one set in each cooler that is not the lead cooler. Seal the original COC form in a ziplock bag and tape to the inside of the lead cooler, and seal one copy of the COC form(s) in a ziplock bag and place in each of the other cooler(s).

13. For CLP samples, retain one copy of the COC form for the SMO and one copy for transmission to the RSCC (send within 1 day). For non-CLP samples, retain one copy of the COC form for the SMO.

14. Close the cooler and seal with strapping tape. If visibly dirty, wipe down the outside of the cooler. Apply two signed and dated custody seals to the cooler diagonally across from each other where the cooler lid meets the cooler. Apply the custody seals in such a manner as to reveal if the cooler was opened during transit.

15. Place an address label on the outside of each cooler and cover with clear tape. If more than one cooler is being sent to one destination, label each cooler appropriately, *e.g.*, 1 of X, 2 of X, etc. Attach the airbill to one of the coolers. Generally, the samples are sent via overnight carrier for next day delivery. This should be confirmed with the Field Team Leader.

16. Notify the laboratory of the shipment before 9:00 a.m. on the day after shipping. For CLP samples, fill out the Sample Shipping Call-In Form. Call or fax the shipping information to RSCC by 9:00 am the following morning. For non-CLP samples, follow the notification system agreed to in the subcontract.

17. Instructions for shipping and packaging CLP samples can be found in the CLP Guidance for Field Samplers. Appendix E of the guidance contains sampling checklist which should be followed.

Note: Some samples have very short holding times. In some limited instances, the samples may need to be either hand delivered to a laboratory or picked up by the laboratory's courier service.

VIII. References

USEPA 2007, Introduction to the Analytical Services Branch (ABS) Contract Laboratory Program, EPA 540-R-07-02, OSWER 9240.0-42, January 2007

USEPA 2007, Contract Laboratory Program Guidance for Field Samplers, OSWER 9240.0-44, EPA 540-R-07-06, July 2007

FORMS II Lite, Version 5.1, User's Guide

Attachment 3

SOP No. 2:
Procedure to Conduct Sample Preservation

Procedure to Conduct Sample Preservation

I. Introduction

This guideline is to provide reference information on the accepted methods of sample preservation. Not all portions of this guideline may be applicable to the current sampling activities of this project.

II. Materials

Preservatives:

a. 1:1 HCl - (Hydrochloric Acid/Deionized Water)
b. HNO_3 - full strength (Nitric Acid)
c. NaOH - 10 N (Sodium Hydroxide)
d. H_2SO_4 - full strength (Sulfuric Acid)
f. Zinc Acetate
g.. Ice

Additional Materials:

a. Disposable Pasteur pipettes
b. Pipette pumps - 10 mL or 2 mL
c. Latex pipette bulbs
d. Squeeze bottle with deionized water
e. Clear wide mouth glass jar for water pipette
f. Paper towels
g. Lead acetate paper
h. Cadmium nitrate or cadmium carbonate (if using lead acetate paper)
i. Potassium iodide - starch test paper (KI-starch paper)
j. Ascorbic Acid (if using KI starch paper)
k. Filter paper
l. Filter funnels (disposable or decontaminated)
m. Filter vessel with hand pump
n. pH paper
o. Scale
p. Cooler(s)

Safety Materials:

a. safety glasses
b. nitrile or latex gloves
c. labcoats or aprons
d. MSDS sheets for all of the chemicals
e. Eyewash

III. Discussion

Complete and unequivocal preservation of samples is a practical impossibility. At best, preservation techniques slow down the chemical and biological changes that inevitably continue after the sample is removed from the parent source. The changes that take place in a sample are either chemical or biological. In the former case, certain changes occur in the chemical structure of the constituents that are a function of physical conditions. Metal cations may precipitate as hydroxides or form complexes with other constituents; cations or anions may change valence states under certain reducing or oxidizing conditions; other constituents may dissolve or volatilize with the passage of time; and metal cations may also adsorb onto surfaces (glass, plastic, quartz, *etc.*). Biological changes taking place in a sample may change the valence of an element or a radical to a different valence. Soluble constituents may be converted to organically bound materials in cell structures, or cell lysis may result in release of cellular material into solution. The well known nitrogen and phosphorus cycles are examples of biological influence on sample composition. Therefore, as a general rule, it is best to analyze the samples as soon as possible after collection. This is especially true when the analyte concentration is expected to be in the low ug/L range.

Methods of preservation are relatively limited and are intended generally to (1) retard biological action, (2) retard hydrolysis of chemical compounds and complexes, (3) reduce volatility of constituents, and (4) reduce absorption effects. Preservation methods not outlined below are generally limited to pH control, chemical addition, refrigeration, and freezing.

IV. Guidelines

All Samples

With few exceptions, most samples need to be cooled to between 2-6 °C immediately after sample collection.

Preserving non-Soil/Sediment Solid Matrix Samples

Equipment

This procedure should be used when preserving concrete core samples. No preservation requirements are indicated for asbestos, paint chip, or window caulking samples until arrival at the laboratory. No chemical preservation is required for solid matrix samples. Field personnel should have the following materials on hand for sample preservation:

1. Ice
2. Coolers

Preservation Procedure

1. Each cement core and associated chips/debris should be stored double-bagged in re-sealable plastic bags marked with the required sample information.
2. Place each sample in a cooler with enough ice to maintain the sample temperature between 2 and 6 °C prior to shipment to the analytical laboratory.
3. The samples should be stored between 2 and 6 °C until the time of analysis.

Preserving Aqueous Samples for Volatile Organic Compound (VOC) Analysis and Samples for Methane, Ethane and Ethene Analysis

Equipment

Field personnel should take the following materials to the sampling locations:

1. One 40-mL VOA vial containing 1:1 HCl.

   The 1:1 HCl should be transferred on site from a 1-liter plastic-coated glass bottle to one properly labeled 40-mL glass vial by using a glass funnel. Hand and eye protection must be worn during the transfer and handling of hydrochloric acid. Field personnel must attempt to keep the 40 mL vial in an upright position during field sampling. The 40-mL vial must be kept in a plastic ziplock bag.

2. Plastic ziplock bag containing pH indicator strips for each sampling location.
3. Nitrile or latex gloves
4. Eye protection
5. Plastic ziplock bag for disposal of used pH indicator strips and latex gloves.

Preservation Procedures

1. For each different type of aqueous sample to be collected, a test sample must be preserved to determine if the preservation procedure will cause an adverse reaction. Note that a test vial must also be collected when the temperature changes (*e.g.*, each season) and whenever a sample is significantly different in appearance than the test sample. First, fill a test vial one-half full with the sample matrix to be collected. Note the color and clarity of the sample.
2. Test the pH by inserting one pH paper strip into the test vial. If the pH is less than 2.0, as indicated by a blue color on the strip, collect the samples without acidifying. Document this in the field application. The field sample management officer must document the sample as not preserved on the COC. If the pH is greater than 2.0, continue to Step 3. The pH indicator paper strip should be put into a plastic bag for later disposal.
3. Dispense 10 drops of 1:1 HCl from the pipette. Tap the vial gently to mix. If color develops, precipitates form, effervescing occurs, or an exothermic reaction (heat generation determined by holding the vial firmly) occurs, do not acidify the samples and document the reason for not acidifying in the field application. This information should also be included on the COC. If no adverse reactions occur when acid is added to the sample, proceed to Step 4.
4. Test the pH of the sample. If the pH is less than 2.0, proceed to Step 5. If the pH is greater than 2.0, add 1:1 HCl a few drops at a time (keeping count) until the pH is less than 2.0; then proceed to Step 5.
5. Fill the test vial with sample until the vial is nearly full to the top. Gently tap the side of the vial to mix, and test the pH of the sample. If the pH is less than 2.0 proceed to the next step.
6. If the pH is greater than 2.0, again add 1:1 HCl a few drops at a time (keeping count) until the pH falls below 2.0. Proceed to the next step.
7. Note the amount of 1:1 HCl added to the test vial. Add this amount of 1:1 HCl to all of the 40 mL vials, using the same glass pipette, before collecting the samples. These samples are

then packaged and cooled to between 2-6 °C prior to shipping to the CLP laboratory or the assigned subcontract laboratory.

8. Store the samples between 2-6 °C until the time of analysis.
9. Properly dispose of the test vials and all used sample preservation equipment.

Preserving Aqueous Inorganic Samples with Acid

1. Add the acid to the sample using a pipette. Typically, depending on the size of the pipette and the original pH of the sample, approximately ½ a pipette of acid is required per liter of sample. Recap the sample bottle and turn it gently upside down to mix the contents.
2. Check the pH by pouring a few drops of the sample over the pH paper; do no dip the pH paper directly into the sample. The pH of the sample should be < 2.
3. If the sample contains a significant particulate fraction, acidification without filtration could result in deceptively high values for the aqueous sample. Varying amounts of particulate matter can also give large differences in metal values for duplicate acidified aqueous samples. Observation, therefore, should be made and recorded in the field application and also noted on the COC. If an obvious change is observed during sample preservation, which may bias the results, the Site Quality Control Officer (SQO) should be consulted.
4. If the pH is still > 2, repeat steps 1 and 2 until the pH is < 2.
5. Store the samples at 2-6 °C until the time of analysis.

Preserving Aqueous Samples for Sulfide Analyses with Zinc Acetate and NaOH to pH >9.

1. Wear hand and eye protection. To a 500 mL samples collected in a glass bottle add 2 mL of Zinc Acetate with a pipette.
2. Add NaOH to the sample using a pipette. Hand and eye protection must be worn during the transfer and handling of NaOH. Typically, depending on the original pH of the sample, approximately 1 mL of NaOH per 500 mL of sample is required. Recap the sample bottle and turn it gently upside down to mix the contents.
3. Check the pH by pouring an aliquot of the sample over the pH paper; do not dip the pH paper directly into the sample. The pH of the sample should be > 9.
4. If the pH is still < 9, repeat steps 2 and 3 until the pH is > 9.
5. Store the samples at 2-6 °C until the time of analysis.

Preserving Aqueous Samples Collected for Total Organic Carbon Analyses with Sulfuric Acid.

1. Hand and eye protection must be worn during the transfer and handling of H_2SO_4
2. Add H_2SO_4 the sample using a pipette. Recap the sample bottle and turn it gently upside down to mix the contents.
3. Check the pH by pouring an aliquot of the sample over the pH paper; do not dip the pH paper directly into the sample. The pH of the sample should be < 2.
4. If the pH is still greater than 2, repeat steps 2 and 3 until the pH is less than 2.
5. Store the samples at 2-6 °C until the time of analysis.

Notes:

1. Refer to the sample preservation table in the QAPP Worksheet #19 for specific sample preservation requirements.
2. When collecting samples for the EPA Contract Laboratory Program (CLP), refer to the CLP Guidance for Field Samplers, which can be found at http://www.epa.gov/superfund/programs/clp/guidance.htm for more detailed information on preserving and shipping CLP samples.
3. As an alternative to preserving the samples employing chemicals in the field, it is acceptable to collect the samples in sample containers prepared by the laboratory with the appropriate preservatives already added to the sample containers. The pH of the samples should still be checked to insure they are in the correct range. Eye protection and gloves must still be worn during sample collection.

Attachment 4

Example of Chain of Custody

ATTACHMENT 4. EXAMPLE OF NON-CLP CHAIN OF CUSTODY FORM

Chain of Custody Record	TAT:	TAL Metals: _____ VOCs _____ Cyanide: _____ SVOCs _____ PCB Aroclors _____ Other: _____	Case No: _____ DAS No: _____ SDG No: _____

	Chain of Custody Record		For Lab Use Only
Date Shipped:		Sampler Signature:	
Carrier Name:	Relinquished by: (Date/Time)	Reveived by: (Date/Time)	
Airbill:	1.	1.	Lab Contract No:
Shipped to:	2.	2.	Unit Price:
	3.	3.	Transfer to:
	4.	4.	

CHAIN OF CUSTODY

Field Sample ID	Matrix	Sampler	Conc/Type	Preservative	Location	Collection Date/Time	TAL Metals including mercury	Cyanide	PCB Arolors	Pesticides	Dioxin/Furans	PCB Congeners	VOCs	SVOCs			Remarks	For Lab Use Only Sample Condition Upon Receipt
1																		
2																		
3																		
4																		
5																		
6																		
7																		
8																		
9																		
10																		
11																		
12																		
13																		

Shipment for case complete (Y or N)?	Sample to be used for laboratory QC:	Additional Sampler Signature(s):		Cooler Temperature upon receipt:	Chain of Custody Seal Number:	
Concentration: L = low, M = low/medium, H = High		Type/Designate: C = Composite, G = Grab			Custody Seal Intact?	Shipment Iced?

Attachment 5

Field Modification Form

FIELD MODIFICATION FORM
FOR
CORNELL-DUBILIER ELECTRONICS SUPERFUND SITE OU-3
MALCOLM PIRNIE, INC.

Date:

Document:

Activity:

Requested Modification:

Rationale:

Attachments:

Malcolm Pirnie Project Manager: _____

Malcolm Pirnie Deputy Project Manager: _____

Malcolm Pirnie Project Quality Control Officer: _____

Attachment 6

SOP No. 3: Technical System Audit

Procedure to Conduct a Technical System Audit (TSA)

I. Introduction

This guideline is to provide information on TSAs to be conducted for the Cornell Dubilier Electronics Superfund Site.

II. Guidelines

The purpose of the TSA is to ensure that the sampling team adheres to the guidelines contained in the Field Sampling Plan (FSP), and Quality Assurance Project Plan (QAPP). Prior to conducting the audit, a copy of the FSP and QAPP will be reviewed by the auditor Project Quality Control (QC) Officer or designee). During the TSA the sampling team's adherence to these guidelines will be verified and any deficiencies from the guidelines will be documented. The effect of the deficiencies will be noted, and any necessary corrective actions will be instituted.

Prior to conducting the audit, the auditor will contact the Deputy Project Manager (DPM) to discuss the audit. This will ensure that the sampling team is properly prepared for the sampling event.

A. Conducting the TSA

The following procedures will be used to conduct the TSA:

1) The auditor will bring the following equipment/documents into the field:

- Copy of the FSP and QAPP and any relevant memos, correspondence or addenda
- TSA audit checklist
- Digital camera

2) The following aspects of the sampling event will be audited:

- QA/QC samples
- Sampling methodologies
- Field documentation
- Sample management tasks
- Decontamination procedures

B. Corrective Action in the Field

Besides observing and reporting, the auditor is responsible for initiating steps for the start-up of corrective action procedures.

If the auditor witnesses discrepancies in the field between the FSP and QAPP and the performance of the sampling team, the auditor has several options available for corrective action. These options are dependent upon the type of deficiencies observed.

Deficiencies observed and the corrective action taken must be documented in the auditor's log book.

- Minor Deficiencies

  Minor deficiencies are problems where the impact, if any, to the data can be easily eliminated and the deficiency can be corrected or the procedure repeated to achieve the desired result. Minor deficiencies that are observed by the auditor will immediately be brought to the attention of the field team. The auditor and the field team will discuss the problem and agree upon what corrective action is necessary. This will allow for the deficiencies to be corrected immediately in the field.

- Major Deficiencies

  Major deficiencies are events or procedures that substantially deviate from approved work plans, will result in increased project costs not previously approved, or will significantly impact the quality of the data.

  Upon witnessing a major deficiency, the auditor will temporarily stop all related site work and will inform the field team of the problem. The auditor and field team will discuss the deficiency as well as what steps are necessary for corrective action. If the deficiency can be corrected in the field, the auditor may allow work to resume as long as all necessary corrective actions are taken. Information regarding the nature of the deficiency as well as the corrective action(s) taken will immediately be transmitted to the USACE Project Manager (USACE PM), the Malcolm Pirnie Project Manager (PM), and the DPM

If the deficiency cannot be corrected in the field, a Stop-Work Order will be issued until appropriate measures can be taken to correct the problem. A written report of the major deficiencies will be prepared by the Project QC Officer and submitted to the USACE PM, the Malcolm Pirnie PM, and the DPM. The Stop-Work Order will remain in effect until the proper corrective action(s) can be implemented.

C. Preparation of a TSA Report

The TSA report provides a means of relaying the events of a sampling episode to key personnel. These events could possibly affect the sample integrity (QA/QC) and therefore, are important to the decisions made regarding analytical data. This report will identify any deficiencies found in the field and will outline the corrective actions that were recommended/implemented to address any minor deficiencies observed. The field audit report will also recommend appropriate corrective actions for any major deficiency noted. Follow-up reports describing completed corrective actions which addressed major deficiencies will be submitted by the Malcolm Pirnie PM to the USACE PM.

A quality control field audit report will usually contain the following information:

- Date and location of field audit
- Sample matrices witnessed
- Name of personnel conducting the sampling
- Summary of sample methodology
- Description of any infractions that occurred and the corrective actions taken
- Conclusions
- Recommendations
- Quality control field audit checklist

The following are examples of audit checklists:

QUALITY CONTROL FIELD AUDIT REPORT

SUMMARY INFORMATION

1. PROJECT NAME: _____

2. PROJECT ADDRESS: _____

_____

_____

3. INVESTIGATION _BUILDING MATERIALS _____ SOILS_____

_PRE-REMEDIAL ACTION OF VICINITY PROPERTY: _____

4. DATE(S) OF QC FIELD AUDIT _____

5. AUDITOR'S NAME _____PHONE_____

6. FACILITY CONTACT _____PHONE_____

7. CONTRACTOR CONTACT_____PHONE_____

8. PERSONNEL ON-SITE

NAME	REPRESENTING	PHONE

9. AUDITOR'S COMMENTS

_____

_____

_____

_____

10. WEATHER CONDITIONS

SUNNY ; PARTLY SUNNY ; PARTLY CLOUDY ; CLOUDY ; RAIN ; DRIZZLE ; SNOW ; SLEET

TEMPERATURE_____ WIND SPEED_____ WIND DIRECTION_____

QUALITY CONTROL FIELD AUDIT REPORT, Continued

11. LEVEL OF PERSONNEL PROTECTION REQUIRED IN WORK PLAN

A B C D

LEVEL OF PERSONNEL PROTECTION ACTUALLY DONNED:

A B C D

12. FIELD SURVEY EQUIPMENT

INSTRUMENT	MODEL	CALIBRATION CHECK	CALIBRATION STANDARD	SPAN SETTING
CONDUCTIVITY METER	_____	_____	_____	_____
DISSOLVED O_2 METER	_____	_____	_____	_____
PH METER	_____	_____	_____	_____
COMBUSTIBLE GAS INDICATOR (LEL/O_2)	_____	_____	_____	_____
FLAME IONIZATION DETECTOR (OVA)	_____	_____	_____	_____
PHOTOIONIZATION DETECTOR (HNU)	_____	_____	_____	_____
TOTAL GAS INDICATOR (CO, HS)	_____	_____	_____	_____
OTHER _____	_____	_____	_____	_____

OBSERVATIONS _____

_____

_____

13. DID THE SAMPLING TEAM TAKE PERIODIC SURVEYS OF THE AMBIENT AIR CONDITIONS?

YES NO N/A

14. DID THE SAMPLING TEAM PROVIDE A DECON ZONE DESIGNATING CLEAN AND CONTAMINATED AREAS?

YES NO N/A

15. WERE PHOTOGRAPHS TAKEN? YES NO

16. AUDITOR'S COMMENTS

_____

_____

_____

NON-AQUEOUS SAMPLE INFORMATION

1. NON-AQUEOUS MATRIX SAMPLED:

SOIL SEDIMENT SLUDGE CHEMICAL SOLIDS WASTE PILE

OTHER _____

2. TYPE OF SAMPLE: GRAB COMPOSITE IF COMPOSITE - SAMPLES/COMPOSITE _____

3. WAS THE VOA SAMPLE COLLECTED FIRST FROM A DISCRETE LOCATION PRIOR TO HOMOGENIZATION?

YES NO N/A

4. WAS THE SAMPLE HOMOGENIZED PRIOR TO ACQUISITION INTO THE SAMPLE CONTAINERS? YESNO

5. TYPE OF SAMPLING EQUIPMENT:

	MATERIAL OF CONSTRUCTION			
	STAINLESS STEEL	TEFLON	GLASS	OTHER
SPOON/SPATULA	_____	_____	_____	_____
TROWEL/SCOOP	_____	_____	_____	_____
BUCKET AUGER	_____	_____	_____	_____
SPLIT SPOON	_____	_____	_____	_____
SHELBY TUBE	_____	_____	_____	_____
TRIER	_____	_____	_____	_____
PONAR DREDGE	_____	_____	_____	_____

6. WAS THE DRILL RIG, AUGER FLIGHTS, RODS, ETC. DECONTAMINATED ACCORDING TO STANDARD PROCEDURES BETWEEN EACH SAMPLE LOCATION? YESNO N/A

IF NO, METHOD OF DECONTAMINATION _____

7. IF MUD ROTARY DRILLING WAS UTILIZED WHAT WAS THE SOURCE OF THE WATER? _____

8. WAS THE SAMPLING EQUIPMENT DEDICATED? YES_____ NO _____

9. WAS THE SAMPLING EQUIPMENT: LAB DECONTAMINATED? FIELD DECONTAMINATED?

10. WAS THE SAMPLING EQUIPMENT DECONTAMINATED ACCORDING TO STANDARD PROCEDURES?

YES NO IF NO, METHOD OF DECONTAMINATION: _____

11. WAS THE DECONTAMINATION AREA LOCATED AWAY FROM THE SOURCE OF CONTAMINATION? YES NO N/A

12. ARE DISPOSABLE GLOVES WORN AND CHANGED BETWEEN EACH SAMPLE LOCATION? YES NO N/A

13. AUDITOR'S COMMENTS

_____

_____

QA/QC INFORMATION

1. LABORATORY:

NAME _____ PHONE _____

CONTACT PERSON _____

CLP CLP CAPABLE CERTIFIED OTHER _____

2. SAMPLE INFORMATION:

MATRIX	PARAMETER	PRESERVATIVE	CONTAINER DESCRIPTION

3. WHAT ORDER BY ANALYTICAL PARAMETER ARE SAMPLES COLLECTED: _____

_____

4. FIELD BLANKS: YES NO N/A FREQUENCY _____

METHOD: _____

WAS IDENTICAL BOTTLE TO BOTTLE TRANSFER OF WATER UTILIZED? YES NO

5. TRIP BLANKS: YES NO N/A FREQUENCY _____

6. WHAT WAS THE SOURCE OF THE BLANK WATER? LABORATORY DEMONSTRATED ANALYTE-FREE
OTHER_____

7. SAMPLE PACKAGING AND HANDLING:

SAMPLE CONTAINERS LABELED	YES	NO	N/A
COC FORMS COMPLETED	YES	NO	N/A
CUSTODY SEALS	YES	NO	N/A
SAMPLES PRESERVED TO 4^{B}C:	YES	NO	N/A

8. AUDITOR'S COMMENTS

_____

_____

_____

_____

QC Audit Checklist For Bedrock Sampling
Reference Stone Environmental Inc. SOP SEI-6.37)

1. Were the Cores covered before processing and processed quickly to minimize the loss of VOCs (per SEI-6.37 Section 4.2)?

   YES NO NA

   Comment: _____

2. Were the Core Subsamples for chlorinated volatile organic compounds collected from the appropriate locations in the core per as described in the FSP/QAPP?

   YES NO NA

   Comment: _____

3. Were the core subsamples collected for chlorinated volatile organic compounds wrapped and labeled properly per the SOP/QAPP?

   YES NO NA

   Comment: _____

4. Were the core subsamples collected for chlorinated volatile organic compounds crushed and preserved per the Stone Environmental SOP?

   YES NO NA

   Comment: _____

5. Were the cells used to crush the bedrock cores adequately decontaminated before use?

   YES NO NA

   Comment: _____

6. Did the Mobile Laboratory calibrate their equipment, run blanks, perform QC analysis and follow their lab SOP, while analyzing the subsamples for the chlorinated volatile organic compounds?

   YES NO NA

   Comment: _____

   _____

7. Did the Mobile Laboratory appear to rapidly process the samples and report the analytical data in a timely manner?

   YES NO NA

   Comment: _____

   _____

8. Were the Core Subsamples collected for PCB Aroclors per the FSP/QAPP?

   YES NO NA

   Comment: _____

9. Were the Core Subsamples collected for Physical Property Tests (such Bulk Density, Moisture and Specific Gravity) per the FSP/QAPP?

   YES NO NA

   Comment: _____

10. Were the rock barrels cleaned using clean water and with a pressure washer between runs?

YES NO NA

Comment: _____

11. Were the sampling and trimming chisels decontaminated before re-use?

YES NO NA

Comment: _____

12. Were the appropriate duplicate samples collected at a rate of 1 in 20?

YES NO NA

Comment: _____

13. Were the Equipment Blanks collected per the SOP/QAPP?

YES NO NA

Comment: _____

14. Were the Chain of Custody forms properly filled out?

YES NO NA

Comment: _____

Additonal Auditor Comments:

_____

_____

_____

Attachment 7

SOP No. 5:
Procedure to Collecting of Equipment Blanks

Procedure to Conduct Collection of Equipment Blanks

I. Introduction

This guideline provides procedures on the collection of equipment blanks. Equipment blanks are QA/QC samples intended to provide control over the collection of environmental measurements and information on subsequent validation, review, and interpretation of generated data.

II. Definition

Equipment blanks are also called rinse blanks or rinsate blanks. Equipment blanks are rinse water samples taken from clean sample containers and sampling equipment to determine if residual contaminants are present in the equipment prior to sampling. If contamination is present, the decontamination procedure or source of equipment must be modified to eliminate non-sample contamination. Frequency of equipment blanks will be based upon the requirements in the QAPP, but typically should be collected at a rate of one per 20 environmental samples or per decontamination event, whichever is more frequent but no more than one per day.

The following is the definition of equipment blank from the USEPA publication EPA-505-B-04-900A, Uniform Federal Policy for Quality Assurance Project Plans, March 2005.

Equipment blanks – A sample of water free of measurable contaminants poured over or through decontaminated field sampling equipment that is considered ready to collect or process an additional sample. The purpose of this blank is to assess the adequacy of the decontamination process.

III. Safety

Wear goggles and gloves when working with and collecting the blank rinse water samples. Also, conform to other safety and clean-hands practices. Perform the procedure with a helper if possible. Clean up all spilled water and sample preservatives immediately.

IV. Supplies

1. Sample bottles and labels.
2. Sample preservatives
3. Plastic sheeting

4. Aluminum foil
5. Gloves
6. Goggles
7. Paper towels
8. pH indicator paper
9. disposable glass pipets and bulb
10. Chemicals for sample preservation.

V. Procedure

1. If the samples will not be going through the CLP Program for laboratory analysis, contact a subcontract commercial laboratory and inform them that you will be collecting equipment blanks and request for sample bottles, preservatives, labels, and chain-of-custody forms. Also ask the laboratory concerning volume of water they would prefer for each parameter and which parameters may be combined into the same sample bottle. If possible, have the laboratory supply the analyte-free water that will be used to collect the equipment blank. If the samples will be going to a CLP laboratory, obtain the bottles and all necessary supplies, including analyte-free water, from the Equipment Facility. The bottles need to be certified precleaned. All preservation and holding time requirements as per aqueous (water) samples apply. See table below for preservation and holding time of some of the more common parameters.

2. The equipment blank must be collected in a designated clean area of the site or field office. Spread out new plastic sheeting on a surface to be work off of. Have paper towels handy to wipe up accidental spills. It is preferable to collect the equipment blank prior to collecting the environmental samples; in which case, the rinsed equipment can subsequently be dried and used to collect environmental samples afterward.

3. Label the capped sample bottles with an identification number that will later be used for cross-referencing with associated environmental samples. All other label information will also need to be filled out prior to shipment to the laboratory. Place clear tape over the label to prevent the information on the label from being smeared if it should get wet. Record all information into the field logbook consistent with regular sample collection procedures.

4. Put on gloves and goggles. If equipment blanks are to be collected on the same day from more than one decontamination event (different days), different sets of blanks will need to be collected. Change gloves when handling the different sets of decontaminated equipment.

5. If large stainless steel bowls are part of the decontaminated equipment, use it to initially collect the rinsate water. And if the bowls are wrapped in aluminum foil, unwrap the bowls first. Minimize the handling of the decontaminated equipment to prevent the introduction of new contamination. Unwrap all other equipment (trowels, spoons, auger heads, etc.) and place them in the bowl. Larger equipment that does not fit in the bowl should be handled last. If large bowls are not part of the equipment, consider using other types decontaminated equipment that can hold water. Otherwise, the rinsate water will need to be captured directly into the sample bottles.

   Other types of equipment that will require equipment blanks may be water tubings, filters, and pumps. In this situation, connect and power up the equipment and pump water from the analyte-free water container, or from the pump submerged in a container containing analyte-free water, directly into the sample bottle(s).

6. Pour analyte-free water over the surfaces of the equipment, slowly and liberally, that is expected to contacts samples. Spoons and trowels may be placed in the stainless steel bowl and analyte-free water poured into. Analyte-free water may be poured into coring sleeves with the water captured by the stainless steel bowl or directly into sample bottles.

7. Sample bottles are to be filled in a specific order. VOA bottles (typically 40 millimeter size with approximately four drops of trace-analysis or reagent grade HCl) are to be filled first and completely. Organics are to be filled next followed by inorganics. If multiple rinses are required to collect enough volume of rinsate water to fill all the sample bottles, proportion the water into each bottle stepwise until all bottles (except for VOA) are filled to the proper level with the final equipment rinse.

8. Add preservatives as appropriate, using disposable glass pipets. For safety purposes after use, rinse pipet containing residual preservatives prior to disposal. See the table below or refer to the determinative method for preservation guidance. Cap the bottle(s) and invert several times to mix. Then check the pH of the equipment blank sample by pouring a few drops onto the pipet and from that onto the pH paper. Never dip the pH paper into the sample bottle as this may introduce new contaminant into the equipment blank sample. Add additional preservative if needed.

9. Place sample bottles into zip-lock bags and then immediately into cooler(s) containing large amount of bagged ice. All volatile blanks (and volatile environmental samples, if also being couriered) must be placed into the same cooler. Cushion bottles to prevent breakage, but not with bags of ice. Ice will lose it's cushioning abilities when melted.

10. Afterwards, if purchased 4-L analyte-free water was used and there is substantial remaining in an opened bottle, recap the bottle tightly and place a signed and dated

custody seal over the cap and neck of the bottle. The bottle of water may be used for the next equipment blank collection, and can be stored until the vendor's expiration date on the bottle or one year from opening, whichever is sooner. When ready, pack and ship equipment blanks consistent with shipping procedures.

Example Aqueous Sample Handling Requirements

Parameter	Containers	Preservation		Maximum Holding Time From Collection to Analysis[1,2]
		Temperature	Other	
TCL VOCs	3 x 40-mL Glass with Teflon septum caps	Cool 4°C ±2°C	HCl to pH<2 No bubbles	14 days
Metals	1 x 1-L HDPE or Glass	Cool 4°C ±2°C	HNO3 to pH<2	28 days (Hg) 6 months (Other metals)
Sulfide by EPA 376.2	500 mL Glass	Cool 4°C ±2°C	2mL Zinc Acetate NaOH to pH>9	7 days
Anions Chloride, Sulfate and Nitrate by EPA 300	500 mL Glass or Plastic	Cool 4°C ±2°C		28 days
Methane, Ethane, Ethene RSK-175	5 x 40-mL Glass with Teflon septum caps	Cool 4°C ±2°C	HCl to pH<2 No bubbles	14 days
TOC by SW 846 9060	250 mL Glass	Cool 4°C ±2°C	H2SO4 to pH<2 Store in dark	28 days

1. Refer to QAPP Worksheet #19, the CLP SOW requirements or the applicable EPA method.
2. The commercial laboratory should be contacted regarding what sample volumes, preservatives, and type of sample bottles they recommend. Refer to the determinative method or commercial laboratory for parameters not specified in the table above. Contact EPA RSCC for samples going through the CLP Program.

Attachment 8
Cornell Dubilier Electronics Superfund Site
Non-Conformance/Quality Control Report

CORNELL DUBILIER ELECTRONICS SUPERFUND SITE
NON-CONFORMANCE/QUALITY CONTROL REPORT

Date: _____

Organization Name: _____

Initiator's Name & Title: _____

Problem Description:

_____
_____
_____
_____
_____
_____
_____

Reported To: _____

Corrective Action:

_____
_____
_____
_____
_____
_____
_____

Reviewed and Implemented by: _____

Attachment 9.1

USEPA, SOP NO. HW-32 SOP for Implementing the National Strategy For Procuring Analytical Services

SOP NO. HW-32

STANDARD OPERATING PROCEDURE

FOR

IMPLEMENTING THE NATIONAL STRATEGY FOR PROCURING ANALYTICAL SERVICES FOR ALL OSWER PROGRAMS (SUPERFUND, RCRA AND BROWNFIELDS)

Revision 5, March 17, 2005

By: _____ Date: 3/21/05
Jennifer E. Feranda, CLP Project Officer
Hazardous Waste Support Section

Concurred By: _____ Date: 3/21/05
Linda Mauel, Chief
Hazardous Waste Support Section

Approved By: _____ Date: 3/21/05
Robert Runyon, Chief
Hazardous Waste Support Branch

Overview

Superfund's Field and Analytical Services Teaming Advisory Committee (FASTAC) analytical services strategy requires Agency personnel to utilize a tiered decision tree for procuring Superfund analytical services for all non-time critical data collection projects. Region 2 has and continues to utilize that sequential decision tree for procuring Superfund analytical services. The decision tree is as follows:

Tier 1: EPA Region 2 DESA laboratory (including ESAT support)
Tier 2: National Analytical Services Contract Laboratories (CLP RAS and Non-RAS)
Tier 3: Region Specific Analytical Services (SAS) Contract Laboratories
Tier 4: Contractor, IAGs and Field Contractor Subcontract laboratories

Region 2 has taken many steps to assure that the FASTAC process is being followed and that Regional and National laboratory resources are being utilized to the fullest extent by the Superfund program. Some of these steps include:

1. Centralization of all Superfund analytical services procurement activities through the Regional Sample Control Coordinator (RSCC).

2. Coordination between the RSCC and the Regional DESA laboratory.

3. Outreach/training on the FASTAC process and procuring analytical services to the Superfund Program Office on a regular basis.

4. Participation in annual (or more frequent as needed) meetings with Superfund field contractors and their EPA Project Officers to outline the FASTAC process and associated requirements.

5. Operation of a Regional non-RAS tracking database, in addition to the National Analytical Services Tracking System (ANSETS) database, to track what non-RAS analytical services are being provided by the Regional DESA laboratory and what is being subcontracted out. The Regional database and ANSETS are compared on a monthly basis to assure that the FASTAC sequential decision tree for procuring analytical services is being followed.

The following is the step by step process by which the RSCC receives and processes RAS and non-RAS analytical requests.

Definitions:

Routine Analytical Services (RAS): Standard Target Compound List (TCL) (VOA/Semi-VOA/Pesticide/PCB Aroclors) and Target Analyte List (TAL) (metals and cyanide)

Non-Routine Analytical Services (Non-RAS): All analytical services not considered as RAS as follows:

1. Non-RAS analytical services able to be performed using the CLP RAS contracts flex clause
2. Non-RAS National CLP methods (Dioxin, PCB Congener, Air, etc.)
3. All other Non-RAS

Analytical Service Requestor: The EPA site Project Manager (OSC, RPM, etc.) or their designated representative (i.e., field contractor, State, Army Corp of Engineers, etc.).

Process:

RSCC Contact Information

Jennifer Feranda: Primary RSCC and CLP Project Officer, e-mail: feranda.jennifer@epa.gov ; phone# (732) 321-6687
Adly Michael: RSCC, Database Coordinator, e-mail: Michael.adly@epa.gov; phone# (732) 906-6161
Robert Toth: RSCC, e-mail: toth.robert@epa.gov; phone# (732) 906-6171

The procurement of analytical services often starts with EPA PMs and/or their representatives contacting the RSCC to determine appropriate steps for procuring their analytical services and what documentation is required to submit these requests. The following steps delineate the process beginning with the RSCC's receipt of the formal request for analytical services:

1. **All analytical requests should be submitted to the RSCC at the earliest possible date.** Requests for all Superfund analytical services, both RAS and Non-RAS, are submitted to the RSCC up to one week prior to the sampling event. It is strongly recommended that all requests for non-Routine services be submitted at least four (4) weeks prior to the actual sampling event.* All requests should be submitted to RSCC by noon Tuesday. Any requests submitted after noon on Tuesday may not be considered until the following week.

2. DESA supplies analytical service requestors with electronic versions of the following analytical request forms: "U.S. EPA Region 2 CLP Analytical Services Request Form" (Attachment 1) and "U.S. EPA Region 2 Laboratory Analysis Request Form" (Attachment 2). All requests and subsequent correspondence relating to the request for booking are required to be transmitted electronically via e-mail. All requests should be

E-mailed to the RSCC, Jennifer Feranda with a cc: to Adly Michael and Robert Toth per the contact information provided above. This assures timely consideration of the analytical services request and serves as a Record of Communication (ROC) and the basis for hard copy documentation, as well as traceability for all requests being made.

3. Upon RSCC receipt of a request, a case folder is generated for that project. Hard copies of requests, ROC(s), additional e-mail and documentation relating to that case are placed in the file as they are received.

4. Upon receipt, analytical services requests are reviewed for completeness, accurate content, and confirmation of an approved (or approval pending) QAPP for the project. **No analytical services will be scheduled without an approved QAPP.**

5. Once requests have been reviewed for accuracy, completeness and QAPP status, they are submitted via e-mail to the DESA Lab. The DESA Lab holds booking meetings on Tuesday afternoons or Wednesday mornings to determine what projects (or portions thereof) they can accommodate.

6. No later then noon of Wednesday following the submittal of the request, the DESA Lab responds to the RSCC as to what analytical services they will provide. **All communications are done via e-mail in order to provide timely communication and a basis for a documented record.**

7. When the RSCC receives the information from the DESA Lab as to what services they will provide, several things occur:

   a. For RAS requests that will not be conducted by the DESA laboratory, the information is entered into the **S**uperfund **P**roject **R**equest **S**ystem (SUPRS) to be processed through the CLP. SUPRS is a national web based database that provides regional information to the Sample Management Office (SMO) to enable them to procure the appropriate CLP laboratory for the services requested.

   b. For non-RAS samples the CLP Organic and Inorganic Program Managers (PM) (located in HQ-Analytical Services Branch [ASB]) are consulted to see if special analyses can be performed through the CLP RAS contract(s) using the contracts "Flexibility Clause". If they can be analyzed through the CLP, RAS contract flex clause modifications are written by the PMs and requests are entered into SUPRS.

   c. Requests for Non-RAS national contracts (Dioxin, PCB Congener and Air analysis) are provided to the DESA Non-RAS program Task Order Project Officer (TOPO) for processing. The TOPO will write a specific Task Order Request for each individual project and submit it to ASB and the HQ Office of

Acquisition Management (OAM) Contracting Officer (CO) for review and to be offered for bid to contract labs under the National Non-RAS Blanket Purchase Agreements (BPAs). Once the lab(s) come back with a bid price, a lab is selected by the OAM CO and a procurement is prepared by the Region to transfer funds in to the national Non-RAS contracts. Laboratory information and special instructions are forwarded, via-email, to the EPA PM and their sampling representative at the time the procurement is to be prepared. Analytical services can be initiated upon OAM's receipt of the procurement.

d. For non-RAS requests that can be accommodated by DESA and/or the CLP flex clause, the analytical service requestor will be notified of such and will be provided contact and delivery, as well as any special, instructions.

e. For non-RAS requests that can not be accommodated through the DESA Lab, the CLP flex clause or the national Non-RAS program, the analytical services requestor is notified via e-mail that alternative means for analytical services will have to be obtained i.e., subcontract.

8. For work to be performed by the CLP for RAS and flexibility clause Non-RAS, SMO provides the RSCC, via e-mail, a Case number and laboratory assignments.

9. Once laboratory and case information are received from SMO (for CLP RAS and flex clause non-RAS), RSCC transmits this information, via e-mail to analytical services requestor. This information is transmitted via e-mail.

10. Two databases are maintained by the RSCC:

a. **Database #1:** This database tracks all RAS, flexibility clause non-RAS, and national contract non-RAS which are analyzed through the DESA Laboratory and the CLP (this database tracks all information from the time the samples are booked through sampling, analysis, data validation, and archiving). RAS analyses performed by the DESA lab are entered into this database, but only to the point of sample scheduling by the DESA lab. All work done after the scheduling is tracked by the DESA lab. (Attachment 3)

b. **Database #2:** This database tracks all other non-RAS analyses whether handled by the DESA laboratory or subcontracted out. It tracks non-RAS Superfund analytical work not tracked in Database #1(flex clause non-RAS and Non-RAS done through national contracts). The information in this database includes specifics about the project, the required analysis, number of samples, how those samples are analyzed (DESA vs. sub-contract lab), etc. (Attachment 4)

By COB Friday of each week, all new projects and relevant information are entered into each database as appropriate. The databases are updated with new information as needed.

11. At the end of each month, ANSETS (non-RAS tracking) information is provided to the RSCC by the analytical service requestors (Attachment 5). The information is entered into the Regional Database # 2 and the national ANSETS database. Monthly ANSETS reports are sent to the RSCC, by SMO, for their review. Concurrently, these reports are compared to the Regional Database #2 to try and determine whether the Superfund program and their representatives are following the FASTAC process as outlined by EPA HQ and Region 2.

12. If, after lab assignments are made, there is a change in the sampling event (i.e., change in date; cancellation; change in the number of samples being collected, etc.) the RSCC must be notified immediately upon the knowledge of any changes to the project, via e-mail. If there is a change in the sampling date or the number of samples being collected, a new laboratory request form (for the DESA laboratory or the CLP, as appropriate) must be submitted to the RSCC. (Reference Attachment 6).

* In FY' 03/04 DESA staff initiated a concerted effort to have RPMs involve DESA staff in scoping meetings for their projects. Early involvement in project planning enables the DESA lab to better accommodate the needs for the individual projects.

Names and Organizations Involved in the Procurement Process

<u>RSCC</u>: Jennifer Feranda and Adly Michael
EPA Region 2, Division of Environmental Science and Assessment (DESA)
Hazardous Waste Support Branch (HWSB), Hazardous Waste Support Section (HWSS)
DESA-HWSB-HWSS

- Regional Sample "Broker" all analytical services; oversight National CLP and National Non-RAS

<u>**DESA Lab Analytical Coordinator**</u>: John Birri,
EPA Region 2, DESA Laboratory Branch (LAB), DESA-LAB

- Coordination/Contact for all samples analyzed by the DESA Laboratory

Contractors and EPA Project Officers (PO):

Contractor: CDM Federal RACS
EPA PO: Fernando Rosado
CDM : Jeniffer Oxford

Contractor: Tetra Tech Environmental RACS
EPA PO: Keith Moncino
Tetra Tech: Lynn Arabia

Contractor: Weston RST
EPA PO: Helen Eng
Weston: Smita Sumbaly

Contractor: Weston SAT
EPA PO: Helen Eng
SAT: Yunru Yang

IAG: U.S. Army Corp of Engineers
EPA PO: Shaheer Alvi
USACE: Andrea Pouliet

ATTACHMENT 1

U.S. EPA REGION II

CLP ROUTINE ANALYTICAL SERVICES REQUEST FORM

Assigned CLP Case #:

Canceled:

Site Name:

City/State:

Site Spill ID:

CERCLIS ID:

Operable Unit:

Purpose Code:

Sampling Contact:

Phone#:

Organization:

EPA Project Manager:

QAPP Approved by EPA: Y/N

Date of QAPP Approval:

E-Mail Lab Assignments: Y/N

E-Mail for Lab Info:

E-Mail for Data:

Proposed Sampling Date(s):

Proposed Ship Date(s):

Saturday Delivery: Y/N

Oversight/Split Sampling (PRP/Fed. Facility): Y/N

Labs Used by PRP,FF:

Contaminant(s) of Concern (If known):

Number of Samples	Conc. Level L/M	Matrix (soil, aqueous, etc.)	Analysis	Turn Around Time 7, 14, or 21 days	SOW# and/or Method (i.e. OLM0x.x ,Modified 5035)	LabAssignment

Comments:

Sampling Project Manager: _____ **Signature/Date:** _____

RSCC: _____ **Signature/Date:** _____

ATTACHMENT 2

US EPA REGION 2 LABORATORY
non-RAS ANALYSIS REQUEST FORM

SURVEY NAME: _____ DATE OF INITIAL REQUEST _____ REVISED REQUEST DATE _____

REQUESTOR: _____ PHONE NUMBER _____ E-MAIL: _____

AFFILIATION: _____ Purpose Code: _____ Site Spill ID: _____ Operable Unit: _____

Address for Final Report: Check to E-mail Final Report: ☐

# SAMPLES	ANALYTE	MATRIX	# SAMPLES	ANALYTE	MATRIX	# SAMPLES	ANALYTE	MATRIX	# SAMPLES	ANALYTE	MATRIX
	SANITARY			*NITRITE			*E. COLI			VOA, 524.2 (DW levels)	
	ACIDITY			OIL & GREASE			*ENTERO-COCCUS, MF			HALOACETIC ACIDS	
	ALKALINITY, TOTAL			*ORTHOPHOS-PHATE			*ENTERO-COCCUS, MPN			ORGANOTINS	
	AMMONIA			PHENOLICS			*F-COLIFORM, MF			PCB CONGENERS (identify list)	
	ASPHALTENES			PHOSPHORUS			*F-COLIFORM, MPN			PCBs, TCL	
	*BOD, 5 DAY			SOLIDS, % 105degC			HETERO. PLATE COUNT (HPC)			PCBs, TSCA	
	*CBOD, 5 DAY			SPECIFIC CONDUCT.			*T-COLIFORM, COLILERT			PESTICIDES, TCL	
	CHLORIDE			SULFATE			*T-COLIFORM, MF			*PESTICIDES, WEST NILE	
	COD			SULFIDE			*T-COLIFORM, MPN			PESTICIDES, other (identify)	
	*COLOR			SULFIDE, UNIONIZED			***METALS***			***BIOLOGY***	
	CORROSIVITY			SULFUR			METAL:			#EFFLUENT TOXICITY - ACUTE	
	CYANIDE			TDS			ARSENIC			#EFFLUENT TOXICITY - CHRONIC	
	CYANIDE AMENABLE TO CHLORINATION			TKN			LEAD			#SED. TOXICITY - FRESH WATER	
	CYANIDE, WEAK ACID DISSOC (FREE)			TOC			METALS - SLUDGE			#SED. TOXICITY - MARINE WATER	
	DENSITY			TPH			HARDNESS			***GRAIN SIZE:***	
	*DO (Diss. Oxygen)			TSS			METALS FINISHING		1. Check Grain Size Method:		
	FLUORIDE			*TURBIDITY			METALS TAL (DW levels)		☐	Pipet Method (Plumb 1981) OR	
	*HEX. CHROMIUM			VISCOSITY			METALS, TAL		☐	Hydrometer Method (ASTM 422D-63)	
	IGNITABILITY			***MICRO***			METALS - TCLP		2. Check Sand/Sieve Reporting Units:		
	*MBAS (surfactants)			ASBESTOS			***ORGANICS***		☐	% Total Sand OR	
	*NITRATE			*CLOSTRIDIUM PERFRINGENS			NVOA, TCL		☐	% Sand Fractions (Very Coarse Sand, Coarse Sand, Medium Sand, Fine Sand, Very Fine Sand)	
	NITRATE + NITRITE			*CRYPTO/ GIARDIA			NVOA - TCLP		3. Check Fine or Mud Fraction Reporting Units:		
							PAHs		☐	% Total Fines (Silt + Clay combined) OR	
							TRIHALO-METHANES (THMs)		☐	% Total Silt and % Total Clay	
							VOA, TCL				
							VOA, TCLP				

☐ REQUEST ACCEPTED

☐ REQUEST NOT ACCEPTED

ABOVE STATUS APPROVED BY: JOHN BIRRI

DATE APPROVED: _____

NOTE: NO WEEKEND DELIVERIES ACCEPTED

SAMPLING DATES: from _____ to _____

SAMPLING TIMES FOR SHORT HOLDING TIME [asterisked]TESTS (check one):

☐ 0000 - 0600 ☐ 0600 - 1200 ☐ 1200 - 1800 ☐ 1800 - 2400 HRS

ARRIVAL DATES: from _____ to _____

ARRIVAL TIMES (check one): ☐ 8-11am ☐ 11am-4pm ☐ After 4pm

METHOD OF SHIPMENT: _____

SPECIAL REQUESTS: (e.g. - turnaround time, additional analytes, etc.)

REPORTING REQUIREMENTS (attach separate sheet, if more room is needed):

MATRIX KEY: A = AQUEOUS; D = DISSOLVED; S = SOLID; SL = SLUDGE; N = NON-AQUEOUS LIQUID/ OIL; O = OTHER (Describe)
- Specify organism, endpoints and test duration

VERSION DATE: 1/03/2005

ATTACHMENT 3

Database I / DATA TRACKING SUMMARY REPORT*

Site Name / Location		Case No./ Sampler	Request Rec'd Date	DESA Accept	CLP Accept	Requestor Notified Date	Case Booked Date	LAB	Analysis	Proj'd No. of Sample	TAT	Sampling Start Date	Sampling End Date	Data Due to HWSS	Act'l No. of Sample	Data Rec'd from Lab
Mohonk Road Industrial		33937														
High Falls	NY	USACE														
			2/22/2005	No, Odd Analyte(s)	Yes	3/4/2005	3/4/2005	LIBRTY	LC-VOA+1,4-di	5	14	3/10/2005	3/10/2005	3/25/2005		
Juana Diaz Wells		33938														
Juana Diaz	PR	PREQB														
			2/24/2005	No, Capacity	Yes	3/4/2005	3/10/2005	DATAC	LC-VOA	25	21	3/7/2005	3/11/2005	4/4/2005	17	
			2/24/2005	YES		3/2/2005	3/10/2005	DESA	LC-BNA	18	21	3/7/2005	3/11/2005		0	
			2/24/2005	YES		3/2/2005	3/10/2005	DESA	LC-Pest/PCBs	18	21	3/7/2005	3/11/2005		0	
			2/24/2005	YES		3/2/2005	3/10/2005	DESA	TAL-Metals+Hg	18	21	3/7/2005	3/11/2005		0	
			2/24/2005	YES		3/2/2005	3/10/2005	DESA	CN	18	21	3/7/2005	3/11/2005		0	
Lightman Drum Compan		33939														
Winslow Twp.	NJ	CDM														
			[illegible]/1/2005	No, Capacity	Yes	[illegible]3/4/2005	[illegible]2005	[illegible]A4	TC[illegible]VOA	[illegible]5	21	3/[illegible]2005	3/18/2005	4/4/2005		
			[illegible]/1/2005	YES		[illegible]/1/2005	3/1/[illegible]05	[illegible]ESA	TA[illegible]Metals+Hg	[illegible]3	21	3/[illegible]2005	3/18/2005		0	
Fried Industries		33940														
East Brunswick	NJ	TTFW														
			2/25/2005	YES		3/1/2005	3/1/2005	DESA	TAL-Metals+Hg	7	21	3/7/2005	3/11/2005		0	
			2/25/2005	No, Odd Analyte(s)	Yes	3/4/2005	3/4/2005	LIBRTY	LC-VOA+1,4-di	10	21	3/7/2005	3/11/2005	4/4/2005		

EXAMPLE

* Summary Report of RAS, modified RAS analyses, and national non-RAS contracts information.

ATTACHMENT 4

Database II / DATA TRACKING SUMMARY REPORT*

Site Name	Request Received Date	DESA Accept	Requestor Notified Date	Laboratory Name	Sampling Begin	Sampling End	Projected Samples	Collected Samples	Matrix	Analysis	TAT	Method
Consolidated Iron and Metals												
	6/1/2004	No, Capacity	6/3/2004	GPL Laboratories	6/7/2004	6/30/2004	5	3	Water	Volatiles	1	
	6/1/2004	No, Capacity	6/3/2004	GPL Laboratories	6/7/2004	6/30/2004	10	9	Water	TPH	21	SW-846 8015B
	6/1/2004	No, Capacity	6/3/2004	GPL Laboratories	6/7/2004	6/30/2004	3	2	Soil	TPH	21	SW-846 8015B
							18	14				
Lawrence Aviation Industries												
	4/2/2004	No, Capacity	4/6/2004	STL-Laboratories	4/12/2004	4/23/2004	12	11	Water	Titanium	21	SW-846 6010B
	4/2/2004	No, Capacity	4/6/2004	STL-Laboratories	4/12/2004	4/23/2004	140	136	Soil	pH	21	SW-846 9045C
	4/2/2004	No, Capacity	4/6/2004	STL-Laboratories	4/12/2004	4/23/2004	140	136	Soil	TOC	21	Lloyd Kahn
	4/2/2004	No, Capacity	4/6/2004	STL-Laboratories	4/12/2004	4/23/2004	140	136	Soil	Grain size	21	ASTM-D421-85
	4/2/2004	No, Capacity	4/6/2004	STL-Laboratories	4/12/2004	4/23/2004	130	122	Soil	Titanium	21	SW-846 6010B
							562	541				
							580	555				

EXAMPLE

* Summary Report of non-RAS analyses (other than non-RAS under National Contracts) either by EPA lab or subcontracted out.

ATTACHMENT 5

Analytical Services Tracking System (ANSETS) Reporting in Region 2

The following details EPA Region 2's procedures/process for reporting under ANSETS.

1. All sampling organizations (EPA, state, US Army Corp, and contractors) in Region 2 that procure analytical services under Tier 4 of the FASTAC process (sub-contract, IAG, etc.) must submit a form (Exhibit 1)detailing these analytical services (i.e., site information, matrix, analysis, number of samples, laboratory, etc) to the Regional Sample Control Coordinator (RSCC) on a monthly basis.

2. All of the data provided by the sampling organizations are entered into the Regional Database #2 and a Regional copy of the ANSETS database.

3. The information from the Regional ANSETS database is then exported (on a monthly basis) to the Sample Management Office (SMO) for incorporation into the National ANSETs Database.

4. Once SMO collects all of the information, they prepare a report of all the information collected from the Region and send the RSCC a report detailing information on each site in which information that was submitted for that month as well as a running total of the different types of analyses reported for the Fiscal Year. This report is sent to the CLP Project Officer (PO)/RSCC monthly.

5. The CLP PO/RSCC reviews the report, compares it with the regional database (Database #2) and disseminates the information as necessary to the DESA Lab, HWSS and HWSB management. The CLP PO/RSCC also may contact EPA Project managers or their designated representatives when questions arise as to why sub-contracts were used vs. the EPA DESA lab or the CLP.

Contacts for ANSETS Reporting

Jennifer Feranda: EPA Region 2 CLP PO/RSCC; DESA-HWSB-HWSS
- Overall coordination; review of reports and dissemination of information

Adly Michael: EPA Region 2 RSCC
- Regional ANSETs Database management and data entry
- Maintains files on all ANSETS documentation submitted to RSCC

Sampling Organization Contacts (Responsible for submitting ANSETS information)

Dianne Salkie: US EPA Region 2 DESA-HWSB-SCST
Jenniffer Oxford: CDM Federal Programs
Lynn Arabia: Tetra Tech Environmental Corp.
Smita Sumbaly: Weston Removal Support Team (RST)
Yunru Yang: Weston Site Assessment Team (SAT)

Frank Sorce: NJ Department of Environmental Protection (NJDEP)
Lisa Greco-Segazi: Malcolm Pirnie Inc. (MPI)
David Evans: U.S. Army Corp of Engineers, Kansas City District

United States Environmental Protection Agency

EXHIBIT 1

Attachment 2 - ANSETS Data Requirement
(List of Required Data)

Date:

Sampling Start Date:
Sampling End Date:

Project Numbers			
Project Number:	Regional Account Number:	DAS Number:	Assoc. CLP Case No:
Site Information			
Site Name:		City:	State:
CERCLIS ID:	Operable Unit:	Action:	Funding Lead:
Responsible EPA Project Individual:		Sampling Organization:	

Analytical Services Information	
If field analytical services are used during this project write "field analysis" in the Laboratory Name Column. If fixed laboratory is used write the name of the laboratory in the Laboratory Name Column. Please specify in this box all field analytical techniques used.	COST:

Laboratory Name (include location if multiple lab locations)	No. Samples	Matrix	Analysis	Requested Turnaround (Days)

Completed by:	Organization:	Date:

ATTACHMENT 6

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION II

DATE: October 13, 2004

SUBJECT: Procedures for Notification of Changes in Sampling/Analytical Schedules

FROM: Robert Runyon, Chief
Hazardous Waste Support Section (2DESA-HWSB)

TO: Vince Pitruzzello, Chief
Program Support Branch (2ERRD-PSB)

The purpose of this memo is to provide you with an update on requirements for procuring any Superfund analytical services through EPA Region 2's Division of Environmental Science and Assessment (DESA) laboratory and the National Contract Laboratory Program (CLP).

Over the past several months there has been a significant increase in the number of sampling events that have; 1) been postponed; 2) been cancelled; 3) been extended beyond the sampling period previously designated; or 4) changed the number of samples submitted to laboratories for analysis from the number scheduled (increased or decreased, often significantly). While it is understood that sampling and field work can sometimes be unpredictable with delays and/or changes in sample numbers, sampling events requiring changes are occurring more frequently among Region 2 field contractors. In addition, RPMs and/or samplers are not notifying the Regional Sample Control Coordinator (RSCC) and/or DESA lab of any such changes in a timely manner. Often, the RSCC or DESA laboratory are notified after the samples were expected to arrive at the lab(s), and/or DESA staff are required to initiate contact with sampling contractors to determine the status of scheduled samples that have not been received.

Delays in notification of changes in scheduled sampling projects result in costly workload inefficiencies. DESA sample coordinating staff spend unnecessary time tracking down the status of specific projects when samples don't arrive on schedule, and the scheduled analytical resources requested are unavailable for use on other projects while committed to scheduled projects.

The following procedures are being implemented to make most cost effective use of analytical resources, and to ensure that proper communication on sampling issues is being maintained between EPA Project Managers, their contractors, and DESA staff:

1) It is the Site Project Manager's (RPM, OSC, SAM, etc.) and their contractor's responsibility to notify the RSCC of any changes to sampling schedules or numbers of samples being submitted. Notifications should be made immediately upon the knowledge of any changes to the sampling project schedule or sampling numbers. Failure to appropriately notify the RSCC could result in samples not being analyzed or an extension in the time to complete analysis. This holds true for both the DESA laboratory and the CLP.

2) All notification of changes in sampling schedules or the number of samples being submitted for analysis (either to the DESA laboratory or the CLP) **must** go through the RSCC, currently Jennifer Feranda.

3) For any changes in sampling dates or the number of samples being submitted, a new Booking request form must be completed and submitted to the RSCC. The appropriate form must be

used dependent on which lab(s) are scheduled to receive the samples (i.e., the DESA laboratory or the CLP).

4) Once all scheduling issues have been resolved, communication on other sampling and technical issues can be directed to the appropriate contacts: John Birri (732) 906-6886 for the DESA laboratory and Adly Michael (732) 906-6161 for the CLP.

Should you have any questions or require further information, please contact me at (732) 321-6645 or Jennifer Feranda of my staff at (732) 321-6687.

Attachments

cc: Deb Szaro
Linda Mauel
Jennifer Feranda
John Bourbon
John Birri
Adly Michael

Attachment 9.2

USEPA Region 2
Analytical Request Form

US EPA Region 2
Analysis Request Form

CLP Case/Project #:		Date Received by RSCC:		Date Cancelled:	

Site Name:		CERCLIS ID:		Sampling Dates:	
City/Town:		Op Unit:	Site Spill ID:	Start:	
State:		Action Code:		Finish:	

Arrival Time:
- ☐ 0800-1200Hrs
- ☐ 1200-1600Hrs
- ☐ After 1600 Hrs

EPA Project Manager:		Analytical Services Requestor:		Proposed Shipping Dates:	
First Name:		First Name:		Start:	
Last Name:		Last Name:		Finish:	
		Phone #:			
		Organization:		Saturday Delivery?	☐ Yes

EPA Approved QAPP?:	☐ Yes				
Date of QAPP Approval:		Oversight/Split Sampling?: (e.g. PRP/Fed Facility)	☐ Yes	Labs Used: (PRP/FF)	

E-mail for Lab Assignments:	E-mail for Data:	Address for Hard Copy:

Contaminants of Concern (if known):

Known Hazardous Waste Constituents:

Special Requests & Reporting Requirements (attach if more space required):

** Shaded area for RSCC use only **

U.S. EPA Region 2
Analysis Request Form

Site Name:						
# of Samples	Conc. Level (see key)	Matrix (see key)	Analyses	Turnaround Time (for validated data)**	SOW # / Method	Lab Assignment

** See instruction sheet for explanation of Turnaround Time for validated data.

Accepted by:		Date Accepted:	

** Shaded area for RSCC use only **

Attachment 10

USEPA Region 2 Memorandum: Tracking Superfund Non-CLP Analytical Data, Directive #9240.0-2C, January 2003

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION II

DATE:

SUBJECT: Tracking Superfund Non-CLP Analytical Data (ANSETS): Directive # 9240.0-2C

FROM: Jennifer E. Feranda, CLP Project Officer and Regional Sample Control Coordinator
Hazardous Waste Support Section (2DESA-HWSB)

TO: See Addressees

The purpose of this memo is to inform you about OSWER Directive # 9240.0-2C (attached) concerning the requirements for nationally tracking non-Contract Laboratory Program (CLP) analytical services. This directive supersedes OSWER Directives 9240.0-2A and 2B which established the Analytical Services Tracing System (ANSETS). The primary focus of the new directive is on tracking analytical data generated via EPA field contractors and their subcontractors at federal fund lead sites and at sites where EPA is the lead agency overseeing federal facility cleanups under Interagency Agreements. Per the Directive, RS&T Division laboratories, State-funded sites, and Potentially Responsible Parties (PRPs) do not need to comply with ANSETS data submission. However, under the Region 2 non-CLP tracking system that was established in 2000, the Region requires PRPs to report this information. Site Project Managers should be writing this requirement into their action memos, orders, etc. Although this information is not currently required by HQ, the Region will continue to require PRPs to submit this data to the Regional Sample Control Coordinator (RSCC).

The EPA Field and Analytical Services Teaming Advisory Committee (FASTAC) established a decision tree for selecting analytical services. The Tiers (with Tier 1 being the most favorable option) are:

Tier 1: RS&T Division Laboratories (preferred option for special analytical services)
Tier 2: CLP (preferred option for routine analytical services)
Tier 3: Region specific analytical services contracts
Tier 4: Obtaining analytical services using subcontractors via field contracts.

Tier 4 is the least preferred option due to lack of direct oversight of these contractors, quality assurance potentially not meeting EPA standards, and often higher costs for services. By requiring contractors to use the ANSETS tracking system, the Superfund program can determine whether the FASTAC strategy is being implemented, analyze trends in new services needed, track national laboratory analyses acquired for the Superfund program, and plan for quality assurance oversight.

The directive outlines several options for submitting ANSETS data, however, it also allows each Region to use their discretion on how the ANSETS information will be provided to HQ. Region 2 already has a system in place for reporting Non-CLP analytical services information, and will continue to utilize this system. To provide consistency between the Region 2 and National requirements, the region will now utilize the "ANSETS Data Requirement" form (copy attached) in place of the "Non-CLP Tracking Form" which is currently being used. The forms should be completed and submitted to the RSCC, currently myself, by the first of each month for the previous months sampling. Once the information is compiled in the Region, it will be sent to HQ for inclusion in the national database.

The requirements for ANSETS reporting became effective as of January 1, 2003. Per the

directive, Regional Contracting Officers and Project Officers will need to amend their assessment and response contracts to reinforce the ANSETS analytical services tracking requirements. If you have any questions regarding the implementation of these requirements, both national and regional, please contact me at (732) 321-6687.

Attachments

Addressees:

Shaheer Alvi
Helen Eng
Keith Moncino
Richard Graciano
Fernando Rosado
Kathy Moyik
Lisa Guarnieri
Superfund Remedial Project Managers
Superfund Site Assesment Managers
Superfund On-Scene Coordinators
HWSB

cc:
Vince Pitruzzello
Deb Szaro
Kevin Kubik

Attachment 11

Daily Quality Control Report

DAILY QUALITY CONTROL REPORT

Site: _____
Project No.: _____
Date: _____
Field Team: _____

Weather (circle)

	Bright Sun	Clear	Overcast	Rain	T-Storm	Snow
Temp:	0 to 32	32 to 50	50 to 70	70 to 85	> 85	
Wind:	Still	Gusty	Moder.	High	Direction:	
Humidity:	Dry	Moder.	Humid			

Subcontractors and Equipment on Site: _____

Health and Safety Levels: (circle)

D	Mod. D	C	B	A

Summary of Health and Safety Activities: _____

Instrument Used: (circle)	PID	LEL	pH	Cond	Therm.	Turbidity	DO	ORP
Calibrated: (check)								

For actual calibration results, see field calibration forms.

Summary of Work Performed: _____

All samples were collected according to the procedures outlined in the planning documents?

Yes _____ No _____

Problems encountered/corrective actions taken: _____

Time Project Manager Contacted: _____

Problems encountered/corrective actions taken: _____

Name: _____ Signature: _____

Attachment 12

Stone Environmental Inc.
Procedures

Stone Environmental
Summary of Proceduces

1) Summary of Method for Analytical Method

The analyses are performed on a Hewlett Packard model 6890 Gas Chromatograph with computer control. Methanolic extract is injected directly into a polar-deactivated pre-column connected to the analytical column. For the cool on-column method, the sample is injected using a cool on-column injector, set to ramped temperature mode. In the split method, the methanolic extract is injected using split-splitless injector (ratio 1:100) into the analytical column. A column coated with a thick film non-polar stationery phase (5 μm, HP-1, equivalent) is used in the method. Methanol is incompatible with this phase, and consequently elutes in dead time. The less polar analytes, refocused through the retention gap effect, are retained much more strongly and elute later, even when their boiling points are lower than that of MeOH. The use of non-polar stationary phase reduces the possibility of methanol coeluting with one or more of the analytes, which could adversely affect their chromatography and detection.

Core samples are collected and the rock samples are immediately crushed using a device that was designed by the University of Waterloo. For further details on the sampling and preservation technique associated with rock samples, see the Stone's SOP titled "SEI-6.37.0 Field Methods for Retrieval, Collection, Handling, and Preservation of Rock Samples to be Analyzed for VOCs and Physical Properties".

The crushed samples then undergo microwave assisted extraction (MAE). For further details on the MAE procedure, see Stone's SOP titled "SEI-10.17.0 Microwave Assisted Extraction of Volatile Organic Compounds from Rock Samples". Typical solvent extractions using shaking or sonication require five weeks in order to extract all of the contaminant mass even following crushing; using MAE this extraction time is reduced to less than an hour.

2) Summary of Method for MAE Method

The microwave extraction is conducted in a microwave system model Ethos EX Labstation, built by Milestone Srl, Italy. The main component of this system is the MPR-600/12S medium pressure segmented rotor which contains 12 vessels for solvent extraction.

Core samples are collected and the rock samples are immediately crushed using a device that was designed by the University of Waterloo. The crushed samples then undergo microwave assisted extraction (MAE). Typical solvent extractions using shaking or sonication require five weeks in order to extract all of the contaminant mass; using MAE, this extraction time is reduced to less than an hour.

STANDARD OPERATING PROCEDURE

SEI-6.37.0

Field Methods for Retrieval, Collection, Handling, and Preservation of Rock Samples to be Analyzed for VOCs and Physical Properties

NOTE: This SOP contains proprietary information – Do Not Distribute

SOP Number: SEI-6.37.0 | Date Issued: 06/30/08
Revision Number: 0 | Date of Revision: NA

1.0 OBJECTIVE

Rock cores are retrieved by a drilling team from fractured bedrock aquifers and sampled at approximately one foot intervals to be analyzed for volatile organic compounds (VOCs). Rock core samples will be collected for determination of VOC concentrations in the rock matrix, along with representative samples for physical property analyses including porosity, bulk density, organic carbon content, chloride diffusion coefficient and matrix permeability.

2.0 POLICIES

1. According to 40 CFR Part 160, Subpart E, Section 160.81 and the NELA standards, a testing facility shall have standard operating procedures in writing setting forth study methods that management is satisfied are adequate to insure the quality and integrity of the data generated in the course of a study.
2. Personnel will legibly record data and observations in the field to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

3.0 SAFETY ISSUES

1. If necessary and appropriate, a site-specific health and safety plan shall be created for each study site. A template for creating a proper health and safety plan is provided on the SEI network.
2. Care must always be taken when approaching a sampling location. Do not, under any circumstances, place yourself in danger to collect a sample.
3. If necessary and appropriate, all chemicals are required to be received with Material Safety Data Sheets (MSDS) or appropriate application label. These labels or MSDS shall be made available to all personnel involved in the sampling and testing.

4.0 PROCEDURES

4.1 Rock Core Sampling Equipment and Materials

1. Sharpies and Ball point pens
2. Several small coolers
3. Small Dry-Erase White Board (2)
4. Tough-book Field Computer (2)
5. USB Data Stick
6. Printer
7. Wash Tubs (4)
8. Folding Tables (4+)
9. Chairs (3)
10. Scissors
11. Scrub Brushes (several)
12. Squirt Bottles (4+)
13. Required field personnel protective equipment (PPE).
14. Paper towels
15. Teflon tape (heavy duty)
16. Duct tape
17. Rubber bands
18. Aluminum foil
19. Saran wrap
20. Parafilm
21. Phosphate free detergent
22. Distilled Water
23. Bubble wrap
24. Ziploc bags (large and small)
25. Ice Packs
26. Kim-wipes
27. Crack Hammer (2 or more)
28. Steel Chisel, 3" blade (3 or 4)
29. Pick – For removing o-rings
30. MeOH, Purge and Trap grade and Wash grade
31. Enerpac™ Hydraulic Press
32. Sample Trimming Cell
33. Complete Sample Crushing Cell
34. Stainless Rods (for knocking out pucks)
35. O-rings for Pucks
36. Spare Bolts for Base
37. Wood Blocks 6' Plank marked in 0.1 foot intervals.

4.2 Core Retrieval and Sample Collection

Core runs will be HQ-sized (2.5-inch diameter) in five-foot lengths, collected using a triple-tube core barrel system. Immediately upon arrival of the cores at ground surface, they will be removed from the core barrel and placed in aluminum foil-lined PVC trays (6 ft long, 4-inch diameter PVC pipe split lengthwise) and covered with a continuous sheet of clean aluminum foil to minimize volatilization of VOCs. Aluminum foil will be used to keep most of the core covered while the geologist and hydrogeologist inspect the core and select sample locations based on

presence of fractures, lithology, weathering and evidence of groundwater and/or DNAPL fluid flow. The hydrogeologist will flag the sections of the core to be subsampled for VOC analyses and physical property measurements (moisture content, porosity and bulk density, diffusion coefficient, matrix permeability, organic carbon content). The drilling pace should be controlled by the on-site hydrogeologist to ensure that core is not drilled or retrieved before the hydrogeologist and crusher are prepared to receive it (i.e. previous core run has been completely processed or nearly so).

4.2.1 VOC Sample Selection

Subsamples (i.e. core segments ~1 to 2-inches long) for determining CVOC concentrations willbe taken from the cores immediately to minimize chemical losses due to volatilization. The initial core logging will be performed to identify key features for subsampling purposes and samples will be quickly broken from the core using a hammer and chisel, and then wrapped in aluminum foil for VOC and moisture preservation and given a unique field ID. After these samples have been collected, the core will be logged in more detail by the geologist. Additional sample types will then be selected and removed from the core. For future reference, wood blocks will be placed in the core boxes where samples have been removed, indicating the sample ID, length and depth of the removed section and the type of sample.

Samples for CVOC analysis will be collected using three criteria. First, samples will be taken

immediately adjacent to (including fracture surface) and 6 to 12 inches away from identified fractures, both above and below these features. All breaks in core should be suspected as being

fractures insitu, however, emphasis will be made on features with additional lines of evidence for active fluid flow (i.e. secondary mineral coatings/staining, slickensides, report from drillers

regarding fluid loss/gain at specific depths during coring). These samples are intended for measuring the extent of diffusion into the matrix blocks away from fractures that may have once contained DNAPL phase or are pathways for solute transport. Second, additional subsamples will be collected at distinct changes in lithology /mineralogy. Samples will be collected from both sides of such boundaries, referred to as lithology pairs so that representative samples are collected from the different matrix materials. Third, field duplicate samples will be collected at a minimum frequency of 1/20, taken from the same length of core split lengthwise (along the core axis) to provide samples from the same depth

interval and lithology. The hydrogeologist will exercise judgment during sampling to provide an average sample frequency of one sample every 1 to 2 ft, which will likely provide more samples than specified in the Work Plan, to allow flexibility regarding which samples to analyze. Photographs of the core will be taken that identifies the top/bottom and depth interval of the core run, core location and date. The geologist then continues logging the details and the hydrogeologist selects the physical property samples.

4.2.2 Physical Properties Sample Selection

These samples will be collected approximately every 20 ft in the same manner as the CVOC samples described in Section 2.2.1. An effort will be made to obtain samples from different lithologies or where variations in lithology occur as assessed during core logging. Each sample will be a cylindrical disc of the same diameter as the HQ core retrieved from the core barrel, with a height of 2 to 6 inches. These sections of core will be obtained such that the insitu moisture conditions are retained: working quickly, keeping the core covered and out of direct sunlight, and immediately wrapping and sealing the samples. After the sample is broken from the core, it will be immediately wrapped tightly in clean aluminum foil twice around the circumference for complete coverage of the sample, followed by plastic wrap and tape. Finally, the sample will be completely wrapped in parafilm, labeled and placed in a sealable plastic bag. Each sample will be clearly labeled with the sample ID, core location, depth, and date. Details and a full lithology description of each sample will be recorded in the field notebook.

4.3 VOC Sample Preservation

VOC samples will be collected in 40 mL clear glass VOA vials with Teflon-lined septa and screw caps. The rock core sampling requires both purge and trap grade methanol for rock sample extraction/preservation, and wash grade methanol for decontamination, which should be ordered beforehand and available prior to start of drilling. Each labeled sample container and lid will be weighed empty, and re-weighed once the purge and trap grade methanol has been added to accurately determine the weight and volume of methanol extract. Sample vials are prepared with methanol on the day of sample collection where possible; otherwise the weight of the vial with methanol should be re-checked prior to use. At the end of each drilling day, each of the days vials used for rock samples will be weighed so the exact weight of each rock sample is known and then prepared for shipping. There should be a minimum of 15 g of rock in each sample jar. Sample bottles will be stored in a cooler with ice or fridge set to 4°C until they are shipped back to UW.

Following is a step-by-step outline of the process of rock core sampling for VOCs:

a. Lay core in an aluminum foil-lined split PVC tray. Note top/bottom of core and depth interval, measure length and quickly identify features (fractures, breaks, lithology/mineralogy changes, evidence of fluid flow (i.e. secondary minerals, precipitates, slickensides, etc.) to select sample locations. Cover with aluminum foil to minimize CVOC volatilization and evaporation of pore water.

b. Collect CVOC samples: break off a one to two-inch section of core using a rock hammer and chisel, inserting a wood spacer that specifies the sample ID, depth interval that was removed and the type of sample taken. If a field duplicate is planned, the disc length should be doubled and split lengthwise along the core axis to produce two samples from the same depth interval.

c. Completely wrap each sample in a piece of clean aluminum foil to minimize volatilization loss and place in a zip-loc bag with a unique field ID, and place in a cooler with ice for storage prior to crushing. Record sample information including a lithology description, sample type (e.g. lithology pair, duplicate), depth, position relative to nearby fractures and other pertinent details.

d. Following collection of samples for each core run, transport samples to the crushing area.

e. Crush CVOC samples: collect subsample from each CVOC sample using a hammer and chisel and steel chipping tray, with effort made to remove the outer rind of the sample exposed to drilling fluids. Place subsample in a clean, dry crushing cell and crush with the Enerpac™ system.

f. Empty crushed rock sample (minimum of 15 g) into 40 mL VOA sample vial containing a known volume (20 mL) of purge and trap grade methanol, taking care to avoid splashing of methanol out of the vial. Sample vials and screw caps with septum have been previously labeled and weighed both before and after filling with methanol so the mass/volume of methanol is accurately known. Bottle threads should be wiped clean with a kim-wipe prior to screwing on the cap to remove any rock fragments that prevent the cap from sealing properly.

g. Decontaminate the rock crusher components using the procedure outlined in Section 4.4. Multiple crushing cells will be used (at least four or five sets) to allow processing of all samples from most core runs before decontamination is required.

h. Equipment blanks and field duplicates will each be collected after every 20 samples.

i. After drilling is completed each day, weigh vials with the rock samples so the mass of rock is known. Wrap screw cap lid-bottle seam with Teflon tape, wrap vials with bubble wrap and place individually in zip-lock bags for shipping. Place in cooler with trip blanks, which remain with specific batches of samples until arrival at UW, and keep on ice.

j. Chain of custody (COC) forms will be filled out at the end of each day of sample collection and shipped with each cooler back to Stone Environmental.

4.4 Decontamination of Drilling and Subsampling Equipment

Decontamination procedures are designed to remove all traces of contaminants from the equipment to prevent cross-contamination. Core barrels will be cleaned between runs using clean water and pressure washer. The chisel will be sprayed off with wash grade methanol followed by distilled water, and dried with a clean cloth before re-use. Only those parts of the rock crusher that came into contact with the rock subsamples require cleaning, including the crushing cell with top/bottom plates and funnel used to direct the crushed rock sample into the VOA vial. Chipping trays will also be decontaminated between samples. At least four or five sets of crushing cells will be used, which in most cases should allow processing of all of the samples collected from a 5-ft core run. The procedure for cleaning these components consists of four steps. First, the parts will be cleaned in a phosphate-free detergent (e.g. Alconox) wash to get rid of obvious sediment. The second step is full immersion in a clean water rinse, followed by a methanol (wash grade) rinse using a squirt bottle to remove any traces of contaminants not removed previously. The final step is a rinse with distilled (organic free) water using a squirt bottle to remove all traces of methanol. Parts are then dried using clean paper towels. The soap and water baths will be changed on a regular basis, and equipment blanks will be collected after every twenty samples by wiping inside a clean crushing cell with a kim-wipe soaked in purge and trap grade methanol which is then placed in a 40 mL VOA vial to be submitted for analysis along with the rock samples. Decontamination fluids and remnant rock fragments will be barreled.

4.5 Field QA/QC

Equipment blanks, collected as described above, will be taken after every 20 samples. Trip blanks, consisting of purge and trap grade methanol in 40 mL vials will be placed with each batch of samples stored/shipped in coolers. These are filled with methanol at the same time as the sample vials, and handled with the prepared vials and methanol preserved VOC samples, and remain unopened until returned to Stone Environmental for analysis. Field duplicate samples will be taken every 20 samples on core sections of double length split along core axis, so that the samples are taken from the same depth interval and lithology. Methanol blanks, used to identify pre-existing contamination of the purge and trap grade methanol, are taken at the start and end of each bottle (typically 1 L) while filling sample vials and stored/shipped separately from the samples. All QA/QC samples will be identified on the chain of custody forms and tracked throughout the sample handling and analysis process.

5.0 RESPONSIBILITIES

1. All personnel will legibly record data and observations (including phone conversations) in accordance with this SOP to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

6.0 DEFINITIONS

1. *EPA* means the U.S. Environmental Protection Agency.

2. *Observations & Remarks Form (O&R):* A pre-printed form, which contains mostly blank space for general note taking. The form typically prompts the user for the study or project designation, the SEI project number, the client or sponsor name, the total number of pages (page *n* of n) and requires a signature and date. The form is generally used to capture notes of one person when another, more specific forms is not available.

3. *Raw data* means any laboratory worksheets, records, memoranda, notes, or exact copies thereof, that are the result of original observations and activities of a study and are necessary for the reconstruction and evaluation of the report of that study. In the event that exact transcripts of raw data have been prepared (e.g., tapes which have been transcribed verbatim, dated, and verified accurate by signature), the exact copy or exact transcript may be substituted for the original source as raw data. Raw data may include photographs, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

7.0 REFERENCES

40 CFR Part 160 Good Laboratory Practice Standards, August, 1989.

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

None

9.0 AUTHORIZATION

Written by:_____ Date:_____

Lee Rosberg, Staff Scientist

Approved by:_____ Date:_____

Chris Stone, President

STANDARD OPERATING PROCEDURE

SEI-10.17.0

MICROWAVE ASSISTED EXTRACTION OF VOLATILE ORGANIC COMPOUNDS FROM ROCK SAMPLES

NOTE: This SOP contains proprietary information – Do Not Distribute

SOP Number: SEI-10.17.0
Revision Number: 0

Date Issued: 07/02/08
Date of Revision: na

1.0 OBJECTIVE

The Microwave Assisted Extraction (MAE) method was designed at the University of Waterloo and is used to extract Volatile Organic Compounds (VOC) from rock samples.

2.0 SUMMARY OF METHOD

The microwave extraction is conducted in a microwave system model Ethos EX Labstation, built by Milestone Srl, Italy. The main component of this system is the MPR-600/12S medium pressure segmented rotor which contains 12 vessels for solvent extraction.

Core samples are collected and the rock samples are immediately crushed using a device that was designed by the University of Waterloo. The crushed samples then undergo microwave assisted extraction (MAE). Typical solvent extractions using shaking or sonication require five weeks in order to extract all of the contaminant mass; using MAE, this extraction time is reduced to less than an hour.

3.0 DEFINITIONS

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of a similar matrix, meeting the above-mentioned criteria.

Corrective Action: action taken to eliminate the causes of an existing non-conformance, defect or other undesirable situation in order to prevent recurrence.

Instrument Blank: a blank matrix that is the same as the processed sample matrix (i.e. extract,

digestate, condensate) and introduced onto the instrument for analysis.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

Quality Control (QC): the overall system of technical activities, the purpose of which is to measure and control the quality of a product or service.

Spike: a known amount of an analyte added to a blank, sample or sub-sample.

Standard Operating Procedure (SOP): a written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks.

VOC – Volatile Organic Compounds

QA/QC – Quality Assurance/Quality Control

4.0 INTERFERENCES

When analyzing for VOCs, samples can be contaminated by diffusion of volatile organics (particularly chlorofluorocarbons, chloroform and methylene chloride), through the sample container septum during shipment and storage. A trip blank prepared from organic-free reagent methanol and carried through sampling and subsequent storage and handling must serve as a check on such interferences.

Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed in sequence. Whenever an unusually concentrated sample is encountered, it is recommended to prepare methanol blanks in the vessels used for the extraction of highly concentrated samples. Extraction vessels and syringes should be adequately cleaned and flushed prior to use. All glassware must be kept scrupulously cleaned. Clean all glassware as soon as possible after use by rinsing with the last solvent used or analyte-free water. Clean dry glassware should be stored in a clean environment.

5.0 SAFETY ISSUES

Laboratory personnel should take extra care when working with standards. When working with neat standards, make certain that there is adequate ventilation and that Nitrile or Latex gloves are worn and if necessary safety glasses.

If necessary and appropriate, a site-specific health and safety plan shall be created for each study site and followed. All samples as well as standards should be treated as a potential health hazard. Exposure

to each should be reduced to the lowest level possible using gloves and a hood. Material Safety Data Sheets (MSDS) should be available to all personnel.

Field visits may involve accessing remote areas. Health and safety concerns regarding these field visits are minimal, however, hazards such as slip, trip and falls, poisonous plant and dangerous animals, as well as getting vehicles stuck in remote areas, do present considerable health and safety issues. To help ensure field staff's health and safety in remote areas, all field staff are to have daily communication with the project manager or another appropriate SEI employee.
All chemicals are required to be received with a Material Safety Data Sheets (MSDS). MSDSs shall be made available to all personnel involved in the sampling and testing.

6.0 EQUIPMENT AND SUPPLIES

- The microwave system is an Ethos EX Labstation, built by Milestone Srl. (Italy), equipped with the MPR-600/12S medium pressure segmented rotor containing 12 vessels for solvent extraction. The vessels are made of TFM Teflon, chemically inert to most organic solvents and combinations thereof, with very high stability to temperature extremes. It is microwave transparent; maximum working temperature for extended use is 260°C, and 300°C for brief use. Each vessel is protected by a safety shield, for which the maximum temperature for extended use is 250°C. The microwave system is controlled by a dedicated computer.
- Analytical balance
- Glass disposable pipettes
- 4 mL glass vials
- 1.5 mL GC vials
- Crimper
- Purge and trap grade methanol
- Wash grade methanol
- Analyte free water
- Spatulas
- Cleaning brushes
- Centrifuge

7.0 REAGENTS AND STANDARDS

Organic-free reagent water demonstrated to be free of compounds of interest (spring water, carbon filtered and/or deionized).

Purge and Trap Grade or equivalent grade methanol (demonstrated to be free of analytes and stored away from other solvents).

8.0 SAMPLE COLLECTION AND HANDLING

8.1 Sample Collection

Rock core samples are collected by trained geologist. Core samples are collected and the rock samples are immediately crushed using a new device that was designed by Parker's research team at the University of Waterloo. The core sample is placed in a closed stainless steel crushing cell fitted with "O"-rings and crushed with a stainless steel piston at pressures as high as 3000 psi. The crushed sample is then extruded from the cylinder directly into a pre-weighed 40 mL VOA vial containing a known volume of purge and trap grade methanol. The amount of air passing by the sample while being crushed is minimized as well as the time required to completely crush the rock fragments into the desired particle size. The stainless steel crushing vessels and associated tools are thoroughly decontaminated following each use. The samples are weighed upon receipt at the laboratory and recorded in the Log in summary sheet (Table 1.) For further details on the sampling and preservation technique of rock samples, see the following SOP "SEI-6.37.0 *Field Methods for Retrieval, Collection, Handling, and Preservation of Rock Samples to be Analyzed for VOCs and Physical Properties*".

The crushed samples then undergo microwave assisted extraction (MAE). Typical solvent extractions using shaking or sonication require five weeks in order to extract all of the contaminant mass even following crushing.

8.2 Handling and Holding Times

Rock samples collected in methanol are stored chilled at 4° C (<6 °C) from collection. Samples are allowed to come to room temperature just prior to analysis.

If necessary, a separate rock sample will be collected in a 40 mL vial or equivalent for percent moisture content so that results can be adjusted for %moisture and reported as dry weight rather than as received. Percent moisture is determined as follows: Percent Moisture Determination - Immediately after weighing the sample for analysis, weigh 5-10 g of the rock into a tared crucible. Determine the percent moisture by drying overnight at 105 °C. If available, allow to cool in a dessicator before weighing. Concentrations of individual analytes will be reported relative to the dry weight of rock.

Percent moisture - %Moisture = gms of wet sample – gms of dry sample/gms of wet

9.0 PROCEDURE

Step 1: Sample Receipt

1. Unwrap samples carefully
2. Check all samples against the chain of custody forms
3. Sign all chains of custody and file them
4. Fill out the Stone Environmental sample login sheet (Table 1)
5. Assign unique SEI lab number to each sample received
6. Unwrap the teflon tape from each vial and weigh each sample in its vial
7. Record weight on login sheet
8. Rewrap the teflon tape around the vials

Step 2: Preparing Vials

1. Choose 12 samples for the current extraction batch and return the other samples to the refrigerator.
2. Each sample will require one 4-ml glass vial and two 1.5-ml glass autosampler vials. Each of these vials needs to be labeled with the sample's SEI number. When labeling the autosampler vials, make sure to label one with SEI # - A and the other with SEI # - B. The labeling process can be done while the microwave extraction (Step 3) is in progress.
3. Use the Dymo label software to print out labels for the vials. Attach the labels and organize the vials in racks for use later in the extraction process. NOTE: Make sure to affix labels as low as possible on the 1.5-ml autosampler vials so the autosampler does not encounter interference.

Step 3: Microwave Extraction

1. Assign a microwave vessel to each of the twelve samples in the batch and record that vessel number on the sample login sheet. NOTE: If there are both samples containing rock and samples not containing rock (e.g., trip blank) within the same extraction batch, make sure that the reference microwave vessel (Vessel #1) is used for a sample containing rock.
2. Turn on the microwave at the power switch. This also turns on the control screen. On the control screen, select Administrator and type in password (123456). Touch the PressPrep button and then go to the Method tab and the Param subtab. Make sure that the loaded method is rockcore.mpr and that the Twist and Start-Param buttons are checked. Also make sure that the Control for... dropdown menu is set to T1 and that Ventilation is set to 73 minutes. Use the vent button (looks like a fan) to rotate the rotor to access a certain vessel.
3. Prepare samples one at a time for microwave extraction via the following steps:.

a. Remove the appropriate segment from the rotor body

b. Place the workstation flat on the microwave door so that the ridge on the bottom edge is aligned with the base of the inside of the microwave for stability.

c. Using the torque wrench, unscrew the amber-colored cap of the microwave segment to release the vessel. Remove the vessel from the rotor segment by tilting it to the right and lifting it out.

d. Carry the vessel to the hood and open it by removing the brown spring and adapter plate, the teflon indicator ring, the teflon cover to expose the teflon vessel and the brown protection shield. Remove the vessel from the protection shield. There should be six separate pieces.

e. Empty the contents of the sample vial into the vessel.

f. Using the repipettor, introduce 5 ml methanol to the sample vial to rinse any remaining sample. Pour this rinse methanol into the vessel as well.

g. Weigh the teflon vessel with the sample in it and record the weight on the sample log in sheet.

h. Reassemble the entire vessel and return it to the segment. Use torque wrench to tighten down the cap until the wrench makes a loud click. Replace in the proper position within the rotor body and then move to the next vessel, using the vent button on the control screen to rotate the rotor if necessary.

i. After all vessels have been loaded with samples, put the round rotor cover on top of all the segments so they are locked into place. Install the temperature sensor by inserting the fiber optic sensor into the thermowell hole on the top of the reference vessel (vessel #1) until it is firmly set. Attach the other end of the temperature sensor (it is blue) to the microwave by plugging it into the top hole on the left inside wall of the microwave. Again, press in until it is firmly set.

j. To start the microwave extraction, press the green start button on the control screen. Before actually starting, the microwave will ask you two warning questions. The first asks you if you want to continue even if your pressure sensor is not monitoring. Say yes to continue. The second warning wants you to check your stirrer. We do not have this function enabled, so continue through this message too. At this point, the microwave program should begin, and proceed according to the rockcore.mpr method. The program should take two hours to complete. This includes a ventilation step that will cool the vessels down to approximately 30 degrees C. Turn the hood blower on.

k. Once the microwave program has completed, the ventilation fan will turn off. As long as the temperature is reading <30 degrees C, the vessels can be opened.

l. With the labeled 4-ml vials ready nearby, you can begin transferring the samples from the microwave vessels to the 4-ml vials. Start with the reference vessel and remove the temperature sensor from the reference vessel port and from the port inside the microwave. Place the temperature sensor on the shelf, out of harm's way. NOTE: Once you remove the sensor, the temperature reading on the control screen will spike to a very high reading. This is ok. Now remove the round rotor cover so that you can access the vessels.

m. From here, prepare the samples for the centrifuge step. The centrifuge can only accept six samples at a time, so transfer the samples from the microwave to the 4-ml vials in two batches of six samples. Proceed to transfer the samples one at a time via the following steps:

   i. Use the workstation and torque wrench to unscrew the cap and remove the vessel from its segment.

   ii. Carry the vessel into the hood and carefully remove the spring and plate, indicator ring and teflon cover. Remove the vessel from the protection shield. NOTE: Use extra caution when removing the teflon cover from the reference vessel. Lift it straight up to prevent cracking the thermowell.

   iii. Weigh the sample in the teflon vessel. Record the weight on the login sheet.

   iv. Using a fresh glass transfer pipette, transfer approximately 3 ml of sample from the teflon vessel to an appropriately labeled 4-ml glass vial.

   v. Pour the remaining sample volume into the methanol waste container and rinse it 3x with 5ml methanol from the repipettor.

   vi. Make sure the vessel is dry before you seal it back up. You may want to set up a drying area in the hood, but make sure you keep all components for each vessel together without mixing them. Each component should be marked with a number corresponding to its vessel number.

Step 4: Centrifuge and Transfer Samples to Autosampler Vials

1. Load six 4-ml sample vials into the metal tubes of the centrifuge (the tubes are labeled 1-6).
2. Close the lid and turn on the centrifuge by setting the dial to 30 minutes.
3. While the first six samples are being centrifuged, prepare the second batch of six samples for the centrifuge step (i.e., transfer to labeled 4-ml vials).
4. After the first batch has centrifuged for 30 minutes, unload those vials and reload the centrifuge with the second batch of samples. Centrifuge for 30 minutes.
5. Once the centrifuge step is complete, transfer each sample from the 4-ml vial to two appropriately labeled 1.5-ml autosampler vials. Place a septum on top of the vial and crimp it securely with the hand crimper until the septum will not move/rotate on the vial. Until you are ready to run the samples on the GC, keep them in the freezer.

Step 4: Decontamination of Extraction Vessels

1. After extraction and removal of methanol and rock sample, the vessels are first cleaned by rinsing with copious amounts of reagent free water to remove residual methanol and solids.

2. The vessels are then rinsed with methanol by placing 15 mL of methanol into each vessel, capping the vessel and shaking for approximately 10-seconds.
3. The final step is to bake each vessel at 110°C for 45-minutes.
4. The effectiveness of the decontamination procedure is demonstrated through the method blanks which include the vessels and are run at a once per day frequency. See QA/QC section below for further discussion of demonstration of decontamination procedure.

10.0 CALCULATIONS

None performed.

11.0 QUALITY CONTROL AND CORRECTIVE ACTION

Quality control samples are generated during sample preparation to determine if contamination or memory effects are occurring. Methanol blanks for all new microwave vessels are prepared before samples are extracted. Also, once a day, one microwave vessel blank is prepared. The vessel is chosen at random. It is also recommended to prepare microwave vessel blanks for the vessels used for the extraction of highly concentrated samples.

12.0 POLLUTION PREVENTION & WASTE MANAGEMENT

Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The United States Environmental Protection Agency (USEPA) has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

For information about pollution prevention that may be applicable to laboratories and research institutions, consult "Less is Better: Laboratory Chemical Management for Waste Reduction", available from the American Chemical Society's Department of Government Regulations and Science Policy, 1155 16th Street N.W., Washington, D.C. 20036; (202) 872-4477.

The USEPA requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. Excess reagents, samples, and method process wastes should be characterized and disposed of in an acceptable manner. The Agency urges laboratories to protect the air,

water and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management consult the "Waste Management Manual for Laboratory Personnel", available from the American Chemical Society.

13.0 REFERENCES

1. Yongdong Liu, "Microwave Assisted Rapid Extraction of VOCs from Low Permeability Media", M.Sc. thesis, University of Waterloo, 2005.

2. Pawliszyn, Janusz, 1999, Applications of Solid Phase Microextraction, in RSC Chromatography Monographs; Smith, Roger S., Series Editor, Royal Society of Chemistry, Cambridge, UK, 655 p.

3. "Less is Better: Laboratory Chemical Management for Waste Reduction", available from the American Chemical Society's Department of Government Regulations and Science Policy, 1155 16th Street N.W., Washington, D.C. 20036; (202) 872-4477.

14.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

Table 1. Example Sample Login Sheet

ROCK CORE LABORATORY SAMPLE LOGIN								
Sample ID	SEI Number	Sample weight as received (sample + container)	Microwave Vessel ID	Pre-Sample Weight (Vessel and Lid)	Pre-Microwave Weight (sample + teflon vessel and lid)	Post-Microwave Weight (sample + teflon vessel and lid)	Analyst	Extraction Date
		(g)		(g)	(g)	(g)		
			1					7/1/2008
			2					7/1/2008
			3					7/1/2008
			4					7/1/2008
			5					7/1/2008
			6					7/1/2008
			7					7/1/2008
			8					7/1/2008

15.0 AUTHORIZATION

Authored by: _____ Date: _____

Michael Rossi, Laboratory Manager

Approved by: _____ Date: _____

Seth Pitkin, Vice President

16.0 REVISIONS

STANDARD OPERATING PROCEDURE

SEI-10.18.0

THE DETERMINATION OF VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY / DUAL ECD DETECTORS IN ROCK SAMPLES (USING COOL ON COLUMN INJECTION AND SPLIT METHOD INJECTION)

NOTE: This SOP contains proprietary information – Do Not Distribute

SOP Number: SEI-10.18.0 | Date Issued: 07/02/08
Revision Number: 0 | Date of Revision: na

1.0 OBJECTIVE

The method was developed at the University of Waterloo and is used to determine the concentration of volatile organic compounds (VOC) in soil and rock samples using an automated cool on-column injection and/or split method for highly sensitive determination of chlorinated solvents. The method was tested on chlorinated solvents presented in Table 1 and Table 2. For the automated cool on-column injection method, the calibration range is typically from 1.0 µg/L to 500 µg/L for the tri- and tetra-chlorinated compounds and 10 µg/L to 5000 µg/L for the dichlorinated compounds. For the split method, the calibration range is typically from 50 µg/L to 5,000 µg/L for the tri- and tetra-chlorinated compounds and 500 µg/L to 50,000 µg/L for the dichlorinated compounds. Higher values in the samples are measured by appropriate dilution of the samples.

2.0 SUMMARY OF METHOD

The analyses are performed on a Hewlett Packard model 6890 Gas Chromatograph with computer control. Methanolic extract is injected directly into a polar-deactivated pre-column connected to the analytical column. For the cool on-column method, the sample is injected using a cool on-column injector, set to ramped temperature mode. In the split method, the methanolic extract is injected using split-splitless injector (ratio 1:100) into the analytical column. A column coated with a thick film non-polar stationery phase (5 µm, HP-1, equivalent) is used in the method. Methanol is incompatible with this phase, and consequently elutes in dead time. The less polar analytes, refocused through the retention gap effect, are retained much more strongly and elute later, even when their boiling points are lower than that of MeOH. The use of non-polar stationary phase reduces the possibility of methanol coeluting with one or more of the analytes, which could adversely affect their chromatography and detection. Figure 1 presents a sample chromatogram obtained for a standard solution of the analytes obtained using

the cool on-column injection method and Figure 2 presents a sample chromatogram obtained for a standard solution of the analytes using the split method.

Core samples are collected and the rock samples are immediately crushed using a device that was designed by the University of Waterloo. For further details on the sampling and preservation technique associated with rock samples, see the Stone's SOP titled "*SEI-6.37.0 Field Methods for Retrieval, Collection, Handling, and Preservation of Rock Samples to be Analyzed for VOCs and Physical Properties*".

The crushed samples then undergo microwave assisted extraction (MAE). For further details on the MAE procedure, see Stone's SOP titled *"SEI-10.17.0 Microwave Assisted Extraction of Volatile Organic Compounds from Rock Samples"*. Typical solvent extractions using shaking or sonication require five weeks in order to extract all of the contaminant mass even following crushing; using MAE this extraction time is reduced to less than an hour.

3.0 DEFINITIONS

Accuracy: the degree of agreement between a measurement and true or expected value, or between the average of a number of measurements and the true or expected value.

Batch: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of a similar matrix, meeting the above-mentioned criteria.

Corrective Action: action taken to eliminate the causes of an existing non-conformance, defect or other undesirable situation in order to prevent recurrence.

Instrument Blank: a blank matrix that is the same as the processed sample matrix (i.e. extract, digestate, condensate) and introduced onto the instrument for analysis.

Laboratory Control Sample (LCS): a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

Matrix: the substrate of a test sample.

Method Blank: a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

Method Detection Limit: Method detection limits are determined according to the method described in "US EPA's Methods for Organic Chemical Analysis in Industrial Wastewater", EPA-600/4-82-057. For

the purposes of this protocol, a sample of known concentration is analyzed 9 times. The results are averaged, and the standard deviation of the results is calculated. The standard deviation multiplied by the value of Student's t for 8 degrees of freedom (n-1) at the 99% confidence level is considered to be equal to method detection limit. Limit of quantitation is defined as method detection limit multiplied by 3. Reporting limit is set equal to the lowest calibration standard.

Precision: an estimate of variability. It is an estimate of agreement among individual measurements of the same physical or chemical property, under prescribed similar conditions.

Quality Assurance (QA): an integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.

Quality Control (QC): the overall system of technical activities, the purpose of which is to measure and control the quality of a product or service.

Quality Control Sample: a control sample, generated at the laboratory or in the field, or obtained from an independent source, used to monitor a specific element in the sampling and/or testing process.

Quantitation Limit (QL): the minimum amount of a substance that can be quantitatively measured with a specified degree of confidence and within the accuracy and precision guidelines of a specific measurement system. The QL can be based on the method detection limit (MDL), and is generally calculated as 3-5 times the MDL, however, there are analytical techniques and methods where this relationship is not applicable. Also referred to as Practical Quantitation Level (PQL), Estimated Quantitation Level (EQL), Limit of Quantitation (LOQ).

Reporting Limit (RL): The level to which data is reported for a specific test method and/or sample. The RL is generally related to the QL. The RL must be minimally at or above the MDL.

Spike: a known amount of an analyte added to a blank, sample or sub-sample.

Matrix Spike (MS): field sample to which a known amount of target analyte(s) is added.

Standard Operating Procedure (SOP): a written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks.

VOC – Volatile Organic Compounds

QA/QC – Quality Assurance/Quality Control

4.0 INTERFERENCES

When analyzing for VOCs, samples can be contaminated by diffusion of volatile organics (particularly chlorofluorocarbons, chloroform and methylene chloride), through the sample container septum during shipment and storage. A trip blank prepared from organic-free methanol and carried through sampling and subsequent storage and handling must serve as a check on such interferences.

Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed in sequence. To reduce the potential for carryover, the sample syringe or purging device must be rinsed out between samples with an appropriate solvent. Whenever an unusually concentrated sample is encountered, it should be followed by injection of a method blank to check for cross contamination. Extraction vessels and syringes should be adequately cleaned and flushed prior to use. All glassware must be kept scrupulously cleaned. Clean all glassware as soon as possible after use by rinsing with the last solvent used or analyte-free water. Clean, dry glassware should be stored in a clean environment. There are some solvents, which cannot be separated in this method. On the non-polar column used as part of this method, bromodichloromethane always coelutes with trichloroethene and 1,1,1-Trichloroethane and 1,2-Dichloroethane can only be analyzed as a sum using the method. Also, VOC gases such as vinyl chloride and chloromethane can not be determined by this method.

5.0 SAFETY ISSUES

Laboratory personnel should take extra care when working with standards. When working with neat standards, make certain that there is adequate ventilation and that Nitrile or Latex gloves are worn and if necessary safety glasses.

If necessary and appropriate, a site-specific health and safety plan shall be created for each study site and followed. All samples as well as standards should be treated as a potential health hazard. Exposure to each should be reduced to the lowest level possible using gloves and a hood. Material Safety Data Sheets (MSDS) should be available to all personnel.

Field visits may involve accessing remote areas. Health and safety concerns regarding these field visits are minimal, however, hazards such as slip, trip and falls, poisonous plant and dangerous animals, as well as getting vehicles stuck in remote areas, do present considerable health and safety issues. To help ensure field staff's health and safety in remote areas, all field staff are to have daily communication with the project manager or another appropriate SEI employee.
All chemicals are required to be received with a Material Safety Data Sheets (MSDS). MSDSs shall be made available to all personnel involved in the sampling and testing.

6.0 EQUIPMENT AND SUPPLIES

1. Gas Chromatograph: Analytical system complete with gas chromatograph and all required accessories, column supplies, gases, syringes and data system to determine peak areas and perform integrations. Agilent (formerly Hewlett Packard) 6890 with computer control.
2. Cool on-column injector
3. Split-splitless injector
4. Dual ECD Detectors
5. Two auto sampler towers
6. Printer
7. Analytical Columns: Two capillary columns
8. Array of Microsyringes and glass disposable pipettes
9. Balance: Top loading, capable of weighing accurately to 0.01 grams
10. 1.5 mL glass GC vials and caps
11. Crimper
12. 4 mL glass vials
13. VOA vials: 40 mL collection containers with Teflon®-lined septum

7.0 REAGENTS AND STANDARDS

Organic-free reagent water demonstrated to be free of compounds of interest (spring water, carbon filtered and/or deionized).

Purge and Trap Grade or equivalent grade methanol (demonstrated to be free of analytes and stored away from other solvents).

Stock standards:
Stock standards may either be prepared from pure standard materials or purchased as certified solutions.

Secondary dilution standards are prepared using stock standard solutions, which contain the compounds of interest, either as single compounds or mixed together. Typically, for this method the stock standards are obtained from an approved vendor and mixed together. A certificate of analysis is retained by the laboratory and maintained on file or on file with the vendor.

Stone's SOP No. SEI-4.7.5 *Labeling, Preparation and Storage of Reagents, Solutions and Standards* provides procedures on properly labeling, preparing and storing of reagents and standards used in the

mobile laboratory for analytical methods. This SOP should be reviewed by the analyst before labeling, preparing and storing standards within the mobile laboratory.

8.0 SAMPLE COLLECTION AND HANDLING

8.1 Sample Collection

Rock core samples are collected by trained geologists. Core samples are collected and the rock samples are immediately crushed using a device that was designed at the University of Waterloo. The core sample is placed in a closed stainless steel crushing cell fitted with "O"-rings and crushed with a stainless steel piston at pressures as high as 3000 psi. The crushed sample is then extruded from the cylinder directly into a pre-weighed 40 mL VOA vial containing a known volume of purge and trap grade methanol. The amount of air passing by the sample while being crushed is minimized as well as the time required to completely crush the rock fragments into the desired particle size. The stainless steel crushing vessels and associated tools are thoroughly decontaminated following each use. The samples are weighed upon receipt at the laboratory and recorded in the Log in summary sheet.

The crushed samples then undergo microwave assisted extraction (MAE).

8.2 Handling and Holding Times

Rock samples collected in methanol are stored chilled at 4° C (<6 °C) from collection. Samples are allowed to come to room temperature just prior to analysis.

If necessary, a separate rock sample aliquot will be collected in a 40 mL vial or equivalent for percent moisture content so that results can be adjusted for %moisture and reported as dry weight rather than as received. Percent moisture is determined as follows: Percent Moisture Determination - Immediately after weighing the sample for analysis, weigh 5-10 g of the rock into a tared crucible. Determine the percent moisture by drying overnight at 105 °C. If available, allow to cool in a dessicator before weighing. Concentrations of individual analytes will be reported relative to the dry weight of rock.

Percent moisture - %Moisture = gms of wet sample – gms of dry sample/gms of wet

9.0 PROCEDURES

Personnel Qualifications: The chemist performing these analyses should have substantial experience operating and troubleshooting gas chromatographs. Additionally, the analyst must be comfortable using microliter syringes and be familiar with basic analytical chemistry methodology and principles. The assessment of these qualifications is somewhat subjective but an analyst should normally be considered experienced after completing a B.Sc. in chemistry with one-year experience with gas chromatography in an analytical laboratory.

9.1 Typical GC Program for Back Inlet (Cool On-Column) and Front Inlet (split/splitless)

Oven Temp: 55°C (4.50 min), ramp: 10°C /min to 180°C, hold for 3 min.

Flow Rate: Cool on column, helium at 2.0 mL/min; Split/Splitless, helium at 2.3 mL/min.

Detector: uECD front and back detectors, detector temperature 350°C

Injector: 1 uL injection on both front and rear inlets

Make-up gas: Nitrogen

Make-up gas flow: 60 mL/min

Cool on-column injector temperature: 55°C (0.0 min), ramp: 75°C /min to 220°C, hold for remainder of run.

9.2 GC Column

Column 1: Cool on column: Agilent HP-1 30.0 m x 0.320mm ID, 5 um film thickness, equivalent

Column 2: Split/splitless: Supelco SPB-1, 30 m x 0.250mm ID, 3 um film thickness, equivalent

9.3 Material Preparation:

Methanolic standards of the compounds of interest are prepared from a stock solution made by spiking pure chemicals of interest into purge and trap-grade methanol. Analyte concentrations for the standard solutions are calculated by mass. Methanolic standards should be kept in the refrigerator when not in use.

These standards are then diluted in methanol to obtain working (calibration) standards at the required concentrations. The working standards are analyzed in the same manner as the samples.

9.4 Calibration Criteria

Initial Calibration (ICAL):

Six to eight-point calibration for VOC compounds: For the automated cool on-column injection method, the calibration range is typically from 1.0 µg/L to 500 µg/L for the tri- and tetra-chlorinated compounds and 10 µg/L to 5000 µg/L for the dichlorinated compounds. For the split method, the calibration range is typically from 50 µg/L to 5,000 µg/L for the tri- and tetra-chlorinated compounds and 500 µg/L to 50,000 µg/L for the dichlorinated compounds.

The linearity of the calibration curves must be assessed and are used for all quantitation unless it is necessary to drop the high point or the low point. Linear regression is used for quantitation

and the correlation coefficient (r) of each compound must be greater than 0.995. Depending upon the on-site program acceptable correlation coefficient may be set at 0.99 in accordance with SW846 8000.

Initial Calibration Verification Standard (ICV):

A QC sample from a second (independent) source will be analyzed directly after the ICAL and after every 40 samples. The ICV sample will contain all the VOC compounds of interest at the mid-point. Recovery Limits for the ICV will be ±15%,

%Rec. = Actual Conc./ Expected Conc.

Retention Time Windows and Pattern Recognition

Identification of the project- specific VOCs in the sample chromatogram is achieved by comparing the retention times generated by the calibration standards, continuing calibration standard and other fortified QC samples. Retention times must be within ± 0.04 minutes from the ICAL. If a retention time shift is observed in the CCV or daily ICV, then adjustments to the daily retention time windows will be made accordingly. Quantification of the VOCs is determined by comparison of the linear regression for that analyte from the initial calibration curve.

9.5 Sample Preparation, Analytical Sequence and Quality Assurance

Methanolic Sample: Using the Microwave Assisted Extraction (MAE) method (SOP SEI-10.17.n), the rock sample is transferred quantitatively from the vial into a 100 mL microwave vessel and prepared, following which a 5 mL aliquot of methanol is used to rinse the original sample vial. The vessel is weighed before and after addition of the sample and the rinse methanol. The vessel is then tightly sealed in the segment and is microwaved for 40 minutes at 120°C; with temperature ramp and cool down, the overall extraction time is usually one-hour and ten minutes. Twelve vessels can be microwaved at the same time. A 4 mL aliquot is taken from the 100 mL extraction vessel for the analysis. The remaining extract is then either stored or discarded, depending on project's needs. Only the MeOH aliquot is kept (no rock). A GC autosampler vial (about 1 mL of aliquot in each vial) is then prepared for the autosampler.

Analyze Samples on GC/ECD

1. In the Method & Run Control screen of the online method of the GC software, go to Sequence: Sequence Table to load your run sequence(s) for the front and/or back system.
2. Once the sequence has been established, go to Sequence: Sequence Parameters, to set up the data file structure for the run. Type in your initials for Operator Name, Under Data File, select Prefix/Counter and type in A and today's date under Prefix for Signal 1 (ex.

A80606) and B and today's date under Prefix for Signal 2 (ex. B80606). The counter will update itself automatically to create a unique file name for each run.

3. Save sequence by going to Sequence □ Save Sequence As and typing in today's date (ex. 080606.S).
4. Print the sequence for the run log by going to Sequence □ Print Sequence. Once in this screen, check the Sequence Parameters box at the top. Under Sequence Table, check the Method and Injection Info Part box. Under Select Destination for Printout, select File and type the name of the sequence in the box (ex. 080606.TXT). Click Print and then go open the text file that was written. Print from there.
5. Load sample vials into the autosampler, making sure that the loading order matches that on the sequence printout.
6. When everything is ready, click Run Control, Run Sequence.

LCS or Second Source Preparation

Every 40 samples, a fortified blank, containing all of the targets and prepared from a source other than the material used for the calibration curve is analyzed. The QC limits are 80-120%.

Laboratory Duplicate:

Every 20 samples, a random sample is run in duplicate and the results are compared. The relative percent difference (RPD) should not exceed 15%.

Matrix Spike:

Every 40 samples, a matrix spike will be prepared and will contain only the VOC compounds of interest.

Methanol blanks

Every 20 samples. Methanol blanks should produce results lower than the lowest calibration standard

9.6 Data and Records Management

Samples are analyzed in sequences. The name of the sequence is set to be the date on which the analysis is performed. A chromatogram is printed for each analysis and stored in a binder. A sequence table is printed out and stored with the chromatograms of a given sequence. In addition, all chromatogram files are stored electronically. The file and the printed chromatogram can be easily accessed at any time because the analysis date is recorded in the report.

10.0 CALCULATIONS

All calculations are performed by the HP ChemStation software used to control the gas chromatograph. The calculations used to determine the concentration of a compound in an unknown sample are based on the External Standard (ESTD) procedure, in which both calibration standards and unknown samples are analysed under the same conditions. The instrument is first calibrated using a set of seven to nine working standards, prepared according to the procedure described in paragraph 9. The software calculates the calibration curve, which is later used to convert analyte peak areas in the unknown samples into their concentrations. All calculations are performed automatically by choosing appropriate settings in the software.

Typical Reporting qualifiers are as follows:

B = Analyte is found in the associated method blank as well as the sample.

D = Compound is identified in an analysis at a secondary dilution factor.

E = Compound quantitation is above the instrument's calibration range for this analysis.

J = Indicates an estimated quantitation value.

U = Compound was analyzed for but not detected. Value reported is the method reporting imit.

11.0 QUALITY CONTROL AND CORRECTIVE ACTION

A new calibration curve is made at least once a week. A quality control (QC) sample set is analyzed every 20 samples. The QC set consists of a laboratory duplicate, methanol blank and one standard check. The laboratory duplicates and the standard checks should agree within 15% RPD and recovery, respectively. Methanol blanks should produce results lower than the lowest calibration standard. Second source standard or a laboratory control sample should be analyzed every 40 samples, and the results for all the analytes should fall within 20% of the expected values.

Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Initial Calibration (ICAL)	As necessary.	Minimum of 5 up to 8 points for VOCs. A correlation factor (r2) of 0.99 for linear regression.	Verify solution integrity and check instrument performance. Perform necessary maintenance and recalibrate instrument. Reanalyze all affected samples.
Initial Calibration Verification (ICV) and/or Laboratory Control Sample (LCS)	One per calibration (following acceptable ICAL) and every 40 samples	QC limits are 80-120% for target compounds. This same standard may be used as the daily LCS. Note: Limits may be adjusted based on the program should as required by DOD QMS.	Reanalyze once; if still out verify solution integrity or ICAL solution integrity, and instrument performance. Re-prepare and reanalyze all associated samples.
Retention Time (RT) Window Study	Every new column installation	All target compounds in all standards must fall within the established window of ± 0.04 minutes from ICAL	Perform system maintenance. Reanalyze affected samples.
Continuing Calibration Verification (CCV) or Standard checks	To be performed at the start of the analytical day and every 20 samples	± 15%D for VOCs	Verify solution integrity and instrument performance. Reanalyze standard once, if still out, recalibrate and reanalyze affected samples.
Matrix Spike (MS)	One per 40 samples of a similar matrix	QC limits are 70-130%D.	Reanalyze once; if still out, verify solution integrity and instrument performance. If necessary analyze a LCS or ICV and if acceptable, narrate as possible matrix effect.
Method Blanks	One per preparation batch	< the lowest calibration standard or reporting limit	Investigate source of contamination. Re-prepare and reanalyze all associated samples.
Laboratory duplicate	One set per 20 samples of a similar matrix	QC limits are 15 RPD for all detected compounds.	Reanalyze once; if still out, verify solution integrity and instrument performance. If necessary analyze a LCS or ICV and if acceptable, narrate as possible matrix effect.

11.1 Data Review

The analyst is responsible for primary data review of data generated from the sample analysis. Analyses will be documented in the instrument run log. Maintenance is documented in the Instrument Maintenance Logbook. Instrument calibrations and recoveries of all QC samples must be within specified control limits. If instrument calibration or the recoveries of any QC sample exceed specified tolerances, then the affected sample results are evaluated and generally the samples are

submitted for re-analysis. Manual integrations should be kept to a minimum and date and initialed by the analyst. To determine if analytical results are acceptable, a qualified and independent QA/QC program shall conduct a secondary review on a weekly basis. If instrument calibration and recoveries of all QC samples are within the specified criteria, then the data reports will be submitted to the Project Manager as final results with no qualifiers. If recoveries of any QC samples exceed specified limits and re-analysis is not an option, then the samples will be qualified as estimated with a "J" qualifier (J= The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.). Data will not be reported if significant QC issues affect the batch analyses.

12.0 POLLUTION PREVENTION & WASTE MANAGEMENT

Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The United States Environmental Protection Agency (USEPA) has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

For information about pollution prevention that may be applicable to laboratories and research institutions, consult "Less is Better: Laboratory Chemical Management for Waste Reduction", available from the American Chemical Society's Department of Government Regulations and Science Policy, 1155 16th Street N.W., Washington, D.C. 20036; (202) 872-4477.

The USEPA requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. Excess reagents, samples, and method process wastes should be characterized and disposed of in an acceptable manner. The Agency urges laboratories to protect the air, water and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management consult the "Waste Management Manual for Laboratory Personnel", available from the American Chemical Society.

13.0 REFERENCES

1. Maria Gorecka, T. Gorecki, B.L. Parker, "Automated Cool On-Column Injection for Highly Sensitive Determination of Chlorinated Solvents in Soils and Rocks", 24th International Symposium on Capillary Chromatography and Electrophoresis, Las Vegas, NV, May 20-24, 2001.

2. "Less is Better: Laboratory Chemical Management for Waste Reduction", available from the American Chemical Society's Department of Government Regulations and Science Policy, 1155 16th Street N.W., Washington, D.C. 20036; (202) 872-4477.
3. http://www.epa.gov/epaoswer/hazwaste/test/pdfs/8260b.pdf

14.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

Table 1. Summary of the typical detection limits for the on-column method.

Compound	[1]Method Detection limit [3]µg/L]	[1]Method Detection Limit [4]µg/Kg]	Reporting Limit [3]µg/L]	Reporting limit [4]µg/Kg]
1,1-Dichloroethene	0.6	.6	10	10
trans-1,2-Dichloroethene	1.2	1.2	10	10
cis-1,2-Dichloroethene	.29	.29	10	10
Trichloroethene	[2]0.2	[2]0.2	1.0	1.0
Tetrachloroethene	[2]0.2	[2]0.2	1.0	1.0
Trichlorotrifluoroethane	[2]0.2	[2]0.2	1.0	1.0
Chloroform	[2]0.7	[2]0.7	1.0	1.0
1,1,1-Trichloroethane	[2]0.1	[2]0.1	1.0	1.0
Carbon Tetrachloride	[2]0.1	[2]0.1	1.0	1.0

1 = Method detection limits (MDLs) are determined according to the method described in "US EPA's Methods for Organic Chemical Analysis in Industrial Wastewater", EPA-600/4-82-057.
2 = MDLs for these compounds have been adjusted to ten times their statistically derived values so that they can be practically achieved by the method.
3 = Limit is given as mass per volume of methanol extract.
4 = Limit is given as mass per mass of wet rock – assumes 20 ml methanol is combined with 20 grams of wet rock.

Table 2. Summary of the typical detection limits for the split method.

Compound	[1]Method Detection limit [2]µg/L]	[1]Method Detection Limit [3]µg/Kg]	Reporting Limit [2]µg/L]	Reporting limit [3]µg/Kg]
1,1-Dichloroethene	200	200	500	500
trans-1,2-Dichloroethene	230	230	500	500
cis-1,2-Dichloroethene	190	190	500	500
Trichloroethene	19	19	50	50
Tetrachloroethene	17	17	50	50
Trichlorotrifluoroethane	25	25	50	50
Chloroform	18	18	50	50
1,1,1-Trichloroethane	22	22	50	50
Carbon Tetrachloride	22	22	50	50

1 = Method detection limits (MDLs) are determined according to the method described in "US EPA's Methods for Organic Chemical Analysis in Industrial Wastewater", EPA-600/4-82-057.
2 = Limit is given as mass per volume of methanol extract.
3 = Limit is given as mass per mass of wet rock – assumes 20 ml methanol is combined with 20 grams of wet rock.

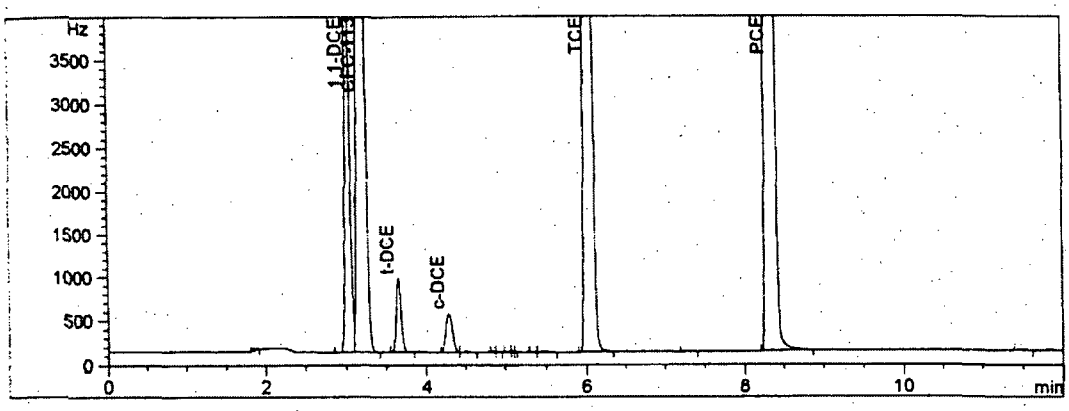


Figure 1. Example of a chromatogram obtained for a standard solution of the analytes using the cool on column method

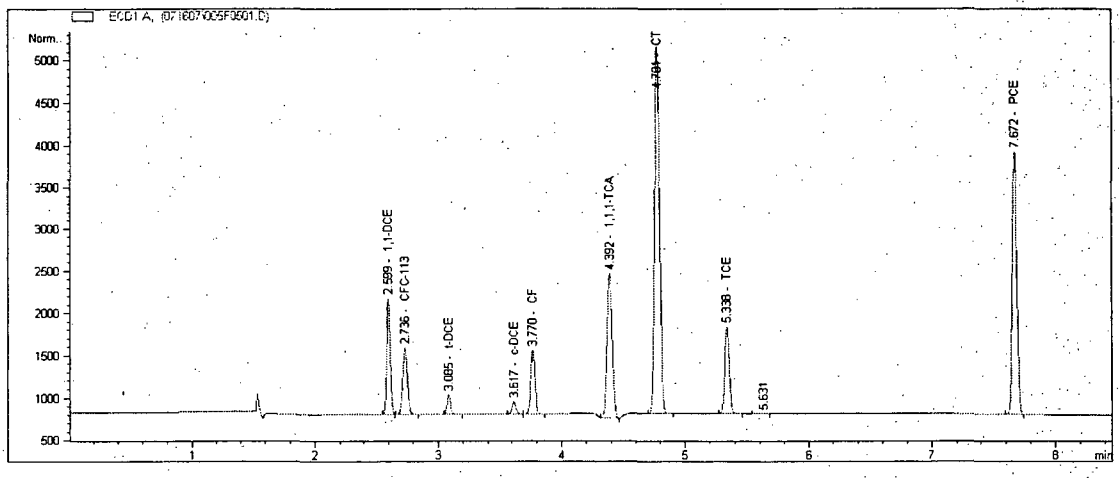


Figure 2. Example of a chromatogram obtained for a standard solution of the analytes using the split method.

AUTHORIZATION

Authored by: _____ Date: _____

Michael Rossi, Laboratory Manager

Approved by: _____ Date: _____

Seth Pitkin, Vice President

15.0 REVISIONS

Attachment 13

Accutest Laboratory Procedures

ACCUTEST LABORATORIES
STANDARD OPERATING PROCEDURE

Lab Manager:_____

QA Manager:_____

Effective Date: _____

TITLE: PROCEDURE FOR PARTICLE SIZE REDUCTION (CRUSHING) OF SOLID MATRICES

REFERENCES: None

Revised Sections: ***NA – new SOP***

1.0 SCOPE & APPLICATION

1.1 This procedure is used, on client request, for the crushing and particle size reduction of specified solid samples before digestion and/or extraction procedures.

2.0 SUMMARY

2.1 The samples are crushed into small pieces using a variety of physical techniques, including a mechanical crusher, and a hammer.

3.0 REPORTING LIMIT AND METHOD DETECTION LIMIT

3.1 Reporting limit. Refer to determinative method for reporting limit information.

3.2 Method Detection Limit. Refer to determinative method for reporting limit information

4.0 DEFINITIONS

BATCH: A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

FIELD DUPLICATES: Independent samples that are collected as close as possible to the same point in space and time. They are two separate samples taken from the same source, stored in separate containers, and analyzed independently. These duplicates are useful in documenting the precision of the sampling process.

MATRIX: The component or substrate (e.g., surface water, drinking water) which contains the analyte of interest.

REAGENT WATER: Water that has been generated by any method that would achieve the performance specifications for ASTM Type II water. For organic analyses, see the definition of organic-free reagent water.

5.0 HEALTH & SAFETY

5.1 The analyst should follow normal safety procedures as outlined in the Accutest Laboratory Safety Manual which includes the use of safety glasses, lab coats, and gloves.

6.0 SAMPLE STORAGE

6.1 All samples must be stored as specified in the determinative method until the analyst is ready to begin the particle size reduction processs. After particle size reduction, the samples should be labeled and returned to the appropriate storage until the time of digestion or extraction.

7.0 APPARATUS AND MATERIALS

7.1.1 Ziploc bags.

7.1.2 Hammer

7.1.3 Mixing Bowl – stainless steel or ceramic.

7.1.4 Mechanical Crusher

7.1.5 Spatula – disposable wood or stainless steel. Inspect spatulas to make sure that there are no chips or rust before use.

7.1.6 Balance, capable of recording to 2 places past the decimal.

8.0 STANDARDS & REAGENTS

8.1 None. Refer to appropriate digestion, extraction, and analytical methods.

9.0 PROCEDURE

9.1 Remove a homogenous aliquot of the sample for crushing. If the sample is non-homogeneous, then obtain representative parts of the various fractions of the sample and combine.

9.2 If the sample is easily broken, and phthalate contamination is not an issue, than place the sample in a clean ziploc bag, close tightly, and crush to a a particle size of approximately 1 cm or less.

9.2.1 This is the prefered handling for metals samples if the crushing can be done without using the mechanical crusher.

9.3 Alternatively, the sample may be placed into a large stainless steel or ceramic bowl and physically broken using a hammer or rod. Make sure that this is done with a cover or in a hood so that particles are not dispensed into the lab.

9.3.1 For metals samples, use the ceramic bowl if possible.

9.4 For samples that will not easily crush, use the mechanical crusher. Break the sample into large pieces that will fit into the crusher. Place the cleaned tray under the crusher and hold it in place. Turn the power on the crusher and continue to crush pieces until sufficient volume is obtained to complete the required analyses.

9.5 Make sure to clean the mixing equipment well between each sample, by rinsing at least three times with deionized water. If there is an oily residue on the mixing equipment, then it should be washed with soap and water and then rinsed a minimum of three times with deionized water between samples.

9.5.1 An optional solvent wipe may also be done after the soap and water wash if oily residues still remain. Check with the supervisor for the appropriate solvent to use. Normally methanol is suggested for this application.

9.6 Stir the crushed sample well and then weigh out sample aliquots as described in the determinative method. Store the crushed sample separately from the original sample in a bottle that is clearly labeled that the crushing process has been completed.

10.0 QUALITY ASSURANCE

10.1 Refer to the extraction, digestion, and analytical methods for appropriate quality control.

11.0 DOCUMENTATION

11.1 All information must be recorded in the particle size reduction (crushing) log including the type of sample (i.e. cement), the type of crushing technique used, the analyst initials, and the date. Sample weight is not required.

11.2 Internal chain of custody must be in place for all sample transfers.

12.0 POLLUTION PREVENTION & WASTE MANAGEMENT

12.1 Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 12.2.

12.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the waste management SOP, ESM003. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

12.2.1 Non hazardous aqueous wastes.

12.2.2 Hazardous aqueous wastes

12.2.3 Chlorinated organic solvents

12.2.4 Non-chlorinated organic solvents

12.2.5 Hazardous solid wastes

12.2.6 Non-hazardous solid wastes

13.0 ADDITIONAL REFERENCES

13.1 Refer to digestion, extraction, and analytical SOP's for additional information.

ACCUTEST LABS
STANDARD OPERATING PROCEDURE

Lab Manager_____

QA Manager_____

Effective Date: _____

TEST NAME: NITRATE/NITRITE AND NITRATE ONLY BY CADMIUM REDUCTION ANALYSIS (Lachat Autoanalyzer).

METHOD REFERENCE: EPA 353.2 and EPA 353.2 modified for soils by Lachat

Revised Sections: ***9.7, 9.7.1, 9.11, 11.2***

1.0 SCOPE AND APPLICATION

1.1 This method is based on EPA method 353.2 and used as a measure of the nitrate/nitrite or nitrate only in water and wastewater samples. A modification of the method can be used as a measure of the nitrate/nitrite in soil samples. The nitrate is reduced to nitrite by a cadmium reduction column. The nitrite is then determined by diazotizing with sulfanilamide and coupling with N-(1-napthyl)-ethylenediamine dihydrochloride to form a high colored azo dye which is measured colorimetrically.

1.2 This method is modified from the watstewater revision of EPA 353.2 in that the application is applied to a Lachat Autoanalyzer instead of the Technicon Autoanalyzer referenced in the method. This method is not modified from the drinking water revision of EPA 353.2. This method is modified for the preparation of soils.

2.0 PROCEDURE SUMMARY

2.1 A filtered sample is passed through a column containing granulated copper-cadmium to reduce nitrate to nitrite. The nitrite (that originally present plus reduced nitrate) is determined by diazotizing with sulfanilamide and coupling with N-(1-naphthyl)-ethylenediamine dihydrochloride to form a highly colored azo dye which is measured colorimetrically. Separate, rather than combined nitrate-nitrite, values are readily obtained by carrying out the procedure first with, and then without, the Cu-Cd reduction step.

3.0 REPORTING LIMIT AND METHOD DETECTION LIMIT

3.1 Reporting Limit. The reporting limit for this method is established at the lowest concentration standard in the calibration curve. Detected concentrations below this concentration cannot be reported without qualification.

3.2 Method Detection Limit. Experimentally determine MDLs using the procedure specified in 40 CFR, Part 136, Appendix B. This value represents the lowest reportable concentration of an individual compound that meets the method qualitative identification criteria.

3.2.1 Experimental MDLs must be determined annually for this method. (MDL's are required every 6 months for drinking water matrices.)

3.2.2 Process all raw data for the replicate analysis in each MDL study. Forward the processed data to the QA group for archiving.

4.0 DEFINITIONS

BATCH: A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

CALIBRATION CHECK STANDARD. The calibration check standard is a mid-range calibration standard. It is recommended that the calibration check standard be run at a frequency of approximately 10 percent. (For some methods this is mandatory and for some it is a recommendation only. Refer to individual method SOP's) For most methods, the mid-level calibration check standard criteria is ± 10 percent of the true value. The exception to this rule is if the recovery on the calibration check standard is high and the samples to be reported are less than the detection limit.

EXTERNAL CHECK STANDARD. The external check standard is a standard from a separate source than the calibration curve that is used to verify the accuracy of the calibration standards. An external check must be run a minimum of once per quarter for all analyses where a check is commercially available. The laboratory should initially assess laboratory performance of a check standard using the control limits generated by the external check supplier. In house limits should also be generated once sufficient external check standard data is available to generate limits (usually a minimum of 20 to 30 analyses). If the external check is outside of the control limits for a given parameter, all samples must be reanalyzed for that parameter after the problem has been resolved.

SPIKE BLANK OR LAB CONTROL SAMPLE. Digest and analyze a laboratory control sample or spike blank with each set of samples. A minimum of one lab control sample or spike blank is required for every 20 samples. Assess laboratory performance against the control limits specified in the SOP. In house limits should also be generated once sufficient external check standard data is available to generate limits (usually a minimum of 20 to 30 analyses). If the lab control or spike blank is outside of the control limits for a parameter, all samples must be redigested or redistilled and reanalyzed for that parameter. The exception is if the lab control or spike blank recovery is high and the results of the samples to be reported are less than the reporting limit. In that case, the sample results can be reported with no flag. Note: If control limits are not specified in the SOP, then default limits of 80 to 120 percent should be used.

MATRIX: The component or substrate (e.g., water, soil) which contains the analyte of interest.

MATRIX DUPLICATE: A duplicate sample is digested at a minimum of 1 in 20 samples. The relative percent difference (RPD) between the duplicate and the sample should be assessed. The duplicate RPD is calculated as shown below. Assess laboratory performance against the control limits that are specified in the SOP. In house limits are generated once sufficient duplicate data is available to generate limits (usually a minimum of 20 to 30 analyses). If a duplicate is out of control, flag the results with the appropriate footnote. If the sample and the duplicate are less than

5 times the reporting limits and are within a range of ± the reporting limit, then the duplicate is considered to be in control. Note: If control limits are not specified in the SOP, use default limits of ± 20% RPD.

$$\frac{(|\text{Sample Result} - \text{Duplicate Result}|) \times 100}{(\text{Sample Result} + \text{Duplicate Result})/2} = \text{Duplicate RPD}$$

MATRIX SPIKE: The laboratory must add a known amount of each analyte to a minimum of 1 in 20 samples. The matrix spike recovery is calculated as shown below. Assess laboratory performance against the control limits that are specified in the SOP. In house limits are generated once sufficient matrix spike data is available to generate limits (usually a minimum of 20 to 30 analyses). If a matrix spike is out of control, then the results should be flagged with the appropriate footnote. If the matrix spike amount is less than one fourth of the sample amount, then the sample cannot be assessed against the control limits and should be footnoted to that effect. Note: If control limits are not specified in the SOP, then default limits of 75 to 125 percent should be used.

$$\frac{(\text{Spiked Sample Result} - \text{Sample Result})}{(\text{Amount Spiked})} \times 100 = \text{Matrix Spike Recovery}$$

MATRIX SPIKE DUPLICATES: Intralaboratory split samples spiked with identical concentrations of target analyte(s). The spiking occurs prior to sample preparation and analysis. They are used to document the precision and bias of a method in a given sample matrix.

METHOD BLANK. The laboratory must digest and analyze a method blank with each set of samples. A minimum of one method blank is required for every 20 samples. For a running batch, a new method blank is required for each different digestion day. If no digestion step is required, then the method blank is equivalent to the reagent blank. The method blank must contain the parameter of interest at levels of less that the reporting limit for that parameter. If the method blank contains levels over the reporting limits, the samples must be redigested or redistilled and reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the method blank level. In addition, if all the samples are less than a client required limit and the method blank is also less than that limit, then the results can be reported as less than that limit.

METHOD DETECTION LIMITS (MDLS). MDLs should be established for all appropriate methods, using a solution spiked at approximately 3 times the estimated detection limit. To determine the MDL values, take seven replicate aliquots of the spiked sample and process through the entire analytical method. The MDL is calculated by multiplying the standard deviation of three replicate analyses by 3.14, which is the student's t value for a 99% confidence level. MDLs should be determined approximately once per year for frequently analyzed parameters.

REAGENT BLANK: The reagent blank is a blank that has the same matrix as the samples, i.e., all added reagents, but did not go through sample preparation procedures. The reagent blank is an indicator for contamination introduced during the analytical procedure. (Note: for methods requiring no preparation step, the reagent blank is equivalent to the method blank.) Either a reagent blank or a method blank must be analyzed with each batch of 20 samples or less. The concentration of the analyte of interest in the reagent blank must be less than the reporting limit for that analyte. If the reagent blank contains levels over the reporting limits, the samples must be reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the reagent blank level. In addition, if all the samples are less than a client required limit and the reagent blank is also less than that limit, then the results can be reported as less than that limit.

REAGENT GRADE: Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents which conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.

REAGENT WATER: Water that has been generated by any method which would achieve the performance specifications for ASTM Type II water. For organic analyses, see the definition of organic-free reagent water.

REFERENCE MATERIAL: A material containing known quantities of target analytes in solution or in a homogeneous matrix. It is used to document the bias of the analytical process.

STANDARD CURVE: A plot of concentrations of known analyte standards versus the instrument response to the analyte. Calibration standards are prepared by successively diluting a standard solution to produce working standards which cover the working range of the instrument. Standards should be prepared at the frequency specified in the appropriate section. The calibration standards should be prepared using the same type of acid or solvent and at the same concentration as will result in the samples following sample preparation. This is applicable to organic and inorganic chemical analyses.

5.0 HEALTH & SAFETY

5.1 The analyst should follow normal safety procedures as outlined in the Accutest Laboratory Safety Manual which includes the use of safety glasses and lab coats. In addition, all acids are corrosive and should be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these reagents should be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets should be made available to all personnel involved in these analyses.

6.0 PRESERVATION & HOLDING TIME

6.1 Both soils and water samples should be kept under refrigeration at 4^0 C until analysis. Water samples should be preserved with sulfuric acid to a pH of less than 2 if they are to be analyzed for nitrate + nitrite.

6.2 If nitrate only is requested, then the sample can be separated into 2 aliquots. The nitrate + nitrate aliquot should be preserved with sulfuric acid and the nitrite aliquot should be unpreserved.

6.3 All preserved samples should be analyzed within 28 days of the date of collection. Unpreserved samples must be analyzed within 48 hours of the time of collection.

6.4 Nitrite must be analyzed within 48 hours.

7.0 INTERFERENCES

7.1 Build up of suspended matter in the reduction column will restrict sample flow. However samples can be filtered to avoid this interference.

7.2 High concentrations of iron, copper, or other heavy metals can cause low results, but this can be avoided by adding EDTA to the samples to complex the metals.

7.3 Residual chlorine can produce a negative interference. This can be eliminated by dechlorinating the sample with sodium thiosulfate.

7.4 Samples that contain large concentrations of oil and grease will coat the surface of the cadmium and decrease column efficiency. This interference can be eliminated by pre-extracting the sample with an organic solvent such as hexane to remove the oil and grease.

8.0 APPARATUS

8.1 Automated continuous flow analyzer designed to deliver and react sample and reagents in the required order and ratios. Currently, two Lachat QuikChem 8000 Automated Ion Analyzers are being used.

8.1.1 Autosampler

8.1.2 Multichannel pump

8.1.3 Reaction manifold.

8.1.4 Colorimetric detector

8.1.5 Real time data acquisition device (either electronic or hard copies.)

8.2 Balance. Analytical balance capable of accurately weighing to the nearest 0.0001 g.

8.3 Volumetric glassware. Class A volumetric pipets and flasks as required. All glassware should be washed with soap and tap water and then well rinsed with distilled, deionized water

9.0 REAGENTS

All chemicals listed below are reagent grade unless otherwise specified. Distilled, deionized water should be used whenever water is required.

9.1 15 N Sodium Hydroxide. In a large container, slowly add 150 g of NaOH to 250 ml of DI water. Swirl until dissolved. Caution - the solution will get very hot

9.2 Ammonium Chloride Buffer Solution. Caution - Prepare this in a hood! To a 1 liter volumetric flask, add 500 ml of DI water, 105 ml of concentrated hydrochloric acid, 95 ml of ammonium hydroxide (NH_4OH) and 1.0 g of disodium ethylene-diamine tetraacetic acid dihydrate ($Na_2EDTA \cdot 2H_2O$). Mix well. Adjust the final pH to 9.1 for preserved samples or to 8.5 for non-preserved samples using either additional ammonium hydroxide or sodium hydroxide Note: This

preparaion is recommended by LACHAT since nitrate contamination can be a problem in reagent grade ammonium chloride.

9.3 Sulfanilamide color reagent. Add 100 ml of concentrated phosphoric acid (H_3PO_4), 40.0 g of sulfanilamide, and 1.0 g of N-(1-napthyl)ethylenediamine dihydrochloride (NED) to a 1 liter volumetric containing approximately 600 mL of DI water. Stir to dissolve and dilute to a final volume of 1 liter with DI water. Store in a dark bottle. This solution is stable for approximately 1 month.

9.4 Hydrochloric Acid, 1 N. Add 8 ml of concentrated HCl to approximately 85 mL of DI water. Dilute to a final volume of 100 mL with DI water.

9.5 Carrier Solution. Acidify DI water to a pH of < 2 with sulfuric acid.

9.6 Two percent copper sulfate solution. Dissolve 20 g of copper sulfate (CuSO4.5H2O) in approximately 800 ml of DI water in a 1 liter volumetric flask. Dilute to a final volume of 1 liter and mix well.

9.7 Cadmium Reduction Column. Cadmium columns are normally purchased. After a new column is received, it is recommended that it be conditioned by running a 1 mg/l standard through the column for 10 minutes and then washing the column with reagents for an additional 20 minutes before use.

9.7.1 A back-up procedure is also provided below for packing cadmium columns in house. This is not normally necessary, but it included as a back-up procedure only.

9.7.1.1 Place 10 to 20 g of coarse cadmium granules in a beaker. Cadmium granules ranging from 0.3 to 1.5 mm in diameter are recommended. Wash the Cd first with acetone and then with DI water. Then wash the Cd with two 50 mL portions of 1 N HCl. Rinse the Cd granules well with DI water.

9.7.1.1.1 Cadmium is toxic. Make sure to wear gloves for all procedures and collect any cadmium waste in a marked solid waste container.

9.7.1.2 Add 100 mL of 2% copper sulfate solution to the cadmium granules. Swirl for about 5 minutes, then decant the liquid and repeat the process with a fresh portion of 2% copper sulfate solution. Continue this process until the blue color of the copper sulfate solution persists.

9.7.1.3 Decant off the copper sulfate solution and rinse the copper granules with at least 5 portions of ammonium chloride buffer to remove the colloidal copper. The cadmium should be black or dark gray at this point and can be stored in a capped bottle under ammonium chloride buffer solution.

9.7.1.4 Open one end of a column, removing the colored lead and the foam plug, and clamp the column so that the open end is pointing up. Fill the column with ammonium chloride buffer. Pour the prepared cadmium granules into the column and tap the column lightly so that the granules settle to the bottom of the column. Fill the column to about 5 mm from the open end. Make sure that there are no air bubbles. Push in the foam plug and screw on the cap. Rinse the outside of the column with DI water.

9.7.1.5 Insert the column into the manifold by first pumping all reagents into the manifold. Then turn the pump off. Connect the column to the appropriate tubing, making sure that no air is in the tubing. Then return the pump to its normal speed.

9.8 Stock nitrate solution, 1000 mg/l. Dissolve 7.218 g of KNO_3 and dilute to 1 liter with DI water in a 1 liter volumetric flask. Preserve with 2 ml of chloroform per liter. This solution is stable for 6 months. Note: The chloroform must be visible in this stock solution. If no chloroform is visible, a new stock solution should be made.

9.9 Standard nitrate solution, 100 mg/l. Dilute 10.0 ml of 1000 mg/l nitrate solution to 100 ml with DI water in a volumetric flask.

9.10 Standard nitrate solution, 10.0 mg/l. Dilute 10.0 ml of 1000 mg/l nitrate solution to 1000 ml with DI water in a volumetric flask.

9.11 Nitrate Calibration Standards. Calibration Standards should be made from the above standard solutions. Suggested levels and preparations are shown below. Other levels may be used as appropriate. Class A volumetric pipets and flasks should be used for all dilutions. All standards should be brought to a final volume of 100 ml with carrier solution. A calibration check standard should also be prepared that is at or near the mid-point of the calibration curve.

ml of 100 mg/l Standard	ml of 10.00 mg/l Standard	Final Conc in mg/l
NA	0.00	0.00
NA	1.00	0.10
NA	2.00	0.20
NA	5.00	0.50
NA	10.00	1.00
2.50	NA	2.50
5.00	NA	5.00

9.12 Stock Nitrite solution, 1000 mg/l. Dissolve 6.072 g of KNO_2 in 500 ml of DI water and dilute to 1 liter in a 1 liter volumetric flask. Preserve with 2 ml of chloroform and keep under refrigeration. Refer to the nitrite analysis SOP, EGN103, for information on standardization of this stock solution.

9.13 Nitrite Efficiency Spike Solutions, 100 mg/l. Dilute 10 ml (adjust volume for standardized concentration of the nitrite stock) of 1000 mg/l nitrite standard to 100 ml final volume. Note: The nitrite solution is used to check the efficiency of the cadmium reduction column, by comparing a nitrite standard directly with a nitrate standard of the same concentration. It is recommended that the efficiency check standards be prepared by adding 1.0 ml of 100 mg/l nitrite stock to a final volume of 50.0 ml with DI water.

10.0 PROCEDURE

Below is a step-by-step procedure for the analysis of samples for the determination of nitrate/nitrite and nitrate only. At the end of this SOP is a short summary outlining the overall procedure.

10.1 Install the nitrate reaction manifold, excluding the cadmium column. Check all tubing and change any tubing that is flat, dirty, etc. Install the appropriate sample loop and the appropriate filter. Place the tubing in the bottles for the sulfanilamide color reagent, the ammonia buffer solution, and the acidified DI water carrier. Also make sure that the waste container is in place. Refer to the Lachat methods manual for additional information.

10.2 For soil samples, follow the digestion procedure described below. The matrix spike should be prepared by adding 2.0 ml of 100 mg/l nitrate solution directly to the soil and mixing well before the acid digestion. The spike blank should also be prepared by digesting 2.00 ml of the 100 mg/l standard. Make sure to prepare a method blank with each batch of samples.

10.2.1 Weigh out 1.0 g of sample (dry weight) into a 200 ml erlenmeyer flask. Add 50 ml of DI water and 4 drops of concentrated sulfuric acid. Add another 50 ml of DI water and then boil the sample on a hot plate for 15 minutes. NOTE: This procedure is operationally defined, so make sure that the same heating time is used for all samples.

10.2.2 Transfer the sample to a centrifuge tube and centrifuge for 5 to 10 minutes. Decant the wash into a 200 ml volumetric flask.

10.2.3 Add 50 ml of DI water to the solids in the centrifuge tube and mix well. Then centrifuge the sample for 5 to 10 minutes and decant the wash into the volumetric flask.

10.2.4 Repeat the above step a second time and again decant the wash into the volumetric flask.

10.2.5 Bring the sample to a final volume of 200 ml with DI water. Filter the sample through a 0.45 um filter before analysis.

10.3 For water samples no preparation is necessary unless the sample is turbid. If the sample is turbid, filter before analysis. The matrix spike should be prepared by adding 0.50 ml of 100 mg/l nitrate solution to 50 ml of sample. The spike blank should be prepared by diluting 1.0 ml of 100 mg/l nitrate solution to a final volume of 50.0 ml with DI water. Make sure to analyze a method blank for each batch of samples.

10.3.1 If a sample is filtered, then the associated method blank and spike blank must also be fitlered.

10.4 Check the pH of each sample using wide range pH paper and adjust the pH to between 5 and 9 using either concentrated HCl or ammonium hydroxide as necessary. (Ammonium hydoxide is used to adjust preserved samples to the range of pH 5 to 9). Document that the pH is in the correct range on the instrument autosampler table.

10.5 Start pumping reagents through the system. When reagents are pumped through the manifold, then the cadmium column can be installed. Note: Be careful not to get any air into the column!

10.6 Use the following commands for running the older QuikChem software.

10.6.1 Select the "Flow Injection Analysis" in the Omnion FLA Data System. Go into "File" and click on "Open Method". Select the appropriate method. Note: If an appropriate method is not available, then contact the supervisor or the manager and refer to the Lachat manuals for help in creating a new method.

10.6.2 Once you are into the method select the "New Tray" option and click on "Gil240rck" for the type of rack you will use (sampler Gilson 240-position) and then click on "default dqm". Define your new tray, type in the calibration standards and samples identification number. Also type after the calibration ICV, ICB, CCV, CCB and CCV, CCB pairs every 10 samples. Specify the number of replicates and levels. The levels for the calibration standards should match what you already specified in the "Analyte Table". The level for samples is always zero. Save the new tray by clicking on File, "Save Tray As". Specify a tray description. Click OK to continue. To open an already existing tray, click on "Tray", select the tray you want and click OK.

10.6.3 Load your standards and the samples in the autosampler. Before you start the analysis, click on "Methods" and select "calibration clear" to clear the previous calibration.

10.6.4 To start the analysis click on "Run Tray". Leave the "Method" and "Tray" blank to use the ones currently loaded. You must specify the data file name. The autosampler position indicates which sample rack will be run. The "Skip Recalibration" block can be checked when you want to run a tray that contains calibration standards and samples and you want only the samples to be run.

10.6.5 While the tray is running, you can view the peaks and the run time report. If the baseline does not appear on the screen, go into "Method", click on "Display options" and change the voltage scale.

10.6.6 While the tray is running, you will see a stop sign on the tool bar. To suspend the tray click on the stop sign. The tray will give you the choices of aborting or resuming the tray.

10.6.7 The analysis will be completed when the stop sign turns back into "Run Tray". View the calibration curve by clicking on "Review". If the calibration meets the method criteria, it will automatically be approved by the system. If the calibration does not meet the criteria, the instrument must be recalibrated. To go back to the main menu click "exit".

10.6.8 When the run is complete, prepare a customized report by clicking on "Custom".

10.6.9 At the end of the run, rinse out the remainder of the system with DI water.

10.7 Use the following commands for running the newer QuikChem software.

10.7.1 Select the "NO3" icon from Windows. Click on Window and Tile to open. The run worksheet, run properties, and channel NO3 windows.

10.7.2 Type in the calibration standards and the EFFCHK, ICV, ICB. Also type in the CCV/CCB pairs to run at the beginning, at the end, and after every 10 samples.

10.7.3 To insert sample lines, click on the gray box to the left of the sample line. Right click on the box and choose "append many" and enter the corresponding number of sample lines. Then type in the sample identification numbers. Next change the cup numbers to correspond with the correct position on the autosampler tray.

10.7.4 Load the autosampler tray and the click on the start icon to start the tray running.

10.7.5 After the calibration standards have finished running, click on the last icon in the channel 1 window to view the curve. If the calibration does not meet the criteria, then the instrument must be recalibrated. If the calibration meets criteria, then proceed with the samples and continuing quality control.

10.7.5.1 While the tray is running, you will see a stop sign on the tool bar. To suspend the tray, click on the stop sign and select the appropriate option.

10.7.6 When the run is complete when the stop sign goes away. Prepare a customized report by clicking on "tools" and choosing the custom report option. Click on "report" and choose "open format" and then choose "Accutest".

10.7.7 At the end of the run, rinse out the remainder of the system with DI water.

10.8 Calculations may be either done manually, using the Lachat software, or using the Accutest LIMS system. Check with the lab supervisor or manager for more instructions.

10.8.1 For water samples, the following calculations should be used.

original sample conc. of nitrate in mg/l = Calculated value in mg/l * dilution factor

10.8.2 For soil samples, the following calculations should be used

$$\text{original sample conc. of nitrate in mg/kg} = \frac{(\text{Calculated value in mg/l}) * (\text{Final volume in l})}{(\text{initial sample w in kg}) \times (\%\ \text{solids}/100)}$$

10.9 Review data and assemble a data package including the following items

- Results report, showing dilution and weight correction factors.
- Printout of peaks.
- Autosampler tray numbering sheet and sample bottle numbers with pH checks.
- Distillation log (if needed)
- Reagent sheet
- Standards preparation sheets
- Computer workgroup sheets.

11.0 QUALITY CONTROL

Below is a summary of the quality control requirements for this method. Make sure to check with the laboratory supervisor or manager for any additional client specific quality control requirements.

11.1 Method Detection Limits (MDLs). MDLs should be established using a solution spiked at approximately 3 times the estimated detection limit. To determine the MDL values, take seven replicate aliquots of the spiked sample and process through the entire analytical method. The MDL is calculated by multiplying the standard deviation of the replicate analyses by 3.14, which is the student's t value for a 99% confidence level. MDLs should be determined approximately once per year or whenever there is a significant change in the background or instrument response. For drinking water samples, MDL studies should be performed every 6 months.

11.2 Linear Dynamic Range (LDR). For each instrument, the upper limit of the linear dynamic range must be established. A linear calibration should be prepared from 3 standards, one of which is close to the upper limit of the linear range. The LDR is determined by analyzing succeedingly higher standard concentrations of an analyte until the observed analyte concentration is no more than 10 percent below the true value of the standard. The linear range should be verified at a frequency of every 6 month or whenever instrument conditions change subtantially.

11.3 Quality Control Sample (also referred to as Initial Calibration Verification Standard (ICV)). At a minimum of once per quarter, a standard from a different source than the calibration standard must be analyzed. Normally this is analyzed at the beginning of the run before the CCV and CCB checks. For this method, the ICV should be within 10 percent of the true value. Note: It is recommended that this standard be analyzed with each run.

11.4 Method Blank. The laboratory must digest and analyze a method blank with each set of samples. A minimum of one method blank is required for every 20 samples. For a running batch, a new method blank is required for each different digestion day. The method blank must contain the analyte at less that the reporting limit. If the method blank contains over that limit, the samples must be redigested or reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the method blank level. In addition, if all the samples are less than a client required limit and the method blank is also less than that limit, then the results can be reported as less than that limit.

11.4.1 For drinking waters, the method states that values that exceed the MDL should be suspect and corrective actions must be taken before continuing the analysis. However, in practice, the MDL is 50 times lower than the RDL and values slightly over the MDL do not affect data quality. Method blanks with results of less than half of the RL will be generally be considered acceptable for this method.

11.5 Spike Blank. The laboratory must digest and analyze a spike blank with each set of samples. A minimum of one spike blank is required for every 20 samples. For a running batch, a new spike blank is required for each different digestion/analysis day. The laboratory should assess laboratory performance of the spike blank for non-potable samples against recovery limits of 80 to 120 percent. For drinking water samples, the spike blank should be in the limits of 90 to 110 percent. If the spike blank recovery is high and the results of the samples to be reported are less than the reporting limit, then the sample results can be reported with no flag.

11.6 Matrix Spike. The laboratory must add a known amount of each analyte to a minimum of 1 in 20 samples for non-potable samples and to 1 in 10 samples for potable samples.

11.6.1 For non-potable samples, the spike recovery should be assessed using in house limits. Until these limits can be generated, then default limits of 75 to 125 percent recovery should be applied. If a matrix spike is out of control, then the results should be flagged with the appropriate footnote. If the matrix spike amount is less than one fourth of the sample amount, then the sample cannot be assessed against the control limits and should be footnoted to that effect.

11.6.2 For drinking water samples, the spike recovery should be assessed against limits of 90 to 110 percent. If a matrix spike is out of control, then the results should be flagged with the appropriate footnote. If the matrix spike amount is less than one

fourth of the sample amount, then the sample cannot be assessed against the control limits and should be footnoted to that effect.

11.6.3 Both the matrix spike recovery should be calculated as shown below.

$$\frac{\text{(Spiked Sample Result - Sample Result)}}{\text{(Amount Spiked)}} \times 100 = \text{MS Recovery}$$

11.7 Matrix Duplicate. The laboratory must digest a duplicate sample for a minimum of 1 in 20 samples. The relative percent difference (rpd) between the duplicate and the sample should be assessed. The duplicate rpd is calculated as shown below.

11.7.1 The duplicate RPD should be assessed using in house limits. Until these limits can be generated, then default limits of 20 percent RPD should be applied If a duplicate is out of control, then the results should be flagged with the appropriate footnote. If the sample and the duplicate are less than 5 times the reporting limits and are within a range of ± the reporting limit, then the duplicate is considered to be in control.

11.7.2 The duplicate RPD should be calculated as shown below.

$$\frac{\text{(Sample Result - Duplicate Result)} \times 100}{\text{(Sample Result + Duplicate Result)} \times 0.5} = \%\ \text{RPD}$$

11.8 Continuing Calibration Verification. (Also known as the instrument performance check solution.) Analyze the continuing calibration verification solution and the continuing calibration blank after the initial calibration, after every tenth sample, and at the end of the sample run. If the CCV solution is not within 10 percent of the true value, then no samples can be reported in the area bracketed by that CCV. (Note: the exception is if the CCV is biased high and the samples are less than the detection limit. In that case, the samples can be reported with no flag.) The CCV concentration should be at or near the mid-range of the calibration curve.

11.9 Continuing Calibration Blank. Analyze the continuing calibration verification solution and the continuing calibration blank after the initial calibration, after every tenth sample, and at the end of the sample run. If the CCB is not less than the reporting limit, then no samples can be reported in the area bracketed by the failing CCB.

11.10 Calibration Curve. The calibration curve must be analyzed on each analysis day and must consist of a minimum of 5 points and a blank. One standard must be at or below the reporting limit. The calibration must have a correlation coefficient of at least 0.995 and an intercept of less than the reporting limit. If these criteria are not met, then the instrument must be recalibrated.

11.11 Efficiency check standard. At the beginning of each run, or at least once per day, an efficiency check standard made up of nitrite must be analyzed at the same level of a blank spike run using a nitrate standard. The recovery of the nitrite standard in comparison to the nitrate standard shows if the cadmium column is reducing the nitrate to nitrite effectively. A recovery range of 90 to 110% is required for the efficiency check standard. (See calculation below.) For Pennsylvania drinking water samples the recovery should be in the range of 96 to 104%. If the efficiency check is not within this range, then the column must be regenerated or replaced and rechecked before any samples are analyzed.

100 x (Nitrate blank spike result)/(Nitrite Efficiency check result) = Column Efficiency

12.0 DOCUMENTATION REQUIREMENTS

12.1 For every run, the analyst should review all data and assemble a data package consisting of the following information.

- Results report, showing dilution and weight correction factors.
- Quality control sheets.
- Printout of peaks.
- Distillation or digestion log.
- Autosampler tray positions, bottle numbers, and adjusted pH values.
- Computer workgroup sheets.

12.2 Any unusual characteristics of the samples should be noted in the final results section of the final data report. Make sure that all sample ID's and dilutions are labeled on the data.

12.3 In addition, all reagent information such as lot numbers should also be recorded in the reagent log book.

13.0 DATA REVIEW AND REPORTING

13.1 The analyst is responsible for a 100% review of the run. Things that should be checked include the chromatography (checking for air bubbles, unstable baseline, etc.), the results (over calibration range) quality control recoveries, and documentation (reagent lots, etc.)

13.2 The analyst is responsible for updating the run and transferring the data into the LIMS system. They are also responsible for adding spike amounts in the LIMS and adding comments. If a sample was analyzed multiple times, they must designate the analysis to be reported.

13.3 The general chemistry supervisor or designate then does a 100% review of the data before it is approved for client use. The inorganics manager does an additional review on a minimum of 10% of the data.

14.0 POLLUTION PREVENTION & WASTE MANAGEMENT

14.1 Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 14.2.

14.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the waste management SOP, ESM003-02. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

14.2.1 Non hazardous aqueous wastes.

14.2.2 Hazardous aqueous wastes

14.2.3 Chlorinated organic solvents

14.2.4 Non-chlorinated organic solvents

14.2.5 Hazardous solid wastes

14.2.6 Non-hazardous solid wastes

15.0 REFERENCES

15.1 Lachat Quickchem Instrument and Software manuals.

ACCUTEST LABORATORIES
STANDARD OPERATING PROCEDURE

Lab. Manager:_____

QA Manager:_____

Effective Date: _____

TITLE: FERROUS IRON

REFERENCES: "Standard Methods for the Examination of Water and Wastewater", 20th Edition, Method3500-Fe D.

Revision Sections: Reference revision, 3.1

1.0 SCOPE AND APPLICATION

1.1 This method is used as a measure of ferrous iron in aqueous samples and is applicable to all waters and wastewaters.

1.2 This method can also be used to indirectly determine the approximate concentration of ferric iron. An aliquot of the sample is analyzed for total iron in addition to the ferrous iron analysis. Ferric iron is calculated as the difference between the ferrous and the total iron.

2.0 SUMMARY

2.1 An acidified measured aliquot of the sample is combined with phenanthroline and ammonium acetate solution. The ferrous iron reacts to form a pink color and the absorbance of the solution is read at 510 nm. The sample is quantitated against a calibration curve.

3.0 REPORTING LIMIT AND METHOD DETECTION LIMIT

3.1 Reporting Limit. The reporting limit for this method is established at 0.2 mg/l.

3.2 Method Detection Limit. Experimentally determine MDLs using the procedure specified in 40 CFR, Part 136, Appendix B. This value represents the lowest reportable concentration of an individual compound that meets the method qualitative identification criteria.

3.2.1 Experimental MDLs must be determined annually for this method.

3.2.2 Process all raw data for the replicate analysis in each MDL study.

4.0 DEFINITIONS

BATCH: A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

CALIBRATION CHECK STANDARD. The calibration check standard is a mid-range calibration standard. It is recommended that the calibration check standard be run at a frequency of

approximately 10 percent. (For some methods this is mandatory and for some it is a recommendation only. Refer to individual method SOP's) For most methods, the mid-level calibration check standard criteria is ± 10 percent of the true value. The exception to this rule is if the recovery on the calibration check standard is high and the samples to be reported are less than the detection limit.

EXTERNAL CHECK STANDARD. The external check standard is a standard from a separate source than the calibration curve that is used to verify the accuracy of the calibration standards. An external check must be run a minimum of once per quarter for all analyses where a check is commercially available. The laboratory should initially assess laboratory performance of a check standard using the control limits generated by the external check supplier. In house limits should also be generated once sufficient external check standard data is available to generate limits (usually a minimum of 20 to 30 analyses). If the external check is outside of the control limits for a given parameter, all samples must be reanalyzed for that parameter after the problem has been resolved.

SPIKE BLANK OR LAB CONTROL SAMPLE. Digest and analyze a laboratory control sample or spike blank with each set of samples. A minimum of one lab control sample or spike blank is required for every 20 samples. Assess laboratory performance against the control limits specified in the SOP. In house limits should also be generated once sufficient external check standard data is available to generate limits (usually a minimum of 20 to 30 analyses). If the lab control is outside of the control limits for a parameter, all samples must be redigested or redistilled and reanalyzed for that parameter. The exception is if the lab control recovery is high and the results of the samples to be reported are less than the reporting limit. In that case, the sample results can be reported with no flag. Note: If control limits are not specified in the SOP, then default limits of 80 to 120 percent should be used.

MATRIX: The component or substrate (e.g., water, soil) which contains the analyte of interest.

MATRIX DUPLICATE: A duplicate sample is digested at a minimum of 1 in 20 samples. The relative percent difference (RPD) between the duplicate and the sample should be assessed. The duplicate RPD is calculated as shown below. Assess laboratory performance against the control limits that are specified in the SOP. In house limits are generated once sufficient duplicate data is available to generate limits (usually a minimum of 20 to 30 analyses). If a duplicate is out of control, flag the results with the appropriate footnote. If the sample and the duplicate are less than 5 times the reporting limits and are within a range of ± the reporting limit, then the duplicate is considered to be in control. Note: If control limits are not specified in the SOP, use default limits of ± 20% RPD.

$$\frac{(|\text{Sample Result} - \text{Duplicate Result}|) \times 100}{(\text{Sample Result} + \text{Duplicate Result})/2} = \text{Duplicate RPD}$$

MATRIX SPIKE: The laboratory must add a known amount of each analyte to a minimum of 1 in 20 samples. The matrix spike recovery is calculated as shown below. Assess laboratory performance against the control limits that are specified in the SOP. In house limits are generated once sufficient matrix spike data is available to generate limits (usually a minimum of 20 to 30 analyses). If a matrix spike is out of control, then the results should be flagged with the appropriate footnote. If the matrix spike amount is less than one fourth of the sample amount, then the sample cannot be assessed against the control limits and should be footnoted to that effect. Note: If control limits are not specified in the SOP, then default limits of 75 to 125 percent should be used.

$$\frac{(\text{Spiked Sample Result} - \text{Sample Result})}{(\text{Amount Spiked})} \times 100 = \text{Matrix Spike Recovery}$$

METHOD BLANK. The laboratory must digest and analyze a method blank with each set of samples. A minimum of one method blank is required for every 20 samples. For a running batch, a new method blank is required for each different digestion day. If no digestion step is required, then the method blank is equivalent to the reagent blank. The method blank must contain the parameter of interest at levels of less that the reporting limit for that parameter. If the method blank contains levels over the reporting limits, the samples must be redigested or redistilled and reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the method blank level. In addition, if all the samples are less than a client required limit and the method blank is also less than that limit, then the results can be reported as less than that limit.

METHOD DETECTION LIMITS (MDLS). MDLs should be established for all appropriate methods, using a solution spiked at approximately 3 times the estimated detection limit. To determine the MDL values, take seven replicate aliquots of the spiked sample and process through the entire analytical method. The MDL is calculated by multiplying the standard deviation of three replicate analyses by 3.14, which is the student's t value for a 99% confidence level. MDLs should be determined approximately once per year for frequently analyzed parameters.

REAGENT BLANK: The reagent blank is a blank that has the same matrix as the samples, i.e., all added reagents, but did not go through sample preparation procedures. The reagent blank is an indicator for contamination introduced during the analytical procedure. (Note: for methods requiring no preparation step, the reagent blank is equivalent to the method blank.) Either a reagent blank or a method blank must be analyzed with each batch of 20 samples or less. The concentration of the analyte of interest in the reagent blank must be less than the reporting limit for that analyte. If the reagent blank contains levels over the reporting limits, the samples must be reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the reagent blank level. In addition, if all the samples are less than a client required limit and the reagent blank is also less than that limit, then the results can be reported as less than that limit.

REAGENT GRADE: Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents which conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.

REAGENT WATER: Water that has been generated by any method which would achieve the performance specifications for ASTM Type II water. For organic analyses, see the definition of organic-free reagent water.

REFERENCE MATERIAL: A material containing known quantities of target analytes in solution or in a homogeneous matrix. It is used to document the bias of the analytical process.

STANDARD CURVE: A plot of concentrations of known analyte standards versus the instrument response to the analyte. Calibration standards are prepared by successively diluting a standard solution to produce working standards which cover the working range of the instrument. Standards should be prepared at the frequency specified in the appropriate section. The calibration standards should be prepared using the same type of acid or solvent and at the same concentration as will result in the samples following sample preparation. This is applicable to organic and inorganic chemical analyses.

5.0 HEALTH AND SAFETY

5.1 The analyst should follow normal safety procedures as outlined in the Accutest laboratory safety manual. Safety glasses and a lab coat should be worn whenever lab work is being done.

6.0 SAMPLE HANDLING AND PRESERVATION

6.1 Ferrous iron should ideally be determined at the time of sampling because of the possibility of change in the ferrous-ferric ratio with time in acid solutions. Samples received for analysis at the lab should be analyzed as soon as possible.

6.2 Samples should be acidified with 2 ml of concentrated HCl per every 100 ml of sample at the time of analysis.

7.0 APPARATUS AND MATERIALS

The following items are needed for the analysis of samples following the method outlined below:

7.1 50 and 100 ml graduated cylinders, class A.

7.2 Volumetric pipets, class A.

7.3 Spectrophotometer, capable of reading absorbances at 510 nm.

8.0 REAGENTS

All chemicals listed below are reagent grade unless otherwise specified. Deionized water should be used whenever water is required.

8.1 Hydrochloric acid, HCl, concentrated. Trace metals grade.

8.2 Hydroxylamine hydrochloride solution. Dissolve 10 g of hydroxylamine hydrochloride ($NH_2OH\ HCl$) in 100 ml of water and mix well.

8.3 Ammonium acetate buffer solution: Dissolve 250 g of ammonium acetate in 150 ml of water. Add 700 ml of concentrated (glacial) acetic acid. Mix well. Note: Ammonium acetate contains a significant amount of iron. Make sure to use the same lot of ammonium acetate buffer for all samples analyzed with a given calibration curve.

8.4 Sodium acetate solution: Dissolve 200 g of sodium acetate ($NaC2H3O2.3H2O$) in 800 ml of water and mix well.

8.5 Phenanthroline solution: Dissolve 100 mg of 1,10-phenanthroline monohydrate, $C12H8N2.H2O$, in 100 ml water by stirring and adding 2 drops of concentrated HCl.

8.6 Concentrated phenanthroline solution (for samples with high metals): Dissolve 500 mg of 1,10-phenanthroline monohydrate, C12H8N2.H2O, in 100 ml water by stirring and adding 2 drops of concentrated HCl.

8.7 Stock Iron Solution, 200 mg/l. Slowly add 20 ml of concentrated sulfuric acid to 50 ml of water and then dissolve 1.404 g of ferrous ammonium sulfate ($Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$). Add 0.1N potassium permanganate ($KMnO_4$) dropwise until a faint pink color persists. Dilute to a final volume of 1000 ml with water and mix well. This stock solution should be stable for 2 months.

8.8 Working Iron Standard Solution, 10.00 mg/l. Dilute 10.00 ml of 200 mg/l stock iron solution to a final volume of 200 ml in DI water and mix well. This stock solution should be made fresh daily.

9.0 INTERFERENCES

9.1 If the sample is not immediately analyzed, there is the possibility of change in the ferrous-ferric iron ratio over time.

9.2 Interfering substances in this method include strong oxidizing agents, cyanide, nitrite, and phosphates, chromium, high levels of zinc (> 10 times Fe concentration), high levels of cobalt and copper (> 5 mg/l), and high levels of nickel (> 2 mg/L). Bismuth, cadmium, mercury, molybdenum, and silver precipitate phenanthroline. Interfering metals ions can complex the phenanthroline preferentially. Excess phenanthroline must be added when high metals are suspected.

9.3 High levels of color and organic matter may also interfere.

10.0 STANDARD PREPARATION AND ANALYSIS AND SAMPLE ANALYSIS

10.1 Iron contamination is very easy to pick up in the lab. Before starting this procedure, make sure that all glassware has been acid washed and triple rinsed with deionized water.

10.2 It is recommended that a new calibration curve be prepared and analyzed at least once per month. It is required that this be done at least once per quarter. If a full curve is not being run on a given day, then the curve must be verified with a freshly made blank, low standard, and high standard. To prepare the curve or the check standards, make the following dilutions of the 10.0 ppm or 200 ppm iron stocks as appropriate and bring to a final volume of 100 ml in deionized water. Mix well.

ml of 10.0 ppm Iron standard	ml of 200 ppm iron stock	Final volume	Final concentration
0.00 ml	0.000 ml	100 ml	0.000 mg/l
1.00 ml	0.000 ml	100 ml	0.100 mg/l
2.00 ml	0.000 ml	100 ml	0.200 mg/l
5.00 ml	0.000 ml	100 ml	0.500 mg/l
10.0 ml	0.000 ml	100 ml	1.00 mg/l
0.000 ml	1.00 ml	100 ml	2.00 mg/l
0.000 ml	2.00 ml	100 ml	4.00 mg/l
0.000 ml	3.00 ml	100 ml	6.00 mg/l

10.3 Measure 50.0 ml of each standard into a labeled 100 ml volumetric flask .Add 2 ml of concentrated HCL and 1 ml of hydroxylamine hydrochloride solution.

10.3.1 These standards may be digested as outlined in method 3500 Fe-D, but as long as the procedure in this SOP is followed exactly, digestion is not required. A comparative study showed that the procedure as written produces equivalent absorbances on digested and undigested standards.

10.4 At this point, make sure that the spectrophotometer is turned on so that it can warm up at least 30 minutes before use.

10.5 Transfer 50 ml of each standard to a 100 ml volumetric flask or nessler tube. Add 4.0 ml of phenanthroline solution and 10 ml of ammonium acetate solution with vigorous stirring. Dilute to 100 ml and mix well.

10.5.1 When preparing standards, make sure to prepare a large amount of buffer solution, so that the same lot of buffer can be use for the samples and the standards.

10.6 Zero the spectrophotometer following the manufacturers' instructions. Read the absorbance of the standards at 510 nm after 10 to 15 minutes for complete color development. Record the absorbance for each standard.

10.7 Calculate the correlation coefficient, slope, and intercept of the curve before starting the samples. The correlation coefficient must be at least 0.995 and the intercept must be less than the reporting limit. If there is a problem with the curve, do not proceed with the samples until the problem is resolved.

10.7.1 A new curve is not required for each analysis, but should be done a minimum of once per quarter. It is recommended that a new curve be analyzed at least once per month.

10.7.2 If a new curve is not needed, then the old curve must be verified by using a blank, a low, and a high standard. The readings of the standards should be within 10 percent of the true values or a new curve is required.

10.8 Measure out a 50 ml aliquot of acidified sample using a class A graduated cylinder. Add 20 ml of phenanthroline solution and 10 ml of ammonium acetate solution with vigorous stirring. Dilute to 100 ml and read the absorbance of the samples at 510 nm within 5 to 10 minutes. Record the absorbance for each sample. Do not expose the sample to sunlight.

10.8.1 If the sample absorbance is greater than the highest standard, then the sample should be diluted and the dilution should be taken through step 10.8.

10.8.2 If the total Fe is suspected to be higher than 1 mg/l, then a larger amount (or a more concentrated solution) of phenanthroline should be added. Excess phenanthroline may also be needed for higher levels (2 to 5 ppm) of other elements including Cr, Zn, Cu, Co, Ni, Bi, Cd, Hg, Mo, and Ag.

10.8.3 If a larger amount of phenanthroline is added to a sample, a blank and spike must also be prepared with the same amount of phenanthroline.

10.9 Calculate the result for each sample using the equation shown below.

Sample conc. in mg/l = Conc. from cal. curve in mg/l x dilution factor

11.0 QUALITY ASSURANCE

Below is a summary of the quality control requirements for this method. Make sure to check with the laboratory supervisor or manager for any additional client specific quality control requirements.

11.1 Method Detection Limits (MDLs). MDLs should be established using a blank sample spiked at approximately 3 times the estimated detection limit. To determine the MDL values, take seven replicate aliquots of the spiked sample and process through the entire analytical method. The MDL is calculated by multiplying the standard deviation of the replicate analyses by 3.14, which is the student's t value for a 99% confidence level. MDLs should be determined approximately once per year.

11.2 Method Blank. The laboratory must prepare and analyze a method blank with each set of samples. A minimum of one method blank is required for every 20 samples. For a running batch, a new method blank is required for each different analysis day. The method blank must contain the analyte at less that the reporting limit. If the method blank contains over that limit, the samples must reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the method blank level. In addition, if all the samples are less than a client required limit and the method blank is also less than that limit, then the results can be reported as less than that limit.

11.3 Matrix Duplicate. The laboratory should analyze a duplicate sample for a minimum of 1 in 20 samples. The relative percent difference (rpd) between the duplicate and the sample should be assessed. Matrix spike duplicates may be used in place of matrix duplicates. The duplicate rpd is calculated as shown below.

11.3.1 The duplicate RPD should be assessed using in house limits. Until these limits can be generated, then default limits of 20 percent RPD should be applied. If a duplicate is out of control, then the results should be flagged with the appropriate footnote. If the sample and the duplicate are less than 5 times the reporting limits and are within a range of ± the reporting limit, then the duplicate is considered to be in control.

11.3.2 The duplicate RPD should be calculated as shown below.

$$\frac{(\text{Sample Result} - \text{Duplicate Result}) \times 100}{(\text{Sample Result} + \text{Duplicate Result}) \times 0.5} = \%\ \text{RPD}$$

11.4 Quality Control Sample (also referred to as Initial Calibration Verification Standard, (ICV)). It is recommended that a standard from a separate source than the calibration should be run at the beginning of each run. (It is required that this be run a minimum of once per quarter.) This ICV should be within 10 percent of the true value. If it is not, the problem must be resolved before any samples can be analyzed.

11.5 Calibration Curve. The calibration curve must consist of a minimum of 5 standards and a blank. The correlation coefficient must be at least 0.995 and the intercept must be less than the

reporting limit. If there is a problem with the curve, do not proceed with the samples until the problem is resolved.

11.5.1 A new curve is not required for each analysis, but should be done a minimum of once per quarter. It is recommended that a new curve be analyzed at least once per month.

11.5.2 If a new curve is not needed, then the old curve must be verified by using a blank, a low, and a high standard. The readings of the standards should be within 10 percent of the true values or a new curve is required.

11.6 Analyze the continuing calibration verification solution after every tenth sample and at the end of the sample run. If the CCV solution is not within 10 percent of the true value, then no samples can be reported in the area bracketed by that CCV. (Note: the exception is if the CCV is biased high and the samples are less than the detection limit. In that case, the samples can be reported with no flag.) The CCV concentration should be at or near the mid-range of the calibration curve.

12.0 DOCUMENTATION REQUIREMENTS

12.1 A data sheet, showing any dilutions and all appropriate raw data, should be recorded for all samples. Make sure to record any comments regarding unusual sample appearance or any other problems or observations. All reagent reference numbers should be recorded on the data sheet or an attached reagent log.

12.2 All reagent information should be recorded in reagent logs.

12.3 Balance checks and calibrations should be recorded on the balance logs provided with each balance. Each balance must be checked at least once per day before use. The balance checks should be within the criteria listed in the balance log book.

13.0 DATA REVIEW AND REPORTING

13.1 All samples should be updated to analysis (GN) batches in the LIMS system. The analyst should calculate all quality control recoveries.

13.2 All documentation must be completed, including reagent references and spike amounts and spiking solution references.

13.3 A final lab data report should be written up consisting of the calculated raw data with reagent references and a quality control summary. Make sure to check that all samples are within the range of the calibration curve. This report should be turned in to the area supervisor for review.

13.4 All data must go through a 100% review by a senior data reviewer or supervisor. After

13.5 supervisor review and approval in the LIMS, the data is available to the client for use.

13.6 Approximately 10% of all data will then go through a further managerial review before being transferred to the report generation department for archiving.

14.0 POLLUTION PREVENTION & WASTE MANAGEMENT.

14.1 Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 14.2.

14.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the waste management SOP, ESM003. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

14.2.1 Non hazardous aqueous wastes.

14.2.2 Hazardous aqueous wastes.

14.2.3 Chlorinated organic solvents.

14.2.4 Non-chlorinated organic solvents.

14.2.5 Hazardous solid wastes.

14.2.6 Non-hazardous solid wastes.

15.0 ADDITIONAL REFERENCES

15.1 None required.

ACCUTEST LABORATORIES
STANDARD OPERATING PROCEDURE

Lab Manager:_____

QA Manager:_____

Effective Date:_____

TITLE: **Laboratory Analysis of Dissolved Gases in Aqueous Samples**

REFERENCES: USEPA RSK-175; SW846 8015

Revised Sections: ***3.0;5.0; 6.0; 7.0; 9.0; 10.0; 11.0; 12.0; 13.0.***

1.0 Scope & Application

1.1 The analysis of dissolved gases in aqueous samples, primarily groundwater is used to monitor natural attenuation. Natural attenuation is passive remediation of contaminated groundwater that employs natural processes to weather and dissipate contamination constituents. Monitoring specific dissolved gases in groundwater can provide an indication if contamination can be reduced by aerobic or anaerobic bioremediation. This activity is determined by monitoring dissolved methane, ethane, ethene and carbon dioxide.

2.0 Summary

2.1 This method is for the determination of trace levels of dissolved gases in aqueous samples, primarily groundwater. A sealed volatile organic vial with Teflon septa is prepared at ambient temperature by displacing a certain amount of the water with zero grade helium through the septa. The sample is shaken to equilibrate the dissolved gases between the headspace and the water sample. An aliquot of the headspace is injected into a calibrated gas chromatograph equipped with a flame ionization detector (FID) and thermal conductivity detector (TCD). The calibration is performed using the external standard technique employing a multipoint gas standard containing the compounds of interest at ppmv concentrations. The sample is quantitated based on the concentration of the targeted gases in the helium headspace in ppmv. The concentrations in the headspace are converted to the equivalent aqueous concentrations in the sample using a series of calculations that employ Henry's constant, sample temperature, sample vial volume, headspace volume, dilution factor, and the molecular weight of the gas. Results are reported in ug/l for each dissolved gas.

3.0 Method Detection Limits

3.1 Method Detection Limit Study. Experimentally determine MDLs using the procedure specified in 40 CFR, Part 136, Appendix B.

3.1.1 Experimental MDLs must be determined annually for this method.

3.1.2 Seven replicates of headspace gas are prepared from the commercial gas standard and analyzed. The concentrations of methane, ethane and ethene are 0.5 ppmv and is the approximate water equivalent of 0.10 ppb methane, 0.12 ppb ethane and 0.16 ppb ethene. The carbon dioxide concentration 50 ppmv and is the approximate water equivalent of 21 ppb.

3.1.3 The data for each replicate analysis is processed is processed and locally. The MDL study calculations are performed by the QA staff.

3.2 Reporting Limit. The reporting limit for this method is the low calibration standard. Detected analytes below the reporting limit concentrations cannot be reported without qualification.

3.1.1 The following reporting limits are based on the low calibration standard concentration.

Methane:	0.10	ug/l
Ethane:	0.12	ug/l
Ethene:	0.16	ug/l
CO2:	50.0	ug/l

4.0 Definitions

CALIBRATION FACTOR (CF) - a measure of the gas chromatographic response of a target analyte to the mass injected. The calibration factor is analogous to the Relative Response Factor (RRF) used in the Volatile and Semivolatile fractions.

CONTINUING CALIBRATION - analytical standard run every 12hours to verify the initial calibration of the system.

INITIAL CALIBRATION - analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of the photoionization detector/flame ionization detector for the target compounds.

MATRIX DUPLICATE - a second aliquot of the same matrix as the sample analyzed in order to determine the precision of the method.

METHOD BLANK - an analytical control consisting of all reagents, internal standards and surrogate standards (or SMCs for VOA), that is carried throughout the entire analytical procedure. The method blank is used to define the level of laboratory, background and reagent contamination.

PERCENT DIFFERENCE (%D) - As used in this SOP and elsewhere to compare two values, the percent difference indicates both the direction and the magnitude of the comparison, i.e., the percent difference may be either negative, positive, or zero. (In contrast, see relative percent difference.)

RELATIVE PERCENT DIFFERENCE (RPD) - As used in this SOP and elsewhere to compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero. (In contrast, see percent difference.)

5.0 Health & Safety

5.1 The analyst should follow normal safety procedures as outlined in the Accutest Laboratory Safety Manual which includes the use of safety glasses and lab coats. In addition, all acids are corrosive and should be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor

5.2 The following analytes covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: benzene, carbon tetrachloride, chloroform, and vinyl chloride. Primary standards of these toxic compounds should be prepared in a hood. A NIOSH/Mass approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

6.0 Collection, Preservation, & Holding Times

6.1 Samples are collected in a 40ml VO vial with Teflon septa in duplicate. Approximately 0.25ml of a 1:1 hydrochloric acid is used to preserve samples to pH<2 to fix the target gases unless Carbon Dioxide is to be determined. HCl can convert inorganic carbon to CO2. Accordingly, samples for carbon Dioxide determination are not preserved with acid.

6.2 Vials must be filled completely, eliminating headspace to prevent the loss of dissolved gases. Samples are shipped and stored at 4°C until analysis.

6.3 Preserved samples must be analyzed with 14 days from the time of collection. Unpreserved must be analyzed within 7 days from the time of collection.

7.0 Apparatus and Materials

7.1 Gas Chromatograph System

7.1.1 Gas Chromatograph - Hewlett Packard 5890 series II gas chromatograph with flame ionization detector (FID) and thermal conductivity detector (TCD) in series. Injection port is equipped with Electronic Pressure Control (EPC). Refer to Table 1 for recommended operating conditions.

7.1.2 Column – Supelco Carboxen 1006, 30m x 0.53mm or equivalent.

7.2 Data System

7.2.1 Hewlett Packard Enviroquant PC based chemstation software, capable of multi-point calibration technique and full instrument control.

7.2.2 Hewlett Packard "Custom Reports" using a calculation spreadsheet to convert the headspace ppmv concentrations into ug/l final results using Henry's Law constant, temperature of the sample, volume of sample vial, headspace volume, and the molecular weight of the gas.

7.3 "Burrell" Wrist Action Shaker.

7.4 Gas tight syringes

7.4.1 0.01cc, 0.05cc, 0.10cc, 0.50cc, and 1.0cc Hamilton gas tight syringe with style point #5 tip (side-port type) for sample and standard injections.

7.4.2 5cc syringe with luer lock tip and piercing needle to measure displaced sample out of vial.

7.5 Helium delivery system with adjustable regulator and piercing needle set at a flow of approximately 5cc/min for helium delivery to displace sample out of vial and create helium headspace.

7.6 40 ml VO vials with Teflon septa for sampling and method blank preparation.

7.7 NBS calibrated thermometer inserted through the septa of a VO vial containing deionized water for ambient temperature measurement.

7.8 50 ml Class A Graduated Cylinder.

7.9 Wide range pH paper.

7.10 Immersion thermometer (5-50°C)

8.0 Standards & Reagents

8.1 Commercially Available Gas Standards.

8.1.1 Spectra Gas Standard (or equivalent) containing 1000 ppmv of Methane, Ethane, Ethene, Carbon Monoxide (CO), and Carbon Dioxide (CO_2) in helium.

8.1.2 Scott Gas Calibration Standard (or equivalent) containing 24,000 ppmv of methane, 25,000 ppmv each of CO_2 and CO in helium.

8.1.3 Spectra Gas Standard (or equivalent) for use as a second source Laboratory Control Sample containing 1000ppmv each of Methane, Ethane, and Ethene in helium.

8.2 Gases

Chromatograph Carrier Gas:	Helium
Flame Ionization Detector Fuel:	Hydrogen
Flame Ionization Detector Make-Up:	Zero Air

8.3 De-ionized, ASTM Type II water

9.0 Interferences

9.1 Background levels of methane and CO2 can be present in the helium carrier gas. Background CO2 correction is performed by analyzing a helium blank daily and employing the CO2 peak area to background correct all samples.

9.2 Exposure of samples to the atmosphere should be avoided. Background concentrations of target compounds can contaminate samples. **Keep vials sealed at all times to avoid losses of light gases to the atmosphere.**

9.3 The Gas Chromatographic system may be baked out over night after batch analysis to eliminate water vapor buildup on the chromatographic column that can distort sample chromatography.

9.4 System cleanliness must be demonstrated through the analysis of a deionized water blank every 12 hours which is processed through all steps of the procedure. Background presence of target compounds exceeding the MDL must be eliminated before proceeding with sample analysis.

10.0 Procedure

10.1 **Instrument Operation** – Set the gas chromatograph to the operating conditions specified in table one. Prior to sample analysis, the instrument is calibrated and a negative contamination check is conducted.

10.2 **Calibration** – Calibration is performed by the external standard technique. The calibration range encompasses 0.5 ppmv to 1000 ppmv for ethane, ethene and 0.5 ppmv to 25,000 ppmv for methane. The calibration range for CO and CO2 encompasses 20 ppmv to 25000 ppmv.

10.2.1 Standards are drawn directly from gas cylinders, fitted with a regulator and syringe adapter that contain the target analytes at certified concentrations. The gas cylinder outlet pressure is set at approximately 10 psig. Insert syringe without plunger into syringe adapter and purge for 5-10 seconds with the calibration gas to eliminate all dead volume. Insert plunger, turn off cylinder, and adjust syringe to the injection volume required.

10.2.2 When the GC has reached the specified operating conditions, manually inject the first standard and immediately push the "start" button on the GC to begin acquisition.

10.2.3 Low and Medium level Calibration Points – Prepare additional calibration standards by using different volumes of the 1000 ppmv calibration gas standard to complete the calibration as follows:

Cal Gas Volume (cc)	Cal Standard Concentration (ppmv)	Conc (ppmv) Methane, Ethane, Ethene	Conc (ppmv) CO, CO2
0.10	2.5	0.5	-
0.001	1000	2	-
0.002	1000	4	4
0.010	1000	20	20
0.050	1000	100	100
0.25	1000	500	500
0.5	1000	1000	1000
0.10	50000	10000 methane only	10000
0.25	50000	25000 methane only	25000

10.2.4 Calculate the response factor for each target compound at each concentration using the formula specified in section 10.6.1. The response factor is defined as peak area divided by standard concentration in ppmv.

10.2.5 Initial Calibration – Calculate the relative standard deviation (RSD) of the response factors from each calibration standard for every target compound. The RSD must be less than 30% using a minimum of 5 calibration levels.

10.2.5.1 Individual calibration standards may be eliminated form the calibration curve at the extremes of the range. If the low calibration standard is eliminated, the reporting limit for that compound must be adjusted accordingly.

10.2.5.2 If the RSD criteria cannot be achieved, corrective action must be performed. The source of the difficulties must be identified and eliminated and the calibration standards reanalyzed.

10.2.5.3 A single standard in the calibration curve may be reanalyzed once if it is evident that transient technical difficulties caused this analysis to fail, negatively impacting the RSD calculation.

10.2.6 Helium Blank. Prepare a helium blank by withdrawing 500 ul of helium carrier gas from the sampling port of the carrier gas line. Inject the helium sample into the gas chromatograph. Obtain peak areas for all target compounds that are present as

contaminants in the carrier gas. This data will be used to background correct analytes detected in field samples. The background correction will be performed using the dissolved gas calculation worksheet.

10.2.7 Method Blank. Prepare and analyze a method blank before initiating sample analysis.

10.2.7.1 Fill a blank vial with deionized, organic free water making sure that there isn't any headspace.

10.2.7.2 Establish a helium flow of approximately 5cc/min the purge needle attached to the carrier gas sampling valve. Insert the purge needle into the septa of the blank sample vial.

10.2.7.3 Simultaneously insert a 5ml syringe with needle into the blank sample to collect and measure and document the water volume displaced by the helium using a Class A graduated cylinder. Make sure needle tip is below the level of the purging helium needle tip. The pressure of the helium flow will force the water sample up into the collection syringe.

10.2.7.4 When the gas displacement step has been completed, simultaneously remove both needles. You have now displaced 5ml of sample with 5cc of inert helium.

10.2.7.5 Proceed with step 10.4.5.

10.2.7.6 If the method blank contains the presence of target compounds exceeding the MDL, the background contamination must be eliminated before proceeding with sample analysis.

10.2.8 Continuing Calibration – The 100 ppbv calibration check standard is analyzed every 12 hours and at the end of the daily sequence. Inject 0.05cc of the 1000 ppmv stock standard into the gas chromatograph for analysis.

10.2.8.1 Following analysis of the check sample, calculate the concentration of each target compound in the check using peak area.

10.2.8.2 Calculate the percent difference (%D) between the calibration check and the initial calibration. The %D of each target compounds must be <15%.

10.2.8.2.1 If calibration standard exceeds the 15% D criteria, reanalyze a second calibration standard. If a second calibration does not meet the <15% D criteria then a new initial calibration must be prepared and analyzed.

10.2.8.3 If the check standard at the end of the analytical sequence fails, the system must be recalibrated and all previous samples, up to the previous calibration check, must be reanalyzed.

10.3 Retention Time Windows

10.3.1 Retention Time Windows. Retention time windows must be calculated for each analyte on each GC column and whenever an initial calibration is analyzed or new GC column is installed. The retention time data must be retained in the laboratory.

10.3.2 Analyze the standard mixtures three times at approximately equal intervals, during a 72-hr period. Determine the retention time of each analyte in each analysis.

10.3.3 Calculate the standard deviation of the absolute retention times for each the replicate analysis of each target component. Multiply the standard deviation by $\pm$ 3 to establish the retention time range.

10.3.4 Apply the retention time range to the absolute retention time of each standard compound in the continuing calibration. This will be used to define the retention time window for the sample. Update the retention range using the retention range windows each time a continuing calibration is analyzed.

10.4 Sample Preparation and Analysis.

10.4.1 Remove samples from cold storage and allow samples to reach ambient temperature.

10.4.2 Select a sample to be analyzed in duplicate at a minimum of one per ten.

10.4.3 Prepare a blank following the procedure detailed in section 10.2.7.

10.4.4 Prepare all samples and duplicate samples selected for analysis following the procedure detailed in section 10.2.7.

10.4.4.1 After measuring the volume of sample displaced, check the pH with wide range pH paper to determine if it had been properly preserved. Document the ph on the analysis log. Inform clients services if the pH does not meet criteria.

10.4.5 Securely place samples, blanks and duplicate samples on a wrist action shaker and set timer for 20 minutes. When finished remove and place upright in vial rack for analysis.

10.4.6 Place the NIST calibrated thermometer in the sample rack for ambient temperature measurement.

10.4.6.1 Obtain a 40ml VOA vial and fill with water. Cap and insert a thermometer (5-50°C) through the septa into the water and allow to equilibrate

10.5 Sample Analysis.

10.5.1 Establish the sequence of samples to be analyzed. The nominal sample injection volume is 0.5cc. Sequentially inject 0.5 cc of each sample into the gas chromatograph acquiring data for the duration of the chromatographic analysis.

10.5.2 The daily analytical sequence is as follows:

10.5.2.1 Inject 100 ppmv (0.05cc of 1000ppmv standard) daily calibration check standard,

10.5.2.2 Inject 100 ppmv (0.05cc of 1000ppmv alternate standard) Laboratory Control Sample,

10.5.2.3 Inject 0.5cc of the helium blank used to prepare samples. This will be used for correcting any background peak areas,

10.5.2.4 Inject 0.5cc of prepared method blank (DI water prepared identical to samples),

10.5.2.5 Inject 0.5cc of any samples and quality control samples.

10.5.3 Quantitate the helium blank following the procedure in section 10.2.6 to determine background area of target compounds. Typically methane and CO2 are present at low concentrations in the carrier gas and must be background corrected.

10.5.4 Process the data from the method blank and run the custom report template to ensure all compounds are below the detection limits. Initiate corrective action if the blank criteria are not achieved.

10.5.5 Quantitate each sample following analysis to determine if the concentration of the target compound exceeds the calibration range. If the range is exceeded, inject a smaller volume of the headspace gas as a dilution until the concentration of the target analytes is with the established calibration range. Dilution injections must be performed immediately after the undiluted analysis to minimize the loss of target analytes through the septa.

10.5.6 Measure and document the ambient temperature in degrees C (to nearest whole degree) using the thermometer in the sample rack.

10.5.7 Measure and document the volume of water (ml) that remains in each sample, duplicate, and blank using the Class A graduated cylinder. The actual vial volume is then the remaining sample plus the displaced sample volume (5ml). Do not assume that the sample vial is a precise and accurate 40 ml. These vials are actually 40-44cc volume depending on the lot numbers and vendors.

10.6 Calculations.

10.6.1 Calibration Factor (CF). Calculate for each target compound in each calibration standard.

$$CF = \frac{As}{Cs}$$

Where:

As= Area of the peak for the compound being measured.
Cs= Concentration of the compound being measured (µg/l).

10.6.2 Mean RF (XRF). Calculate the mean RF for each target compound in the calibration range.

$$XRF = \frac{R_1 + R_2 + \ldots R_n}{n}$$

Where:

R = Response factor of each individual calibration standard.
n = Number of calibration standard analyzed.

10.6.3 Standard Deviation (SD). Calculate the standard deviation of the XRF for each compound.

$$SD = \frac{\sum_{i=1}^{n} (RF_i - XRF)^2}{n-1}$$

Relative Standard Deviation (RSD). Calculate the relative standard deviation for each calibration compound.

$$RSD = \frac{SD}{XRF} \times 100$$

10.6.5 Percent Difference (% D).

$$\% D = \frac{|XRF - RFc|}{XRF} \times 100$$

Where:

RFc = CF from calibration verification.

10.6.6 Gas Concentration to Aqueous Concentration Conversion. Measured concentrations of target analytes must be converted form gas volume/volume concentrations to aqueous concentrations (ug/l).

10.6.6.1 Sample Concentrations are initially determined as PPMV concentration using the external standard technique from calibration curve.

$$\text{Conc. } (\mu g/l) = \frac{Ac \times D}{XRF}$$

Where:
Ac = Area of peak for compound being measured.
D = Dilution factor based on volume of headspace injected = 0.5/cc injected
XRF = Average response factor from initial calibration.

10.6.6.2 Gas Concentration Conversion to Aqueous Concentration Using Custom Reports.

10.6.6.2.1 From the gas chromatograph PC, open the custom reports file "natatt" under the "Custom Reports" menu. See attachment of template.

10.6.6.3 Enter helium blank background peak areas of target compounds in appropriate field if they exist.

10.6.6.4 Enter headspace created from helium displacement (typically 5cc)

10.6.6.5 Enter volume of sample (ml) remaining in vial. (vial volume – sample volume remaining)

10.6.6.6 The correct ug/l concentration will automatically be calculated based on Henry's Law constant, temperature of the sample, volume of sample vial, headspace volume, Dilution Factor, and the molecular weight of the gas.

10.6.6.7 The attached template describes how all calculations are derived along with a table of Henry's Constants for each gas.

10.6.7 Percent Recovery (% R) for LCS

$$\% R = \frac{\text{Concentration found}}{\text{Concentration spiked}} \times 100$$

10.6.8 Relative Percent Difference (RPD) for sample duplicates.

$$RPD = \frac{|SR1 - SR2|}{(1/2)(SR1+SR2)} \times 100$$

Where:
SR1 = First sample replicate concentration (ug/l).
SR2 = Duplicate sample concentration (ug/l)

11.0 Quality Control.

11.1 QC Requirements Summary

Initial Calibration	-
Calibration Check std.	Every 12 hrs
LCS	Every 12 hrs
Helium Blank	Every 12 hrs
Batch blank	one per batch [a]
Matrix Duplicate	one per batch [a]

(a) The maximum number of samples per analytical batch is twenty or every twenty four (24) hours, whichever occurs first.

11.2 Daily calibration check - refer to section 10.2.8.

11.3 Method blank.

11.3.1 A method blanks is prepared and analyzed every 12 hours immediately following calibration or calibration verification. The method blank is prepared and analyzed using the same procedures as a field sample.

11.3.2 *No compound can be present above the reporting level which is determined after* running custom reports.

11.3.3 If the method blank does not meet the criteria, a second blank can be analyzed.

11.3.3.1 If the second blank does not meet the criteria, the entire batch must be re-prepared and reanalyzed.

11.4 Matrix Duplicate

11.4.1 A sample duplicate must be analyzed with every 10 samples.

11.4.2 The relative percent deviation (RPD) (see section 12.6) is calculated for the original and the duplicate samples. The values are compared to the in-house generated control limits.

11.5 Laboratory Control Sample (LCS)

11.5.1 An LCS is required for each batch of 20 samples or every twenty four (24) hours, whichever occurs first.

11.5.2 The LCS % recoveries must be between 70-130%.

11.5.2.1 If the recovery of LCS is outside the control limits, check the calculation. Recalculate if an error is detected.

11.5.2.2 If no error is detected prepare a new standard and reanalyze the LCS.

12.0 Documentation

12.1 The analysis logbook is used to document the analysis sequence; the logbook must be completed daily. A separate logbook is required for each instrument.

12.1.1 If samples require reanalysis, a brief explanation of the reason must be documented in the comments section of the analysis logbook.

12.1.2 The standards preparation logbook must be completed for all prepared standards. All requested information must be completed; the page must be signed and dated by the appropriate person.

12.1.2.1 The Accutest lot number must be cross-referenced on the gas standard cylinder.

12.1.3 The Instrument Maintenance Logbook must be completed when maintenance is performed on the instrument. A separate maintenance logbook is required for each instrument.

12.1.4 The custom report calculation template must be generated for all samples, method blanks, and duplicates.

13.0 References

13.1 "Analysis of Dissolved Methane, Ethane, and Ethylene in Ground Water by a Standard Gas Chromatographic Technique". Published in Journal of Chromatographic Science, vol. 36 May 1998. Authored by Don H. Kampbell and Steve A. Vandegrift

13.2 USEPA RSK SOP-175; Sample Preparation and Calculations for Dissolved Gas Analysis in Water Samples Using a GC Headspace Equilibrium Technique; Bryan Newel, USEPA Robert S. Kerr Center, Ada, Oklahoma; August 11, 1994.

14.0 POLLUTION PREVENTION & WASTE MANAGEMENT.

14.1 Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 14.2.

14.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the waste management SOP, ESM003. This document describes the proper disposal of all waste materials generated during the testing *of samples as follows:*

14.2.1 Non hazardous aqueous wastes.

14.2.2 Hazardous aqueous wastes

14.2.3 Chlorinated organic solvents

14.2.4 Non-chlorinated organic solvents

14.2.5 Hazardous solid wastes

14.2.6 Non-hazardous solid wastes

Table 1 - Recommended GC Operating Conditions	
Carrier Gas	Helium
Detector Gas	Hydrogen, Zero Air
Injection Type	Splitless
Purge off time	0.8 min
Column Flow	EPC constant flow at 15psig @ 30c
Initial temperature	30c for 1minute
Oven Ramp	30c/min to 180c with no hold
Injection temp	230c
FID temp	300c
TCD temp	180c

ACCUTEST LABORATORIES
Standard Operating Procedures

Lab Manager_____

QA Manager_____

Effective Date

TEST NAME **SW846 8082: DETERMINATION OF POLYCHLORINATED BIPHENYLS (PCBs) USING GC SYSTEM**

METHOD REFERENCE SW846 8082 (Revision 0, December 1996)

Revised Sections: ***Table 3a, 3b, 4a, 4b, 4c, 5a, 5b***

1.0 SCOPE AND APPLICATION

1.1 This SOP describes the analytical procedures, which are utilized by Accutest to acquire samples for analysis of polychlorinated biphenyls (PCBs) as Aroclors, using dual open-tubular, capillary columns with electron capture detectors (ECD).

1.2 This gas chromatographic (GC) method applicable to the determination of the PCB Aroclors listed in Table 1 in extracts from solid and aqueous matrices.

2.0 SUMMARY OF METHOD

2.1 A measured volume or weight of sample (approximately 1 L for liquids, 15 g for solids) is extracted using the appropriate matrix-specific sample extraction technique. Petroleum Products and organic wastes are diluted with an organic solvent and follow SW 846 Method 3580A. Aqueous samples are extracted at neutral pH with methylene chloride using Method 3510 (separatory funnel). Solid samples are extracted with using Method 3545, Pressurized Fluid Extraction.

2.2 Extracts for PCB analysis may be subjected to a sulfuric acid/potassium permanganate cleanup (Method 3665) designed specifically for these analytes. This cleanup technique will remove (destroy) many single component organochlorine or organophosphorus pesticides.

2.3 After cleanup, the extract is analyzed by injecting a 1 or 2-µL aliquot into a gas chromatograph with dual narrow bore fused silica capillary columns and electron capture detectors (GC/ECD). The chromatographic data may be used to determine the seven Aroclors in Table 1.

2.4 The peaks detected are qualitatively identified by comparison to retention times specific to the known target list of PCBs on two different column types (primary and confirmation).

2.5 Once identified, the Aroclor is quantitated by external standard techniques with an average calibration factor generated from a calibration curve.

3.0 REPORTING LIMIT AND METHOD DETECTION LIMIT

3.1 Reporting Limit. The reporting limit for this method is established at the lowest concentration standard in the calibration curve. RL's may vary depending on matrix difficulties and sample volumes or weight and percent moisture. Refer to Table 1 for current reporting limits.

3.2 Method Detection Limit. Experimentally determine MDLs using the procedure specified in 40 CFR, Part 136, Appendix B. This value represents the lowest reportable concentration of an individual compound that meets the method qualitative identification criteria.

3.2.1 Experimental MDLs must be determined annually for this method.

3.2.2 Process all raw data for the replicate analysis in each MDL study. Forward the processed data to the QA group for archiving.

4.0 DEFINITIONS

BLANK - an analytical sample designed to assess specific sources of laboratory contamination. The types of blanks are Method Blank; Instrument Blank, Storage Blank, and Sulfur Blank.

CALIBRATION FACTOR (CF) - a measure of the gas chromatographic response of a target analyte to the mass injected. The calibration factor is analogous to the Relative Response Factor (RRF) used in the Volatile and Semivolatile fractions.

CONTINUING CALIBRATION - analytical standard run every 12 hours and at the end of analytical sequence to verify the initial calibration of the system.

CONTINUOUS LIQUID-LIQUID EXTRACTION - used herein synonymously with the terms continuous extraction, continuous liquid extraction, and liquid extraction. This extraction technique involves boiling the extraction solvent in a flask and condensing the solvent above the aqueous sample. The condensed solvent drips through the sample, extracting the compounds of interest from the aqueous phase.

INITIAL CALIBRATION - analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of the electron capture detector to the target compounds.

MATRIX - the predominant material of which the sample to be analyzed is composed. For the purpose of this SOP, a sample matrix is either water or soil/sediment. Matrix is <u>not</u> synonymous with phase (liquid or solid).

MATRIX SPIKE - aliquot of a matrix (water or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

MATRIX SPIKE DUPLICATE - a second aliquot of the same matrix as the matrix spike (above) that is spiked in order to determine the precision of the method.

METHOD BLANK - an analytical control consisting of all reagents, internal standards and surrogate standards (or SMCs for VOA), that is carried throughout the entire analytical procedure. The method blank is used to define the level of laboratory, background and reagent contamination.

METHOD DETECTION LIMITS (MDLs) - The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. MDLs should be determined approximately once per year for frequently analyzed parameters.

PERCENT DIFFERENCE (%D) - As used in this SOP and elsewhere to compare two values, the percent difference indicates both the direction and the magnitude of the comparison, i.e., the percent difference may be either negative, positive, or zero. (In contrast, see relative percent difference.)

PERCENT MOISTURE - an approximation of the amount of water in a soil/sediment sample made by drying an aliquot of the sample at 105°C. The percent moisture determined in this manner also includes contributions from all compounds that may volatilize at or below 105 °C, including water. Percent moisture may be determined from decanted samples and from samples that are not decanted.

REAGENT WATER - water in which an interferant is not observed at or above the minimum detection limit of the parameters of interest.

RELATIVE PERCENT DIFFERENCE (RPD) - As used in this SOP and elsewhere to compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero. (In contrast, see percent difference.)

RELATIVE RESPONSE FACTOR (RRF) - a measure of the instrument response of an analyte. Response Factors are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples.

RETENTION TIME (RT) - the time required (in minutes) for a standard compound to elute from a chromatographic column.

SURROGATES - for semivolatiles and pesticides/Aroclors, compounds added to every blank, sample, matrix spike, matrix spike duplicate, and standard; used to evaluate analytical efficiency by measuring recoveries. Surrogate are brominated, fluorinated, or isotopically labeled compounds not expected to be detected in environmental media.

INSTRUMENT BLANK - a system evaluation sample containing solvent and surrogate standards added. An instrument blank is used to remove and/or evaluate residual carryover from high level standards, spike samples and field samples.

5.0 HEALTH & SAFETY

5.1 The analyst must follow normal safety procedures as outlined in the Accutest Health and Safety Plan and Personal Protection Policy, which includes the use of safety glasses and lab coats. In addition, all acids are corrosive and should be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these reagents should be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets should be made available to all personnel involved in these analyses.

5.3 Polychlorinated biphenyls have been classified as known or suspected human or mammalian carcinogens. Primary standards of these toxic compounds should be prepared in a hood. A NIOSH/Mass approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

6.0 INTERFERENCES

6.1 The data from all blanks, samples, and spikes must be evaluated for interferences.

6.2 Cross-contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled. Glassware must be scrupulously cleaned. Refer to "The Preparation of Glassware for Extraction of organic contaminants" SOP for practices utilized in the extraction department.

6.3 Interferences may be caused by contaminants that are co-extracted from the sample. The extent of the interferences will vary from source to source, which can be grouped into three broad categories.

6.3.1 Contaminated solvents, reagents, or sample processing hardware.

6.3.2 Contaminated GC carrier gas, parts, column surfaces, or detector surfaces.

6.3.3 Compounds extracted from the sample matrix to which the detector will respond.

6.4 Interferences by phthalate esters introduced during sample preparation can pose a major problem in PCB determination.

6.4.1 Common flexible plastics contain varying amounts of phthalate esters which are easily extracted or leached from such materials during laboratory operations. Avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination can best minimize interference from phthalate esters.

6.4.2 Exhaustive cleanup of solvents, reagent and glassware may be required to eliminate background phthalate ester contamination.

6.4.3 These materials can be removed through the use of Method 3665 (sulfuric acid/permanganate cleanup).

6.5 Elemental sulfur is readily extracted from soil samples and may cause chromatographic interferences in the determination of PCBs. Method 3660 is suggested for removal of sulfur.

6.6 To reduce carryover when high-concentration samples are sequentially analyzed, the syringe must be rinsed out between samples with solvent. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of an instrument blank to check for cross contamination.

7.0 SAMPLE PRESERVATION AND HOLDING TIME

7.1 PRESERVATION

7.1.1 Water Samples

7.1.1.1 Collect samples in 1 liter glass amber bottles without preservatives.

7.1.1.2 A liter of an unpreserved sample is required for extraction. Additional sample volume is necessary for any samples used for matrix spike and matrix spike duplicates. Therefore, 3 liters of at least one sample in every group of 20 field samples are required for analysis to accommodate all quality control requirements.

7.1.2 Soil Samples

7.1.2.1 Samples are collected in a 300-ml amber glass sample bottle. No preservative is required.

7.1.3 Sample should be taken with care so as to prevent any portion of the collected sample coming in contact with the sampler's gloves, thus causing possible phthalate contamination.

7.1.4 The samples must be protected from light and refrigerated at 4 °C (± 2 °C) from the time of receipt until extraction and analysis.

7.2 HOLDING TIME

7.2.1 Aqueous sample must be extracted within 7 days of sampling.

7.2.2 Soil sample must be extracted within 14 days of sampling.

7.2.3 Extracts must be analyzed within 40 days following extraction.

8.0 APPARATUS AND MATERIALS

8.1 GAS CHROMATOGRAPH SYSTEM

8.1.1 Gas Chromatograph-Agilent or Hewlett Packard Model 5890 and 6890. The analytical system complete with a temperature programmable gas chromatograph and all required accessories including syringes, analytical columns, and gases. The injection port is designed for splitless injection with capillary columns. The capillary columns are directly coupled to the detectors.

8.1.2 Columns

8.1.2.1 Column pair 1

8.1.2.1.1 30 m x 0.32 mm fused silica (0.5 µm film thickness) DB-1701 narrow-bore capillary column or equivalent.

8.1.2.1.2 30 m x 0.32 mm fused silica (0,5 µm film thickness) DB-5 narrow-bore capillary column or equivalent.

8.1.2.2 Column pair 2

8.1.2.2.1 30 m x 0.32 mm fused silica (0.5 µm film thickness) RTX CLPI narrow-bore capillary column or equivalent.

8.1.2.2.2 30 m x 0.32 mm fused silica (0.25 µm film thickness) RTX CLPII narrow-bore capillary column or equivalent.

8.1.3 Detectors

8.1.3.1 Electron Capture Detectors (HP).

8.1.3.2 Micro Electron Capture Detectors (HP).

8.2 AUTOSAMPLER

8.2.1 Agilent or Hewlett Packard Model 7673A, capable of holding 100 of 2-ml crimp vials.

8.3 DATA SYSTEM

8.3.1 HP Chemstation, interfaced to the gas chromatograph which allows the continuous acquisition and storage on machine readable media (disc) of all chromatographic data obtained throughout the duration of the analysis.

8.3.2 The ENVIROQUANT data system is capable of quantitation using multi-point calibration.

8.3.3 Lagato Networker with lookup database on 4mm DAT tape for long term, off line magnetic storage of data.

8.4 SYRINGE

8.4.1 Manually held ul-syringes, various volumes (Hamilton or equiv.).

8.4.2 10 µl graduated, auto sampler (Hamilton or equiv.).

9.0 REAGENTS AND STANDARDS

9.1 Refer to Accutest Sample Preparation SOPs EOP001 and EOP040 for reagents and standards used for sample extraction.

9.2 Solvents - Ultra pure, chromatography grade Hexane.

9.3 Stock standard solutions.

9.3.1 Two separate sources of commercially prepared standards with traceability documentation are used. The standards contain Aroclors 1016, 1221, 1232, 1242, 1248, 1254 and 1260.

9.4 Working Solutions

9.4.1 Prepare working solutions, using stock solution, in hexane, as needed, that contain the compounds of interest, either singly or mixed together. Refer to Table 3A, 3B for details.

9.5 Calibration Standards

9.5.1 Initial Calibration Standards

9.5.1.1 A standard containing a mixture of Aroclor 1016 and Aroclor 1260 will include many of the peaks in the other five Aroclor mixtures. As a result, a multi-point calibration employing a mixture of Aroclors 1016 and 1260 at five concentrations should be sufficient to demonstrate the linearity of the detector response without the necessity of performing initial calibration for each of the seven Aroclors. Prepare a minimum of five calibration standards containing equal concentrations of both Aroclor 1016 and Aroclor 1260, including surrogates, by dilution of the above working solutions (Section 9.4) with hexane. Suggested levels and preparations are shown in Table 4A.

9.5.1.2 Separate calibration standards are required for the other five Aroclors. Unless otherwise necessary for a specific project, a single calibration standard near the mid-point of the expected calibration range of each remaining Aroclor is employed to determine its calibration factor. Refer to Table 4B for preparation scheme. Optional curves as shown on Table 4C may also be used for a multi-point calibration per project's specification.

9.5.2 Continuing Calibration Verification (CCV)

9.5.2.1 For Aroclor analyses, the continuing calibration checks should be a mixture of Aroclor 1016 and Aroclor 1260. Two standards at 500 µg/l and 1,000 µg/l are prepared as described in Table 5A. During the analysis, these two solutions are alternated to check the initial calibration.

9.5.2.2 In situations where only a few Aroclors are of interest for a specific project, the calibration checks of each Aroclor of interest may be prepared (Table 5B) and analyzed as the 1016/1260 mixture throughout the analytical sequence.

9.6 Initial Calibration Verification (ICV) - Second Source Calibration Check Standard

9.6.1 Prepare the ICV check standards from separate sources of stock standards from the calibration curve following the procedures in Table 6A, and 6B.

9.6.2 The ICV is prepared at 1,000 µg/l for each Aroclor and is analyzed immediately after and initial calibration.

9.7 Surrogates

9.7.1 Tetrachloro-m-xylene (TCMX) and decachlorobiphenyl (DCB) are used as surrogate standards for this method.

9.7.2 A calibration range must be constructed for the surrogate compounds. Accordingly, appropriate amounts of surrogates are mixed with each calibration solution to define a range similar to the target compounds.

9.7.3 Surrogate compounds are also contained in continuing calibration checks, and second source calibration check standard.

9.7.4 Spike each sample, QC sample and blank with an appropriate amount of corresponding surrogate spiking solution, prior to extraction, for a final concentration in the extract of 40 µg/l of each surrogate compound.

9.8 Storage of Standards

9.8.1 Store unopened stock standard solutions according to the manufacturer's documented holding time and storage temperature recommendations. Protect from light.

9.8.2 Store all other working standard solutions in glass vials with Teflon lined screw caps at 4°C (± 2°C) in the dark.

9.8.3 Opened stock standard solutions must be replaced after one year or sooner if manufacturer's expiration date comes first or comparison with quality control check samples indicates a problem.

9.8.4 All other standards must be replaced after six months or sooner if routine QC indicates a problem or manufacturer's expiration date comes first.

10.0 CALIBRATION

10.1 Initial Calibration

10.1.1 The method reporting limit is established by the concentration of the lowest standard analyzed during the initial calibration. Lower concentration standard may be needed to meet the reporting limit requirements of state specific regulatory program. The linear range covered by this calibration is the highest concentration standard.

10.1.2 The initial calibration for this method consists of two parts, described below.

10.1.2.1 A standard containing a mixture of Aroclor 1016 and Aroclor 1260 will include many of the peaks represented in the other five Aroclor mixtures. Thus, such a standard may be used to demonstrate the linearity of the detectors and that a sample does not contain peaks that represent any one of the Aroclors. This standard can also be used to determine the concentrations of either Aroclor 1016 or Aroclor 1260, should they be present in a sample. The calibration range covered for Aroclor 1016 and Aroclor 1260 employs standards of 50, 250, 500, 1,000, 2,000, and 3,000 µg/l.

10.1.2.2 Standards of the other five Aroclors are necessary for pattern recognition. These standards are also used to determine a single-point calibration factor for each Aroclor, assuming that the Aroclor 1016/1260 mixtures in Section 10.1.2.1 has been used to describe the detector response. The concentration of each Aroclor standard is near the mid-point of the linear range of the detector, usually at 1,000 µg/l. The standards for these five Aroclors should be analyzed before the analysis of any samples, and may be analyzed before or after the analysis of those 1016/1260 standards.

10.1.2.3 In situations where only a few Aroclors are of interest for a specific project, an initial calibration of a minimum of five standards of each Aroclors of interest instead of the 1016/1260 mixture may be performed.

10.1.3 A calibration range must be constructed for each surrogate compound. Accordingly, add appropriate amounts of each surrogate compound to the calibration solution to define a range similar to the target compounds.

10.1.4 Aliquot proper amount of each calibration standard into a 2 ml crimp top vial.

10.1.5 PCBs are quantitatively determined as Aroclors by the external standard technique. The Calibration Factor (CF) for each characteristic Aroclor peak in each of the initial calibration standards is calculated using the equation in Section 14.1.

10.1.5.1 Use at least five peaks for the Aroclor 1016/1260 mixture, none of which should be found in both of these Aroclors. At least five sets of calibration factors will be generated, each set consisting of the calibration factors for each of the five (or more) peaks chosen for this mixture.

10.1.5.2 A minimum of 3 characteristic peaks must be chosen for each of the other Aroclors, and preferably 5 peaks. The peaks must be characteristic of the Aroclor in question. Thus, each single standard will generate at least three calibration factors, one for each selected peak.

10.1.5.3 Choose peaks in the Aroclor standards that are at least 25% of the height of the largest Aroclor peak. For each Aroclor, the set of 3 to 6 peaks should include at least one peak that is unique to that Aroclor.

10.1.5.4 The calibration factors from the initial calibration are used to evaluate the linearity of the initial calibration. When the Aroclor 1016/1260 mixture is used to demonstrate the detector response, the calibration model chosen for this mixture must be applied to the other five Aroclors for which only single standards are analyzed. If multi-point calibration is performed for individual Aroclors, use the calibration factors from those standards to evaluate linearity.

10.1.6 For the initial calibration to be valid, the percent relative standard deviation (% RSD) (see Section 14.2) must be less than 20 % for each Aroclor of interest on each column. *If any analyte exceeds the 20% acceptance limit for a given calibration, corrective action* must be taken.

10.1.6.1 If the problem is associated with specific standards, reanalyze the standard and recalculate the RSD.

10.1.6.2 Alternatively, narrow the calibration range by replacing one or more of the calibration standards that cover a narrow range.

10.1.6.2.1 The changes to the upper end of the calibration range will affect the need to dilute samples above the range. If the instrument response indicates signs of detector saturation, the concentration of the standard at the upper limit will be reduced. The changes to the lower end will affect the overall sensitivity of the method. Consider the regulatory limits or action levels associated with the target analytes when adjusting the lower end of the range.

10.2 Initial Calibration Verification (ICV) - Second Source Calibration Check Standard

10.2.1 The initial calibration is verified with an ICV, a second source calibration check standard from an external source (Section 9.6). It must be performed right after the initial calibration.

10.2.2 *The percent difference (%D) (Section 14.3) for this standard must meet the %D criteria* of 15% used for calibration verification on each column.

10.2.2.1 If %D is greater than 15%, reanalyze the second source check. If the limit cannot be met upon re-injection, re-prepare the second source solution using a fresh ampoule and repeat the process.

10.2.2.2 If the %D criteria cannot be achieved after re-preparation of the second source, prepare a third source and repeat the process. Make fresh calibration standards using one of the two standard sources that matches each other.

10.3 Continuing Calibration Verification (CCV)

10.3.1 Continuing calibration verificatiion (CCV) standards (Section 9.5.2) must be acquired at the beginning of each 12-hour shift, after every 10 samples analyzed and at the end of the analysis sequence. The 500 µg/l check standard is alternated with 1,000 µg/l standard for calibration verification.

10.3.2 For Aroclor analyses, the calibration verification standard should be a mixture of Aroclor 1016 and Aroclor 1260. The calibration verification process does not require analysis of the other Aroclor standards used for pattern recognition, but the analyst may wish to include a standard for one of these Aroclors after the 1016/1260 mixture used for calibration verification throughout the analytical sequence.

10.3.3 The percent difference (%D) (see section 14.3) must be less than 15 % for each Aroclor of interest on each column.

10.3.4 Each sample analysis must be bracketed by periodic analyses of acceptable calibration verification standards every 10 samples or 12 hours, whichever is more frequent. If %D criteria fails during a mid sequence calibration check or at the end of the analysis sequence, a continuing calibration check is allowed to be repeated only once; if the second trial fails, a new initial calibration must be performed. In situations where the first check fails to meet the criteria, the instrument logbook should have clear documented notations as to what the problem was and what corrective action was implemented to enable the second check to pass.

10.3.5 A continuing calibration standard is analyzed whenever the analyst suspects that the analytical system is out of calibration. If the calibration cannot be verified, corrective action is performed to bring the system into control. Analysis may not continue until the system is under control.

10.3.6 When a calibration verification standard fails to meet the QC criteria at the end of the analysis sequence, all samples injected after the last standard that met the QC criteria must be evaluated to prevent mis-quantitations, and re-injection of the sample extracts may be required.

10.3.6.1 The analyte was not detected in the specific samples analyzed during the analytical shift, then the extracts for those samples do not need to be reanalyzed when the calibration standard response is above the initial calibration response.

10.3.6.2 Whether the analyte was detected in the specific samples analyzed during the analytical shift, or the calibration standard response is below the initial calibration response, then the extracts for those samples need to be reanalyzed.

10.3.7 Each subsequent injection of a continuing calibration standard during the 12-hour analytical shift must be checked against the retention time windows established in Section 11.0. If any of these subsequent standards fall outside their absolute retention time windows, the GC system is out of control. Determine the cause of the problem and correct it. If the problem cannot be corrected, a new initial calibration must be performed.

11.0 RETENTION TIME WINDOWS

11.1 Absolute retention times are used for the identification of PCBs as Aroclors. Retention time windows must be calculated for each surrogate and at least 3 to 5 characteristic peaks of each Aroclor on each GC column and whenever a new chromatographic column is installed, or when there are significant changes in the operating conditions. The retention time windows must be reported with the analysis results in support of the identifications made.

11.2 Employ the following approach to establish retention time windows:

11.2.1 Make three injections of each Aroclor at approximately equal intervals during the 72-hr period.

11.2.2 For each Aroclor, choose three or five major peaks and calculate the mean and standard deviation of the three retention times for that peak. The peak chosen should be fairly immune to losses due to degradation and weathering in the samples. Record the retention time to three decimal places (e.g. 10.015 min) for each Aroclor.

11.2.3 In those cases where the standard deviations of the retention time window for a particular Aroclor is 0.01 minutes or less, the laboratory may either collect data from additional injections of standards or use a default standard deviation of 0.01 minutes.

11.2.4 Apply plus or minus three times the standard deviations to retention time of each Aroclor standard (continuing calibration or middle level of initial calibration). This will be used to define the retention time window for the sample.

11.2.4.1 If default standard deviation of 0.01 minutes is employed, the width of the window will be 0.03 minutes.

11.2.5 Establish the center of the retention time window for each Aroclor and surrogate by using the absolute retention time for each Aroclor and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration.

12.0 PROCEDURE

12.1 Sample Extraction

12.1.1 In general, water samples are extracted at a neutral pH with methylene chloride using a separate funnel (Method 3510) (Refer to SOP: EOP001). Solid samples are extracted using Method 3545, Pressurized Fluid Extraction (Refer to SOP: EOP040).

12.2 Sample Cleanup

12.2.1 Cleanup procedures may not be necessary for a relatively clean sample matrix, but most extracts from environmental and waste samples will require additional preparation before analysis. The specific cleanup procedure used will depend on the nature of the sample to be analyzed and the data quality objectives for the measurements. Refer to appropriate SOPs for details.

12.2.1.1 Interferences by phthalate esters can be removed through the use of a sulfuric acid/potassium permanganate cleanup (Method 3665) designed specifically for PCBs. This method should be used whenever elevated baselines or overly complex chromatograms prevent accurate quantitation of PCBs.

12.2.1.2 Element sulfur, which may be present in certain sediments and industrial wastes, interfere with the electron capture gas chromatography of certain Aroclors. Sulfur should be removed by the technique described in Method 3660.

12.3 Instrument conditions.

12.3.1 Recommended instrument conditions are listed in Table 2. Modifications of parameters specified with an asterisk are allowed as long as criteria of calibration are met. Any modification should be approved by team leader/manager.

12.4 Initial calibration

12.4.1 Refer to Section 10.1.

12.5 Initial calibration Verification (ICV) -Second source calibration check standard

12.5.1 Refer to Section 10.2.

12.6 Continuing calibration Verifications (CCV)

12.6.1 Refer to Section 10.3.

12.7 Sample analysis (Primary)

12.7.1 All samples and quality control samples are injected into the Gas Chromatograph using the autosampler. Program the sampler for an appropriate number of syringe rinses and a 1ul or 2 µl injection size. A splitless injection technology is used.

12.7.2 Sample concentrations are calculated by comparing sample responses with the initial calibration of the system (Section 14.4). If sample response exceeds the limits of the initial calibration range, dilute the extract and reanalyze. Extracts should be

diluted so that all peaks are on scale, as overlapping peaks are not always evident when peaks are off scale.

12.7.3 Sample injections may continue for as long as the calibration verification standards and standards interspersed with the sample meet instrument QC requirements. The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria are exceeded.

12.7.4 If the peak response is less than 2.5 times the baseline noise level, the validity of the quantitative result may be questionable. The analyst should consult with the source of the sample to determine whether further concentration of the sample is warranted.

12.7.5 If compound identification or quantitation is precluded due to interference (e.g., broad, rounded peaks or ill-defined baselines are present) cleanup of the extract or replacement of the capillary column or detector is warranted. Rerun the sample on another instrument to determine if the problem results from analytical hardware or the sample matrix.

12.8 Confirmation analysis.

12.8.1 Confirmation analysis is to confirm the presence of Aroclors tentatively identified in the primary analysis.

12.8.1.1 All instrument performance quality control criteria for calibration and retention time must be satisfied on the confirmation analysis.

12.8.2 Each tentative identification must be confirmed: using a second GC column of dissimilar stationary phase (as in the dual-column analysis), based on a clearly identifiable Aroclor pattern, or using another technique such as GC/MS.

12.8.2.1 The primary and secondary analysis is conducted simultaneously in the dual-column analysis.

12.8.2.2 GC/MS confirmation may be used in conjunction with dual-column analysis if the concentration is sufficient for detection in GC/MS, normally a concentration of approximately 10 ng/µl in the final extract for each Aroclor is required. Method 8270 is recommended as a confirmation technique when sensitivity permits.

12.8.3 Once the identification has been confirmed, the agreement between the quantitative results on both columns should be checked.

12.9 Sample Dilution

12.9.1 Establish dilution of sample in order to fall within calibration range or to minimize the matrix interference.

- Utilize screen data (specific project only).

- Utilize acquired sample data.
- Utilize the history program or approval from client/project.
- Sample characteristics (appearance, odor).

12.9.2 If no lower dilution has been reported, the dilution factor chosen should keep the response of the largest peak for a target analyte in the upper half of the initial calibration range of the instrument.

12.9.3 Preparing Dilutions.

12.9.3.1 Prepare sample dilutions quantitatively. Dilute the sample extract with hexane using logical volume to volume ratios, i.e., 1:5, 1:10, 1:50, etc.

12.9.3.2 Syringe Dilutions - A calibrated 1ml syringe must be used to prepare dilutions. Gently shake to disperse the extract throughout the solvent prior to loading on the auto-sampler tray for further analysis.

12.9.3.3 Volumetric Flask Dilutions – Dilutions can also be made with a Class A volumetric flask. Measure appropriate sample extract volume in a calibrated syringe and bring to a final volume with dilution solvent in a Class A volumetric flask. Gently shake to disperse the extract throughout the solvent prior to loading on the auto-sampler tray for further analysis.

12.10 Data interpretation

12.10.1 Qualitative identification

12.10.1.1 Analyst shall identify the target analytes with competent knowledge interpreting retention time and/or chromatographic pattern by comparison of the sample to the standard of the suspected Aroclor. The criteria required for a positive identification are:

12.10.1.1.1 The quantitation of PCB residues as Aroclors is accomplished by comparison of the sample chromatogram to that of the most similar Aroclor standard. A choice must be made as to which Aroclor is most similar to that of the residue and whether that standard is truly representative of the PCBs in the sample.

12.10.1.1.2 The target analytes must elute within the daily absolute retention time window on both primary and confirmation column.

12.10.1.1.3 For PCBs, at least five major peaks are selected. The retention time window for each peak is determined from the initial calibration analysis. This identification of PCBs as Aroclors is based on agreement between the retention times of peaks in the sample chromatogram with the retention time windows established through the analysis of standards of multi-component target analytes. Tentative identification of an analyte occurs when a peak from a sample extract falls within the established retention time window for a specific target analyte.

12.10.1.1.4 Be aware of matrix interfering effects on peak shape and relative peak ratios which could distort the pattern. Interpretation of this phenomenon may require a highly experienced chromatographer or at least a second opinion.

12.10.2 Quantitative analysis

12.10.2.1 Once the Aroclor pattern has been identified, compare the responses of at least 3 major peaks in the single-point calibration standard for that Aroclor with the peaks observed in the sample extract. The amount of Aroclor is calculated using the individual calibration factor for each corresponding peak and the linear calibration established from the multi-point calibration of the 1016/1260 mixture. A concentration (see section 14.4) based on the integrated area/or height of each of the characteristic peaks is determined and then those resulting concentrations are averaged to provide the final result for the sample.

12.10.2.2 Weathering of PCBs in the environment and changes resulting from waste treatment processes may alter the PCBs to the point that the pattern of a specific Aroclor is no longer recognizable. The quantitation may then be performed by measuring the total area of the PCB pattern and quantitating on the basis of the Aroclor standard that is most similar to the sample. Any peaks that are not identifiable as PCBs on the basis of retention times should be subtracted from the total area. When quantitation is performed in this manner, the problems should be fully described for the data user and the specific procedures employed by the analyst should be thoroughly documented.

12.10.2.3 When sample results are confirmed using two dissimilar columns or with two dissimilar detectors, the agreement between the quantitative results must be evaluated after the identification has been confirmed. Calculate the relative percent difference (RPD) between the two results using the formula in Section 14.6. The lower result is reported.

12.10.2.3.1 A program to perform the RPD calculation had been developed and incorporated into ENVIROQUANT software. Whenever an analyte has a RPD exceeds 40% between the results on the primary and secondary columns, a "P" qualifier will be shown on the quantitation report next to its results.

12.10.2.3.2 If one result is significantly higher (e.g., >40%), check the chromatograms to see if an obviously overlapping peak is causing an erroneously high result. If no overlapping peaks are noted, examine the baseline parameters established by the instrument data system (or operator) during peak integration.

12.10.2.3.3 If no anomalies are noted, review the chromatographic conditions. If there is no evidence of chromatographic problems, report the

lower result with the footnote (remark) indicating "More than 40% RPD for detected concentrations between two GC columns".

13.0 QUALITY CONTROL

13.1 QC Requirements Summary

Initial Calibration	Whenever needed
Initial Calibration Verification (ICV)	Following initial calibration
Continuing Calibration Verifications (CCV)	Every 12-hour shift , after every 10 samples and at the end of analysis sequence
Method blank	One per extraction batch*
Blank Spike	one per extraction batch*
Matrix Spike	one per extraction batch*
Matrix Spike Duplicate	one per extraction batch*
Surrogates	every sample and standard

*The maximum number of samples per batch is twenty or per project specification.

13.2 Initial Calibration.

13.2.1 Refer to Section 10.1.

13.3 Initial Calibration Verification (ICV) -Second Source Calibration Check.

13.3.1 Refer to Section 10.2.

13.4 Continuing Calibration Verifications (CCV)

13.4.1 Refer to Section 10.3.

13.5 Method Blank.

13.5.1 The method blank is either DI water or sodium sulfate (depending upon the sample matrix) which must be extracted with each set of 20 or less samples. For a running batch, a new method blank is required for each different extraction day. The method blank should be carried through all stages of the sample preparation and measurement.

13.5.2 If the method blank contains a target analyte above its MDL (Appendix I), the entire batch must be re-extracted and reanalyzed.

13.5.3 Surrogate compounds are added to the method blank prior to extraction and analysis. If the surrogate accuracy in the blank does not meet criteria (Appendix I), the entire batch must be re-extracted and reanalyzed.

13.6 Blank Spike.

13.6.1 A blank spike must be extracted with each set of 20 or less samples. For a running batch, a new blank spike is required for each different day. The blank spike consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. It is spiked with the same analyte at the same concentration as matrix spike. When the presence of specific Aroclors is not anticipated, the Aroclor 1016/1260 mixture may be appropriate choice for spiking. In situations where the other Aroclors are of interest for a specific project, the analyst may employ different spiking mixtures. The blank spike is prepared at a concentration of 2 µg/l or 66.7 µg/kg (on a dry weight basis) for each Aroclor.

13.6.2 The blank spike recoveries should be assessed using in house limits specified in Appendix I.

13.6.3 If a blank spike is out of control, the following corrective actions must be taken. In the case where the blank spike recovery is high and no hits reported in associated samples and QC batch the sample results can be reported with footnote (remark) and no further action is required.

13.6.3.1 Check to be sure that there are no errors in the calculations, or spike solutions. If errors are found, recalculate the data accordingly.

13.6.3.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample batch.

13.6.3.3 If no problem is found, re-extract and reanalyze the sample batch.

13.7 Matrix Spike (MS) / Matrix Spike Duplicate (MSD).

13.7.1 One sample is randomly selected from each extraction batch and spiked in duplicate with select Aroclors to assess the performance of the method as applied to a particular matrix and to provide information on the homogeneity of the matrix. Both the MS and MSD are carried through the complete sample preparation, cleanup, and determinative procedures.

13.7.2 The MS and MSD should be spiked with the Aroclors of interest. If samples are not expected to contain target analytes, a matrix spike and matrix spike duplicate pair should be spiked with Aroclor 1016/1260 mixture. However, when specific Aroclors are known to be present or expected in samples, the specific Aroclor should be used for spiking.

13.7.3 Matrix spikes are prepared by spiking an actual sample at a concentration 2 µg/l or 66.7µg/kg on a dry weight basis.

13.7.4 Assess the matrix spike recoveries and relative percent difference (RPD) against the control limits in Appendix I.

13.7.5 If the matrix spike accuracy of any individual Aroclor is out of control, the accuracy for that Aroclor in the blank spike must be within control. Matrix interference is assumed and the data is reportable. No further corrective action is required.

13.8 Surrogates.

13.8.1 Tetrachloro-m-xylene (TCMX) and Decachlrobiphenyl (DCB) are used as surrogate standards. All blanks, samples, matrix spikes, and calibration standards contain surrogate compounds which are used to monitor performance of the extraction, cleanup (when used), and analytical system.

13.8.2 The recoveries (Section 14.5) of the surrogates must be evaluated versus the surrogate control limits (Appendix I) developed by the laboratory annually.

13.8.3 If surrogate recoveries are not within established control limits, corrective action must be performed if surrogate recoveries indicate that a procedural error may have occurred during the analysis of the sample.

13.8.3.1 Check the surrogate calculations for calculation or integration errors and perform corrections if detected.

13.8.3.2 Reanalyze the extract if no calculation errors are detected. If the surrogate recoveries for the reanalyzed extract are in control, report the data from the reanalysis only.

13.8.3.3 If the data from the reanalysis is also out of control, re-extract and reanalyze the sample.

13.8.3.4 If, upon reanalysis, the surrogate recoveries are acceptable, report the reanalysis data. If the holding time has expired prior to the reanalysis, report both the original and reanalysis results and note the holding time problem.

13.8.3.5 If the recovery is again not within limits, the problem is considered to be matrix interference. Submit both data sets with the original analysis being reported.

13.8.4 The retention time shift for surrogate must be evaluated after the analysis of each sample. The sample must be reanalyzed when the retention times for both surrogates are outside the retention time window.

13.8.4.1 Reanalyses are not required for samples having visible matrix interference, defined as excessive signal levels from target or non-target interfering peaks. This judgment should be approved by a team leader or supervisor.

14.0 CALCULATION

14.1 Calibration Factor (CF).

$$CF = \frac{A_s}{C_s}$$

where:
A_s = Area of the peak for the compound being measured.
C_s = Concentration of the compound being measured (µg/l).

14.2 Percent Relative Standard Deviation (% RSD).

$$\%RSD = \frac{SD}{CF_{av}} \times 100$$

where:
SD = Standard Deviation.
CF_{av} = Average calibration factor from initial calibration.

14.3 Percent Difference (% D).

$$\% D = \frac{|CF_{av} - CF_c|}{CF_{av}} \times 100$$

where:
CF_c = CF from continuing calibration (CBCHK).

14.4 Concentration (Conc.).

<u>For water:</u>

$$\text{Conc. } (\mu g/l) = \frac{A_c \times M}{CF_{av}}$$

$$M = \frac{V_f \times D}{V_i}$$

<u>For soil/sediment</u> (on a dry weight basis):

$$\text{Conc. } (\mu g/kg) = \frac{A_c \times M}{CF_{av}}$$

$$M = \frac{V_f \times D}{W_s \times S}$$

where:
A_c = Area of peak for compound being measured.
V_f = Final Volume of total extract (ml).
D = Secondary dilution factor.
V_i = Initial volume of water extracted (ml).
W_s = Weight of sample extracted (g).
S = (100 - % moisture in sample) / 100 or % solid/100.
M = Multiplier.

14.5 Percent Recovery (% R).

$$\% R = \frac{\text{Concentration found}}{\text{Concentration spiked}} \times 100$$

14.6 Relative Percent Difference (RPD).

$$RPD = \frac{|C_1 - C_2|}{(1/2)(C_1 + C_2)} \times 100$$

where:
C_1 = Matrix Spike Concentration or the result on column 1.
C_2 = Matrix Spike Duplicate Concentration or the result on column 2.

15.0 DOCUMENTATION

15.1 The Analytical Logbook is a record of the analysis sequence; the logbook must be completed daily. Each instrument will have a separate logbook.

15.1.1 If samples require reanalysis, a brief explanation of the reason must be documented in this log. For consistency, if surrogates are high or low indicate it as (↑) for high and (↓) for low.

15.2 The Standard Preparation Logbook must be completed for all standard preparations. All information requested must be completed, the page must be signed and dated by the respective person.

15.2.1 The Accutest Lot Number must be cross-reference on the standard vial.

15.3 The Instrument Maintenance Logbook must be completed when any type of maintenance is performed on the instrument. Each instrument will have a separate log.

15.4 Any corrections to laboratory data must be done using a single line through the error. The initials of the person and date of correction must appear next to the correction.

15.5 Unused blocks of any form must be x'ed and z'ed by the analyst before submitting the data for review.

15.6 Supervisory (or peer) personnel must routinely review (at least once per month) all laboratory logbooks to ensure that information is being recorded properly. Additionally, the

maintenance of the logbooks and the accuracy of the recorded information should also be verified during this review.

16.0 DATA REVIEW AND REPORTING

16.1 Initial and continuing calibration check. Verify that all calibration and continuing calibration criteria have been achieved. If the criteria had not been achieved, corrective action must be performed to bring the system in control before analyzing any samples.

16.1.1 If samples had been analyzed under non-compliant calibration criteria, all sample extracts must be re-analyzed once the system is brought into control.

16.2 Quality Control Data Review. Review all QC data. If QC criteria were not achieved, perform corrective action before proceeding with analysis.

16.2.1 In some situation, corrective action may demand that the entire sample batch be re-extracted and re-analyzed before processing data.

16.3 Chromatogram Review. The chromatogram of each sample is evaluated for target analytes.

16.3.1 Check specific retention time windows for each target compound for the presence of the target compound in each chromatogram.

16.3.1.1 Each sample may require the reporting of different target analytes. Review the login to assure that the correct target compounds are identified.

16.3.2 The Aroclor must be identified on the primary and confirmatory column before assigning a qualitative identification.

16.3.3 Manual integration of chromatographic peaks must be identified by the analysts by initialing and dating the changes made to the report.

16.4 Transfer to LIMS. Following the initial screen review, transfer the processed data to the LIMS.

16.4.1 Print the processed data and compare the printed values to the original values to verify transfer accuracy.

16.4.2 If transfer errors occurred, the errors must be corrected before the data is re-submitted.

16.5 Hardcopy Print & Data Package Assembly.

16.5.1 After successful transfer is verified, approve the data and print a hard copy.

16.5.2 Assemble the data package combining the LIMS output and instrumental data.

16.5.3 Pass the entire package forward to the supervisor for final review and release approval.

17.0 POLLUTION PREVENTION & WASTE MANAGEMENT

17.1 Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 17.2.

17.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the waste management SOP, EHS004. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

17.2.1 Non hazardous aqueous wastes.

17.2.2 Hazardous aqueous wastes

17.2.3 Chlorinated organic solvents

17.2.4 Non-chlorinated organic solvents

17.2.5 Hazardous solid wastes

17.2.6 Non-hazardous solid wastes

18.0 ADDITIONAL REFERENCES

18.1 No additional references are required for this SOP.

Table 1. PCB Aroclors and Reporting Limits				
Compound	**CAS Number**	**Water (µg/l)**	**Soil (µg/kg)**	**Oil (µg/kg)**
Arochlor – 1016	12674-11-2	0.5	17	2000
Arochlor – 1221	11104-28-2	0.5	17	2000
Arochlor – 1232	11141-16-5	0.5	17	2000
Arochlor – 1242	53469-21-9	0.5	17	2000
Arochlor – 1248	12672-29-6	0.5	17	2000
Arochlor – 1254	11097-69-1	0.5	17	2000
Arochlor – 1260	11096-82-5	0.5	17	2000

Table 2. RECOMMENDED OPERATING CONDITION	
Gas Chromatograph/Electron Capture Detectors	
Carrier Gas	Helium
Make-up gas	5 % Methane/ 95 % Argon
Make-up gas flow	*30 ml/min
Injection port temperature	*235 °C
Injection type	Splitless
Detector temperature	*320 °C
Column flow	*5 ml/min
Gas Chromatograph Temperature Program*	
Initial temperature	*170 °C
Time 1	*2 min
Column temperature rate 1	*30 degrees/min
Temperature 1	*180 °C
Column temperature rate 2	*3.5 degrees/min
Temperature 2	*240 °C
Column temperature rate 3	*10 degrees/min
Final temperature	*280 °C
Time 3	*5 min
Total run time	30-40 min

* Parameter modification allowed for performance optimization as long as QC criteria are achieved.

Table 3A. Aroclors 1016/1260 Mixture and Surrogates Working Solution	
Stock Solution	**Volume Added**
Aroclor 1016 (1,000 µg/ml)	500 µl
Aroclor 1260 (1,000 µg/ml)	500 µl
Pesticides Surrogate Std Spiking Solution (200 µg/ml)	100 µl
Hexane	23.9 ml
Total	25.0 ml

- Aroclors 1016/1260 (20 µg/ml) and Surrogates (0.8 µg/ml) Working Solution: Prepared by measuring 500 µl of 1,000 µg/ml Aroclor 1016, 500µl of 1,000 µg/ml Aroclor 1260, 100 µl of 200 µg/ml pesticides surrogate std spiking solution and bringing to 25 ml with hexane.

Table 3B. Individual Aroclor* and Surrogates Working Solution	
Stock Solution	Volume Added
Individual Aroclor* (1,000 µg/ml)	500 µl
Pesticides Surrogate Std Spiking Solution (200 µg/ml)	100 µl
Hexane	24.4 ml
Total	25 ml

*Aroclor: 1221, 1232, 1242, 1248, or 1254

- Individual Aroclor (20 µg/ml) and Surrogates (0.8 µg/ml) Working Solution: Prepared by measuring 500 µl of 1,000 µg/ml each individual Aroclor, 100 µl of 200 µg/ml pesticides surrogate std spiking solution and bringing to 10 ml with hexane.

Table 4A. Aroclors 1016/1260 Calibration Standard Solutions					
Standard	Working Solution	Concentration (µg/ml)	Volume Added (µl)	Final Volume in Hexane (ml)	Final Concentration(µg/l)
Standard A	Aroclors 1016/1260	20	62.5	25	50
	Surrogates	0.8			2
Standard B	Aroclors 1016/1260	20	312.5	25	250
	Surrogates	0.8			10
Standard C	Aroclors 1016/1260	20	625	25	500
	Surrogates	0.8			20
Standard D	Aroclors 1016/1260	20	1250	25	1,000
	Surrogates	0.8			40
Standard E	Aroclors 1016/1260	20	2,500	25	2,000
	Surrogates	0.8			80
Standard F	Aroclors 1016/1260	20	3,750	25	3,000
	Surrogates	0.8			120

- Standard A: Prepared by measuring 62.5 µl of Aroclors 1016/1260 Mixture and Surrogates Working Solution (Table 3A), and bringing to 25 ml with hexane.

- Standard B: Prepared by measuring 312.5 µl of Aroclors 1016/1260 Mixture and Surrogates Working Solution (Table 3A), and bringing to 25 ml with hexane.

- Standard C: Prepared by measuring 625 µl of Aroclors 1016/1260 Mixture and Surrogates Working Solution (Table 3A), and bringing to 25 ml with hexane.

- Standard D: Prepared by measuring 1,250 µl of Aroclors 1016/1260 Mixture and Surrogates Working Solution (Table 3A), and bringing to 25 ml with hexane.

- Standard E: Prepared by measuring 2,500 µl of Aroclors 1016/1260 Mixture and Surrogates Working Solution (Table 3A), and bringing to 25 ml with hexane.

- Standard F: Prepared by measuring 3,750 µl of Aroclors 1016/1260 Mixture and Surrogates Working Solution (Table 3A), and bringing to 25 ml with hexane.

Table 4B. Single-Point Calibration Standard (1,000 µg/l) for Individual Aroclor*	
Stock Solution	**Volume Added**
Individual Aroclor*/Surrogate Working Solution (20 µg/ml/0.80µg/ml) (Table 3B)	1,250 µl
Hexane	23.75 ml
Total	25 ml

* Aroclor: 1221, 1232, 1242, 1248, or 1254.

- Individual Aroclor Calibration Standard (1,000 µg/l) and Surrogates (40 µg/l) Solution: Prepared by measuring 1,250 µl of individual Aroclor and surrogates working solution, containing 20 µg/ml of each corresponding Aroclor and 0.80 µg/ml of both surrogate compounds, and bringing to 25 ml with hexane.

Table 4C. Multi-point Calibration Standards for Individual Aroclor* (optional)					
Standard	**Stock Solution**	**Concentration (µg/ml)**	**Volume Added (µl)**	**Final Volume In Hexane (ml)**	**Final Concentration(µg/l)**
Standard A	Aroclor*	20	62.5	25	50
	Surrogates	0.8			2
Standard B	Aroclor*	20	312.5	25	250
	Surrogates	0.8			10
Standard C	Aroclor*	20	625	25	500
	Surrogates	0.8			20
Standard D	Aroclor*	20	1250	25	1,000
	Surrogates	0.8			40
Standard E	Aroclor*	20	2,500	25	2,000
	Surrogates	0.8			80
Standard F	Aroclor*	20	3,750	25	3,000
	Surrogates	0.8			120

*Aroclor: 1221, 1232, 1242, 1248, or 1254.

- Standard A: Prepared by measuring 62.5 µl of Individual Aroclor and Surrogates Working Solution (Table 3B), and bringing to 25 ml with hexane.

- Standard B: Prepared by measuring 312.5 µl of Individual Aroclor and Surrogates Working Solution (Table 3B), and bringing to 25 ml with hexane.

- Standard C: Prepared by measuring 625 µl of Individual Aroclor and Surrogates Working Solution (Table 3B), and bringing to 25 ml with hexane.

- Standard D: Prepared by measuring 1,250 µl of Individual Aroclor and Surrogates Working Solution (Table 3B), and bringing to 25 ml with hexane.

- Standard E: Prepared by measuring 2,500 µl of Individual Aroclor and Surrogates Working Solution (Table 3B), and bringing to 25 ml with hexane.

- Standard F: Prepared by measuring 3,750 µl of Individual Aroclor and Surrogates Working Solution (Table 3B), and bringing to 25 ml with hexane.

Table 5A. Continuing Calibration Check Solutions for Aroclors 1016/1260					
Checks	**Working Solution**	**Concentration (µg/ml)**	**Volume Added (µl)**	**Final Volume in Hexane (ml)**	**Final Concentration (µg/l)**
Solution 1	Aroclors 1016/1260	20	625	25	500
	Surrogates	0.8			20
Solution 2	Aroclors 1016/1260	20	1250	25	1,000
	Surrogates	0.8			40

- Solution 1: Prepared by measuring 625 µl of Aroclors 1016/1260 Mixture and Surrogates Working Solution (Table 3A), and bringing to 25 ml with hexane.

- Solution 2: Prepared by measuring 1,250 µl of Aroclors 1016/1260 Mixture and Surrogates Working Solution (Table 3A), and bringing to 25 ml with hexane.

Table 5B. Continuing Calibration Check Solutions for Individual Aroclor*					
Checks	**Working Solution**	**Concentration (µg/ml)**	**Volume Added (µl)**	**Final Volume in Hexane (ml)**	**Final Concentration (µg/l)**
Solution 1	Aroclor*	20	625	25	500
	Surrogates	0.8			20
Solution 2	Aroclor*	20	1250	25	1,000
	Surrogates	0.8			40

* Aroclor: 1221, 1232, 1242, 1248, or 1254

- Solution 1: Prepared by measuring 625 µl of Individual Aroclor and Surrogates Working Solution (Table 3B), and bringing to 25 ml with hexane.

- Solution 2: Prepared by measuring 1,250 µl of Individual Aroclor and Surrogates Working Solution (Table 3B), and bringing to 25 ml with hexane.

Table 6A. Second Source Calibration Check Standard for Aroclors 1016/1260 (1,000 µg/l)	
Stock Solution	**Volume Added**
Aroclors 1016/1260 (25 µg/ml) and Surrogates (2.5 µg/ml) Working Solution	1,000 µl
Hexane	24 ml
Total	25 ml

- Aroclors 1016/1260 (25 µg/ml) and Surrogates (2.5 µg/ml) Working Solution: Prepared by measuring 250 µl of 1,000 µg/ml Aroclors 1016/1260 mix solution (2nd source), 125 µl of 200 µg/ml pesticides surrogate std spiking solution and bringing to 10 ml with hexane.

- Aroclors 1016/1260 (1,000 µg/l) and Surrogates (100 µg/l) Solution: Prepared by measuring 1,000 µl of Aroclors 1016/1260 (25 µg/ml) and surrogates (2.5 µg/ml) working solution and bringing to 25 ml with hexane.

Table 6B. Second Source Calibration Check Standard for Individual Aroclor* (1,000 µg/l)	
Stock Solution	**Volume Added**
Individual Aroclor* (25 µg/ml) and Surrogates (2.5 µg/ml) Working Solution	1,000 µl
Hexane	24 ml
Total	25 ml

*Aroclor: 1221, 1232, 1242, 1248, or 1254.

- Individual Aroclor (25 µg/ml) and Surrogates (2.5 µg/ml) Working Solution: Prepared by measuring 250 µl of 1,000 µg/ml each individual Aroclor stock solution (2nd source), 125 µl of 200 µg/ml pesticides surrogate std spiking solution and bringing to 10 ml with hexane.

- Individual Aroclor (1,000 µg/l) and Surrogates (100 µg/l) Solution: Prepared by measuring 1,000 µl of each individual Aroclor (25 µg/ml) and surrogates (2.5 µg/ml) working solution and bringing to 25 ml with hexane.

ACCUTEST LABS
STANDARD OPERATING PROCEDURE

Lab Manager_____

QA Manager_____

Effective Date: _____

TEST NAME: ALKALINITY, TOTAL (pH 4.5)

METHOD REFERENCE: Method 2320B from the 20th Edition of the Standard Methods for the Examination of Water and Wastewater, and EPA Method 310.1. Soils by a modification of EPA 310.1

Revised Sections : *13.2*

1.0 SCOPE AND APPLICATION

1.1 Alkalinity is the measure of the acid-neutralizing capacity of a water sample, as the sum of all titratable bases. Since the alkalinity of surface waters is primarily a function of carbonate, bicarbonate, and hydroxide content, it may be used as an indicator for the concentration of these constituents. The measured value will also include some borates, phosphates, silicates and other bases if they are present. The sample is not to be diluted, filtered, or altered in any way. This method is applicable for all water samples.

1.2 For samples of low alkalinity (<20 mg $CaCO_3$/l) an extrapolation technique is used. The amount of acid to lower the pH exactly 0.3 pH units is measured, after the initial endpoint has been attained. Because this corresponds to an exact doubling of the hydrogen ion concentration, an extrapolation may be made to the equivalence point.

1.3 Phenolpthalein alkalinity can be done following the same method using an endpoint of 8.3.

1.4 The product used in the LIMS system for this test is ALK.

1.5 *The alkalinity of a neutral extract of a soil sample may also be analyzed following a modification* of this method.

2.0 SUMMARY OF METHOD

2.1 An unaltered sample is titrated to an electrometrically determined end point of pH 4.5. The sample must not be filtered, diluted, concentrated, or altered in any way.

3.0 REPORTING LIMIT AND METHOD DETECTION LIMIT

3.1 Reporting Limit. The reporting limit for this method has been established at 5.0 mg/l.

3.2 Method Detection Limit. Experimentally determine MDLs using the procedure specified in 40 CFR, Part 136, Appendix B. This value represents the lowest reportable concentration of an individual compound that meets the method qualitative identification criteria.

3.2.1 Experimental MDLs must be determined annually for this method.

3.2.2 Process all raw data for the replicate analysis in each MDL study

4.0 DEFINITIONS

BATCH: A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

CALIBRATION CHECK STANDARD. The calibration check standard is a mid-range calibration standard. It is recommended that the calibration check standard be run at a frequency of approximately 10 percent. (For some methods this is mandatory and for some it is a recommendation only. Refer to individual method SOP's)

EXTERNAL CHECK STANDARD. The external check standard is a standard from a separate source than the calibration curve that is used to verify the accuracy of the calibration standards. An external check must be run a minimum of once per quarter for all analyses where a check is commercially available. The laboratory should initially assess laboratory performance of a check standard using the control limits generated by the external check supplier. In house limits should also be generated once sufficient external check standard data is available to generate limits (usually a minimum of 20 to 30 analyses). If the external check is outside of the control limits for a given parameter, all samples must be reanalyzed for that parameter after the problem has been resolved.

SPIKE BLANK OR LAB CONTROL SAMPLE. Digest and analyze a laboratory control sample or spike blank with each set of samples. A minimum of one lab control sample or spike blank is required for every 20 samples. Assess laboratory performance against the control limits specified in the SOP. In house limits should also be generated once sufficient external check standard data is available to generate limits (usually a minimum of 20 to 30 analyses). If the lab control is outside of the control limits for a parameter, all samples must be redigested or redistilled and reanalyzed for that parameter. The exception is if the lab control recovery is high and the results of the samples to be reported are less than the reporting limit. In that case, the sample results can be reported with no flag. Note: If control limits are not specified in the SOP, then default limits of 80 to 120 percent should be used.

MATRIX: The component or substrate (e.g., water, soil) which contains the analyte of interest.

MATRIX DUPLICATE: A duplicate sample is digested at a minimum of 1 in 20 samples. The relative percent difference (RPD) between the duplicate and the sample should be assessed. The duplicate RPD is calculated as shown below. Assess laboratory performance against the control limits that are specified in the SOP. In house limits are generated once sufficient duplicate data is

available to generate limits (usually a minimum of 20 to 30 analyses). If a duplicate is out of control, flag the results with the appropriate footnote. If the sample and the duplicate are less than 5 times the reporting limits and are within a range of ± the reporting limit, then the duplicate is considered to be in control. Note: If control limits are not specified in the SOP, use default limits of ± 20% RPD.

$$\frac{(|\text{Sample Result} - \text{Duplicate Result}|) \times 100}{(\text{Sample Result} + \text{Duplicate Result})/2} = \text{Duplicate RPD}$$

METHOD BLANK. The laboratory must digest and analyze a method blank with each set of samples. A minimum of one method blank is required for every 20 samples. For a running batch, a new method blank is required for each different digestion day. If no digestion step is required, then the method blank is equivalent to the reagent blank. The method blank must contain the parameter of interest at levels of less that the reporting limit for that parameter. If the method blank contains levels over the reporting limits, the samples must be redigested or redistilled and reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the method blank level. In addition, if all the samples are less than a client required limit and the method blank is also less than that limit, then the results can be reported as less than that limit.

METHOD DETECTION LIMITS (MDLS). The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. MDLs should be determined approximately once per year for frequently analyzed parameters.

REAGENT BLANK: The reagent blank is a blank that has the same matrix as the samples, i.e., all added reagents, but did not go through sample preparation procedures. The reagent blank is an indicator for contamination introduced during the analytical procedure. (Note: for methods requiring no preparation step, the reagent blank is equivalent to the method blank.) Either a reagent blank or a method blank must be analyzed with each batch of 20 samples or less. The concentration of the analyte of interest in the reagent blank must be less than the reporting limit for that analyte. If the reagent blank contains levels over the reporting limits, the samples must be reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the reagent blank level. In addition, if all the samples are less than a client required limit and the reagent blank is also less than that limit, then the results can be reported as less than that limit.

REAGENT GRADE: Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents which conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.

REAGENT WATER: Water that has been generated by any method which would achieve the performance specifications for ASTM Type II water. For organic analyses, see the definition of organic-free reagent water.

REFERENCE MATERIAL: A material containing known quantities of target analytes in solution or in a homogeneous matrix. It is used to document the bias of the analytical process.

5.0 HEALTH & SAFETY

5.1 The analyst must follow normal safety procedures as outlined in the Accutest Laboratory Safety Manual which includes the use of safety glasses and lab coats. In addition, all acids are corrosive and must be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical must be treated as a potential health hazard. Exposure to these reagents must be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets must be made available to all personnel involved in these analyses.

6.0 PRESERVATION & HOLDING TIME

6.1 The samples must be stored in a refrigerator at 4^0 C.

6.2 Samples must be analyzed within 14 days of sample collection. Note: If Alkalinity is being done to calculate CO2, then it must be done immediately after collection or as soon as possible (with 24 hours) after receipt at the lab.

7.0 INTERFERENCES

7.1 Samples that contain oil, soaps or suspended solids may coat the electrode and cause a sluggish electrode response. Allow additional time between measurements to reach equilibrium at the electrode.

8.0 APPARATUS

8.1 pH meter with glass electrode capable of reading 0.05 pH unit.

8.2 Titration vessel, 100 ml or 200 ml beaker.

8.3 Magnetic stirrer; stirbars.

8.4 Pipettes, class A.

8.5 Volumetric flasks, class A.

8.6 Burets, 50 m and 10 ml micro.

9.0 REAGENTS

9.1 Sodium Carbonate solution; approximately 0.05 N:Place 2.5 + or - 0.2 g (weigh to nearest 0.1

mg) of sodium carbonate (Na_2CO_3, dried at 250° C for 4 hours and cooled in a desiccator) into a 1 liter flask and dilute to the mark. Do not store for more than 1 week.

9.2 Standard Acid, 0.1 N. This solution must be standardized a minimum of once per quarter.

9.2.1 Sulfuric acid: Add 3.0 ml of concentrated H_2SO_4 to 500 ml of DI water in a 1 liter volumetric flask. Dilute to 1 liter with water, or

9.2.2 Hydrochloric Acid: Add 8.3 ml of concentrated HCl to 500 ml DI water in a 1 liter volumetric Flask. Dilute to 1 liter with water.

9.3 Standard Acid 0.02 N: Dilute 200.0 ml of 0.1 N Standard Acid (use 9.2.1 or 9.2.2) to 1 liter with distilled water. This solution must be standardized a minimum of once per quarter.

9.4 Sodium Phosphate, dibasic: 0.10 M Weigh 26.81 g of sodium phosphate dibasic heptahydrate ($Na_2HPO_4 \cdot 7H_2O$). Dissolve in approximately 600 ml of DI water in a 1000 ml volumetric flask. Dilute to 1 liter. Alkalinity value is 5000 mg/l as $CaCO_3$.

9.5 Sodium Phosphate dibasic, 0.005 m: Dilute 5 ml of 0.10 m sodium phosphate to 100 ml. Alkalinity value is 250 mg/l as $CaCO_3$.

10.0 SOIL SAMPLE PREPARATION PROCEDURE

10.1 For soil samples, weigh out 20 g of sample and, using a class A graduated cylinder, add 200 ml of DI water. Stir on a magnetic stirring plate for ten minutes.

10.1.1 For the method blank, measure out 200 ml of deionized water and stir on a magnetic stirring plate for ten minutes.

10.1.2 For the spike blank, use 200 ml of the sodium phosphate dibasic, 0.005 m solution described in section 9.5. Stir on a magnetic stirring plate for ten minutes.

10.2 Filter the resulting slurry through a Whatman GFB or GFF filter (or equivalent). Note: All QC must be stirred and filtered in the same method as the samples. Analyze for Alkalinity following section 11.0

11.0 PROCEDURE

11.1 If the acid solution has not been standardized within the last 3 months, then it must be restandardized before starting the analysis. The procedure for standardizing the 0.10 N and 0.02 N acid solutions is shown below.

11.1.1 Calibrate the pH meter according to manufacturer's instructions, and document the calibration in the pH meter logbook and on the alkalinity worksheet. Read back the 4, 7, and 10 buffers. If the results are not within 0.05 pH units, then the meter must be recalibrated before proceeding.

11.1.2 Rinse the 50 ml buret with approximately 20 ml of standard acid, then fill it and adjust

volume to 0.0.

11.1.3 If standardizing the 0.10 N acid solution, add 40.0 ml of 0.05N Sodium Carbonate Solution to about 60 ml of distilled water in a 250 ml baker. If standardizing the 0.02 N acid solution, add 15.0 ml of 0.05N sodium carbonate solution to about 60 ml of distilled water in a 250 ml baker.

11.1.4 Add a stir bar and place on a magnetic stirrer. Mix gently.

11.1.5 Place pH probe in the sodium carbonate solution and titrate to a pH of approximately 5. Record the pH after every 0.5 ml acid addition. Remove electrode and rinse in beaker of sodium carbonate.

11.1.6 Cover the beaker with a watch glass and boil the solution for 3- 5 minutes. Allow to cool.

11.1.7 Continue the titration to the pH inflection point and record the pH after every 0.2 ml addition. Continue the titration to pH 3.0.

11.1.8 Plot the pH of the sodium carbonate vs. ml of acid added.

11.1.9 Calculate the normality of the acid as follows;

Normality N =

$$\frac{A \times B}{53.00 \times C}$$

Where:
A = g Na_2CO_3 weighed
B = ml Na_2CO_3 solution
C = ml Acid used at inflection point.

11.2 Procedure for analysis of samples with alkalinity values above 20 mg/l.

11.2.1 Place 100 ml of the sample in the titration vessel (beaker or flask), with minimal agitation. Place a stir bar in the titration vessel.

11.2.2 While stirring gently but thoroughly, measure the pH of the sample. Record the pH on the analysis log.

11.2.3 Slowly add 0.02 N standard acid allowing the pH meter to achieve equilibrium between additions.

11.2.4 Titrate the sample to a final pH of 4.5. Record the volume of acid used for the titration on the analysis log.

11.3 Procedure for the analysis of samples with alkalinity values below 20 mg/l (< 2.0 ml of 0.02 N

titrant)

11.3.1 Titrate the sample as described in 10.2.1 through 10.2.4, using a 10 ml microburette and 0.02 N titrant.

11.3.2 Stop the titration in the pH range of 4.3 to 4.7 and record the volume used.

11.3.3 Very carefully add titrant to lower the pH 0.3 pH units and again record the volume.

11.4 For samples with a concentration above 20 mg/l, use the following calculation to calculate the alkalinity.

$$\text{Alkalinity, mg/l } CaCO_3 = \frac{A \times N \times 50000}{\text{ml sample}}$$

Where: A = ml of standard acid
N = Normality of standard acid

11.5 For Samples with a concentration below 20 mg/l, use the following calculation:

$$\text{Alkalinity, mg.l } CaCO_3 = \frac{(2A - C) \times N \times 50000}{\text{ml sample}}$$

Where: A = ml titrant to first pH
C = total ml titrant to reach pH 0.3 units lower
N = Normality of Acid.

11.6 After every 10 analyses, check the pH meter with the 4.0 pH buffer. The buffer must be within 0.20 pH units of 4.00.

11.6.1 If the check is outside of the 0.20 pH unit criteria, check the probe for problems. If the problems are unrelated to the sample matrix, then resolve the problems and reanalyze the samples and the check standards.

11.6.2 If the 0.20 pH unit criteria cannot be met due to difficulties with the probe caused by the sample matrix, see the area manager or supervisor. Sample results may be reported with a footnote noting the recovery obtained on the check standard.

12.0 QUALITY CONTROL

12.1 The 4, 7, and 10 buffer solutions must be checked prior to the analysis of the samples. The results of these buffer checks must be within 0.05 pH units of the true values. If they are not, then recalibrate the pH meter before proceeding.

12.2 An external check must be analyzed at least once per quarter or whenever the primary standard lot changes. The results of the external check must be within manufacturer's limits, or, if no limits are supplied, within 10%.

12.3 A buffer check standard must be analyzed after approximately every 10 samples. This check must be within 0.2 pH units of the true value. Refer to section 10.6 for the appropriate action to take if the check is outside of these limits.

12.4 When analyzing samples, a duplicate sample must be analyzed with each group of 20 samples. This quality control point should be evaluated using Accutest's in house quality control limits. Note: if the in house limits are not available, then the default limit of 20 rpd should be used.

12.5 A method blank and a spike blank must be analyzed with each batch of 20 samples or daily, whichever is more frequent. The method blank results must be < RDL and the spike blank must have a recovery in the range of 80 to120 % recovery. If this criteria is not met, then the samples must be reanalyzed.

13.0 DOCUMENTATION REQUIREMENTS

13.1 The analyst must document all relevant information, including, but not limited to, all sample volumes, all initial and final pH values, and any relevant comments for any section of the analysis Make sure to record any comments regarding unusual sample appearance or any other problems or observations. All reagent and standard information, including the manufacture, lot, and expiration date must be recorded in a reagent log. In addition, the pH meter that was used and the buffer solutions used must be documented. All work must be dated and signed by the analyst. Any changes must be crossed out with a single line and must be initialed and dated and a reason for the change provided.

14.0 DATA REVIEW AND REPORTING

14.1 All samples must be updated to QC batches in the LIMS system. The analyst is responsible for reviewing all data for compliance with the QC outlined in this SOP. They are responsible for making sure that the raw data is fully documented and it is updated and entered in the LIMS system.

14.2 After the analyst review is completed, the supervisor or a designated reviewer must review the data for technical compliance to the SOP. After the reviews are completed, the data is electronically filed in the report generation department.

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT.

15.1 Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment must be followed. All method users must be familiar with the waste management practices

described in section 15.2.

15.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the waste management SOP, EHS004. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

15.2.1 Non hazardous aqueous wastes.

15.2.2 Hazardous aqueous wastes

15.2.3 Chlorinated organic solvents

15.2.4 Non-chlorinated organic solvents

15.2.5 Hazardous solid wastes

15.2.6 Non-hazardous solid wastes

15.0 ADDITIONAL REFERERENCES

15.1 Refer to pH meter manuals for operation of individual pH meters and for care of pH probes.

ACCUTEST LABORATORIES
STANDARD OPERATING PROCEDURE

Lab Manager_____

QA Manager_____

Effective Date:_____

TITLE: **HARDNESS AS $CACO_3$ BY TITRATION**

REFERENCES: **Method 2340C from "Standard Methods for the Examination of Water and Wastewater", 19th Edition.**

Revised Sections: ***10.4, 10.4.1, 12.3***

1.0 SCOPE AND APPLICATION

1.1 This method is used as a measure of the hardness in a sample and is applicable to all waters and wastewaters.

1.2 The product for hardness is HRD.

1.3 An alternate method for hardness, where the hardness value is calculated from calcium and magnesium values, is cited in the SOP EGN259.

2.0 SUMMARY

2.1 A measured aliquot of sample is buffered and then titrated with standardized EDTA (disodium ethylenediamine tetraacetate) titrant. The end point of the reaction is determined when the last reddish tint in the sample disappears. The indicator has a red color in the presence of calcium and magnesium ions and a blue color when these ions are sequestered.

3.0 REPORTING LIMIT AND METHOD DETECTION LIMIT

3.1 Reporting Limit. The reporting limit for this method has been established at 4.0 mg/l of hardness as calcium carbonate.

3.2 Method Detection Limit. Experimentally determine MDLs using the procedure specified in 40 CFR, Part 136, Appendix B. This value represents the lowest reportable concentration of an individual compound that meets the method qualitative identification criteria.

3.2.1 Experimental MDLs must be determined annually for this method.

4.0 DEFINITIONS

BATCH: A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of

samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

EXTERNAL CHECK STANDARD. The external check standard is a standard from a separate source than the calibration curve or the spike blank that is used to verify the accuracy of the method. An external check must be run a minimum of once per quarter for all analyses where a check is commercially available. The laboratory should initially assess laboratory performance of a check standard using the control limits generated by the external check supplier. In house limits should also be generated once sufficient external check standard data is available to generate limits (usually a minimum of 20 to 30 analyses). If the external check is outside of the control limits for a given parameter, all samples must be reanalyzed for that parameter after the problem has been resolved.

SPIKE BLANK OR LAB CONTROL SAMPLE. Digest and analyze a laboratory control sample or spike blank with each set of samples. A minimum of one lab control sample or spike blank is required for every 20 samples. Assess laboratory performance against the control limits specified in the SOP. In house limits should also be generated once sufficient external check standard data is available to generate limits (usually a minimum of 20 to 30 analyses). If the lab control is outside of the control limits for a parameter, all samples must be redigested or redistilled and reanalyzed for that parameter. The exception is if the lab control recovery is high and the results of the samples to be reported are less than the reporting limit. In that case, the sample results can be reported with no flag. Note: If control limits are not specified in the SOP, then default limits of 80 to 120 percent should be used.

MATRIX: The component or substrate (e.g., water, soil) which contains the analyte of interest.

MATRIX DUPLICATE: A duplicate sample is digested at a minimum of 1 in 20 samples. The relative percent difference (RPD) between the duplicate and the sample should be assessed. The duplicate RPD is calculated as shown below. Assess laboratory performance against the control limits that are specified in the SOP. In house limits are generated once sufficient duplicate data is available to generate limits (usually a minimum of 20 to 30 analyses). If a duplicate is out of control, flag the results with the appropriate footnote. If the sample and the duplicate are less than 5 times the reporting limits and are within a range of ± the reporting limit, then the duplicate is considered to be in control. Note: If control limits are not specified in the SOP, use default limits of ± 20% RPD.

$$\frac{(|\text{Sample Result} - \text{Duplicate Result}|) \times 100}{(\text{Sample Result} + \text{Duplicate Result})/2} = \text{Duplicate RPD}$$

MATRIX SPIKE: The laboratory must add a known amount of each analyte to a minimum of 1 in 20 samples. The matrix spike recovery is calculated as shown below. Assess laboratory performance against the control limits that are specified in the SOP. In house limits are generated once sufficient matrix spike data is available to generate limits (usually a minimum of 20 to 30 analyses). If a matrix spike is out of control, then the results should be flagged with the appropriate footnote. If the matrix spike amount is less than one fourth of the sample amount, then the sample cannot be assessed against the control limits and should be footnoted to that effect. Note: If control limits are not specified in the SOP, then default limits of 75 to 125 percent should be used.

$$\frac{(\text{Spiked Sample Result} - \text{Sample Result})}{(\text{Amount Spiked})} \times 100 = \text{Matrix Spike Recovery}$$

METHOD BLANK. The laboratory must digest and analyze a method blank with each set of samples. A minimum of one method blank is required for every 20 samples. For a running batch, a new method blank is required for each different digestion day. If no digestion step is required, then the method blank is equivalent to the reagent blank. The method blank must contain the parameter of interest at levels of less that the reporting limit for that parameter. If the method blank contains levels over the reporting limits, the samples must be redigested or redistilled and reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the method blank level. In addition, if all the samples are less than a client required limit and the method blank is also less than that limit, then the results can be reported as less than that limit.

METHOD DETECTION LIMITS (MDLS). MDLs should be established for all appropriate methods, using a solution spiked at approximately 3 times the estimated detection limit. To determine the MDL values, take seven replicate aliquots of the spiked sample and process through the entire analytical method. The MDL is calculated by multiplying the standard deviation of three replicate analyses by 3.14, which is the student's t value for a 99% confidence level. MDLs should be determined approximately once per year for frequently analyzed parameters.

REAGENT GRADE: Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents which conform to the current specifications of the Committee on *Analytical Reagents of the American Chemical Society*.

REAGENT WATER: Water that has been generated by any method which would achieve the performance specifications for ASTM Type II water. For organic analyses, see the definition of organic-free reagent water.

REFERENCE MATERIAL: A material containing known quantities of target analytes in solution or in a homogeneous matrix. It is used to document the bias of the analytical process.

5.0 HEALTH & SAFETY

5.1 The analyst should follow normal safety procedures as outlined in the Accutest Laboratory Safety Manual which includes the use of safety glasses and lab coats. In addition, all acids are corrosive and should be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these reagents should be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets should be made available to all personnel involved in these analyses.

6.0 COLLECTION, PRESERVATION, & HOLDING TIME

6.1 Water samples should be acidified to a pH of less than 2 by the addition of concentrated nitric acid.

6.2 All samples should be analyzed within 6 months of the date of collection.

7.0 APPARATUS & MATERIALS

7.1 25 ml microburet, class A.

7.2 Erlenmeyer flasks or disposable beakers

7.3 Stir plates and stir bars.

7.4 Analytical balance, 4 place. The balance calibration must be checked daily before use. If the calibration checks on the balance are not within limits as supplied in the balance logs, then the balance must be taken out of service until it can be recalibrated.

7.5 Narrow range pH paper to read between 10.0 and 10.1.

8.0 STANDARDS & REAGENTS.

8.1 All chemicals listed below are reagent grade unless otherwise specified. Distilled water should be used whenever water is required.

8.2 Buffer solution. Dissolve 1.179 g of analytical grade disodium EDTA (disodium ethylenediamine tetraacetate, $Na_2C_{10}H_{14}O_8N_2 \cdot 2H_2O$) and 0.780 g of $MgSO_4 \cdot 7H_2O$ (or 0.644 g of $MgCl_2 \cdot 6H_2O$) in 50 ml of DI water. Add this solution to a 250 ml volumetric flask containing 16.9 g of ammonium chloride (NH_4Cl) and 143 ml of concentrated ammonium hydroxide (NH_4OH) with mixing and dilute to the mark with DI water. Store in a plastic bottle for no longer than 1 month.

8.2.1 Anhydrous magnesium chloride may also be used in the above buffer solution as long as the weights are adjusted accordingly.

8.2.2 An alternate buffer solution may be prepared by dissolving 16.9 g of ammonium chloride (NH_4Cl) in 143 ml of concentrated ammonium hydroxide (NH4OH). Add 1.25 g of magnesium salt of EDTA (available commercially) and dilute to 250 ml with DI water. Mix well

8.2.3 Commercial buffer solutions are also available. Refer to the method for additional information on these buffer solutions.

8.2.4 One to 2 ml of buffer with the sample should give a pH of 10.0 to 10.1. If the buffer is not obtaining that pH on most samples, it may have deteriorated and fresh buffer should be made.

8.3 Inhibitor solutions. These are to be used only if interferences are evident during the titration. These inhibitor solutions help to give a clear sharp change in color at the end point. Check with the lab supervisor or manager before using any of these inhibitor solutions

8.3.1 Inhibitor I: NaCN powder. (Caution – extremely poisonous). Flush solutions of sample containing this solution down the drain using large quantities of water. Make

sure that there are no acids present that might liberate HCN. Always work in the hood when using this solution.

8.3.2 Inhibitor II: Dissolve 5.0g of $Na_2S \cdot 9H_2O$ in 100 ml of DI water. Cover container with a tightly fitted rubber stopper. This inhibitor deteriorates through air oxidation and can produce a sulfide precipitate that obscures the end point when appreciable concentrations of heavy metals are present.

8.3.3 Inhibitor III: MgCDTA (magnesium salt of 1,2-cylclohexanediamnetetraacetic acid). Add 0.250 g of MgCDTA to each 100 ml of sample before analysis and dissolve completely before adding the buffer solution.

8.4 Indicator. Solid calmagite (1-(1-hydroxy-4-methyl-2phenylazo)-2-maphthol-4-sulfonic acid or a calmagite indicator solution can be used.

8.4.1 Calmagite solution can be prepared by dissolving 0.10 g of Calmagite in 100 ml of DI water.

8.5 Standard EDTA titrant, 0.020 N (0.010 M). Place 3.723 g of analytical grade disodium EDTA in a 1 liter volumetric flask and dilute to the mark with DI water. Check with standard calcium solution by titration as described in the procedure section below. Store in a polyethylene bottle.

8.6 Standard Calcium solution (1000 mg/l). Place 1.000 g of anhydrous calcium carbonate in a 500 ml erlenmeyer flask. Slowly add 1+1 HCl until all of the calcium carbonate has dissolved. Add 200 ml of DI water and boil for several minutes to expel the carbon dioxide. Cool. Then add a few drops of methyl red indicator and adjust to an intermediate orange color by adding 3N ammonium hydroxide or 1 +1 HCl as required. Transfer quantitatively to a 1 liter volumetric flask and dilute to a final volume of 1 liter with DI water. Mix well.

8.7 Hydrochloric acid solution, 1+1. Add one part hydrochloric acid to one part water.

8.8 Methyl Red Indicator. Dissolve 0.10 g of methyl red in DI water in a 100 ml volumetric flask and dilute to the final volume with DI water and mix well. This may also be purchased commercially.

8.9 Ammonium Hydroxide solution, 3 N. Dilute 210 ml of concentrated ammonium hydroxide (NH_4OH) to 1 liter with DI water.

8.10 Ammonium hydroxide solution, 1N. Dilute 70 ml of concentrated ammonium hydroxide to 1 liter with DI water.

9.0 INTERFERENCES

9.1 Some metal ions interfere by causing fading or indistinct endpoints or by stoichiometric comsumption of EDTA. These interferences can be reduced by adding certain inhibitors before titration.

9.2 Conduct titrations at or near room temperature. Color change may be impractically slow at low temperatures.

9.3 Calcium carbonate precipitation may cause a drifting endpoint. Completion of the titration within 5 minutes minimizes the tendency for calcium carbonate to precipitate. Dilution can also minimize this problem. Alternatively, do a preliminary titration and add 90 percent of the expected titrate to the sample before adjusting the pH with the buffer. Then finish the titration.

10.0 PROCEDURE

10.1 Check the standardization of the EDTA solution and make sure that it has been standardized within the past month. If it has not been standardized within a month, or if it is a new lot of EDTA, standardize the solution following the procedure outlined below.

10.1.1 Place 10.0 ml of standard calcium solution in a container containing about 50 ml of DI water. Add 1 ml of the buffer solution and a few crystals of calgamite. Titrate slowly with continuous stirring with the EDTA until the last reddish tinge disappears. Add the last few drops at 3 to 5 second intervals. At the end point, the color should be blue. The total titration duration should be no more than 5 minutes from the time of the buffer addition.

10.1.2 Calculate the normality of the EDTA as shown below.

N of EDTA = (0.200)/(ml of EDTA added)

10.2 Start the titration of the samples and the quality control. A method blank and a spike blank must be analyzed at the beginning of every run and a second spike blank must be analyzed with every 10 additional samples. (For example, if you are running 20 samples, you should analyze a spike blank at the beginning of the run and after the first 10 samples.) A duplicate and a matrix spike must be analyzed with every 20 samples. For some clients and some states, additional QC may be required. (10% duplicates are required for New York samples.)

10.3 Measure 25.0 ml of sample or quality control into the titration vessel. Blank spikes and matrix spikes should be spiked with 4.00 ml of the standard calcium carbonate solution at this point. Neutralize the samples with 1 N ammonium hydroxide and dilute to a final volume of approximately 50 ml.

10.3.1 Highly polluted samples should first go through a metals digestion step before analysis. Check with the lab supervisor or manager if you feel the sample needs extra pretreatment.

10.4 Add 1 to 2 ml of buffer solution and mix. The pH of the samples at this point should be 10.0 to 10.1. Check the pH with narrow range pH paper and verify that it is within the range of 10.0 to 10.1. Document that the pH is within range on the sample worksheet. Then add a few grains of the Calgamite to each sample.

10.4.1 If the pH is not within range, add additional buffer or use a smaller sample size and recheck the pH. Do not proceed with the titration until the pH is within the required range.

10.5 Titrate the sample slowly with continuous stirring with the standard EDTA titrant until the last reddish tint disappears. The solution is normally blue at the end point. Note: Make sure that the titration is complete within 5 minutes of the time that the buffer solution is added in order to minimize calcium carbonate precipitation.

10.5.1 If the endpoint is indistinct or fades, an inhibitor may be needed. Check with the lab supervisor or manager and refer to Standard Methods 2340C, Section 2B. Add the inhibitor immediately after the addition of the buffer and proceed with the analysis as outlined above.

10.6 Calculate the final results using the equation shown below. Make sure to use the normality and not the molarity of the EDTA titrant in this calculation.

Hardness as mg $CaCO_3$/liter = (A x N X 50000)/(ml sample)

Where
A = ml of EDTA titrant
N = normality of EDTA titrant.

11.0 QUALITY CONTROL

Below is a summary of the quality control requirements for this method. Make sure to check with the laboratory supervisor or manager for any additional client specific quality control requirements.

11.1 Method Detection Limits (MDLs). MDLs should be established using a solution spiked at approximately 3 times the estimated detection limit. To determine the MDL values, take seven replicate aliquots of the spiked sample and process through the entire analytical method. The MDL is calculated by multiplying the standard deviation of the replicate analyses by 3.14, which is the student's t value for a 99% confidence level . MDLs should be determined approximately once per year or whenever there is a significant change in the background or instrument response.

11.2 Quality Control Sample (also referred to as the external). At a minimum of once per quarter, a standard from a different source than the spike blank must be analyzed. Note: It is recommended that this standard be analyzed with each run if sufficient standard is available. This sample should be within the manufacturers limits. If no manufacturer's limits are available, then default limits of 90 to 110% should be applied.

11.3 Method Blank. The laboratory must digest and analyze a method blank with each set of samples. A minimum of one method blank is required for every 20 samples or on each analysis day. The method blank must contain hardness as calcium carbonate at a level less than the reporting limit. If the method blank contains over that limit, the samples must be reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the method blank level. In addition, if all the samples are less than a client required limit and the method blank is also less than that limit, then the results can be reported as less than that limit.

11.4 Spike Blank. The laboratory must analyze a spike blank with each set of 10 samples. The laboratory should assess laboratory performance of the spike blank against recovery limits of 80 to 120 percent. If the spike blank recovery is high and the results of the samples to be

reported are less than the reporting limit, then the sample results can be reported with no flag. Otherwise, all associated samples must be reanalyzed.

11.5 Matrix Spike. The laboratory must add a known amount of each analyte to a minimum of 1 in 20 samples. The spike recovery should be assessed using in house limits. Until these limits can be generated, then default limits of 75 to 125 percent recovery should be applied. If a matrix spike is out of control, then the results should be flagged with the appropriate footnote. If the matrix spike amount is less than one fourth of the sample amount, then the sample cannot be assessed against the control limits and should be footnoted to that effect. The matrix spike recovery should be calculated as shown below.

$$\frac{\text{(Spiked Sample Result - Sample Result)}}{\text{(Amount Spiked)}} \times 100 = \text{MS Recovery}$$

11.6 Matrix Duplicate. The laboratory must digest a duplicate sample for a minimum of 1 in 20 samples. The relative percent difference (rpd) between the duplicate and the sample should be assessed. The duplicate rpd is calculated as shown below.

11.6.1 The duplicate RPD should be assessed using in house limits. Until these limits can be generated, then default limits of 20 percent RPD should be applied. If a duplicate is out of control, then the results should be flagged with the appropriate footnote. If the sample and the duplicate are less than 5 times the reporting limits and are within a range of $\pm$ the reporting limit, then the duplicate is considered to be in control.

11.6.2 The duplicate RPD should be calculated as shown below.

$$\frac{\text{(Sample Result - Duplicate Result)} \times 100}{\text{(Sample Result + Duplicate Result)} \times 0.5} = \text{\% RPD}$$

12.0 DOCUMENTATION

12.1 All reagent and standard information, including the manufacturer, lot, and expiration date should be recorded in a general chemistry reagent log.

12.2 Reagent and standard reference numbers should be recorded all on analysis write-ups.

12.3 All initial volumes, dilutions, and final volumes should be recorded on the analysis write-ups. The verification of the pH after the addition of the buffer should also be documented on the analysis write-up.

12.4 Any problems or comments about the samples should be included on the analysis write-ups.

12.5 Balance checks and calibrations should be recorded on the balance logs provided with each balance. Each balance must be checked at least once per day before use. The balance checks should be within the criteria listed in the balance log book.

13.0 DATA REPORTING

13.1 All samples should be updated to QC batches in the LIMS system. The analyst should calculate all duplicate RPD's and spike recoveries.

13.2 All documentation must be completed. The data report should be turned in to the area supervisor for review.

14.0 POLLUTION PREVENTION & WASTE MANAGEMENT.

14.1 Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 14.2.

14.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the waste management SOP, ESM003. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

14.2.1 Non hazardous aqueous wastes.

14.2.2 Hazardous aqueous wastes

14.2.3 Chlorinated organic solvents

14.2.4 Non-chlorinated organic solvents

14.2.5 Hazardous solid wastes

14.2.6 Non-hazardous solid wastes

15.0 REFERENCES

15.1 No additional references were required for this method.

ACCUTEST LABORATORIES
STANDARD OPERATING PROCEDURE

Lab Manager_____

QA Manager_____

Effective Date:_____

TITLE: DETERMINATION OF INORGANIC ANIONS BY ION CHROMATOGRAPHY using the IC2000

METHOD REFERENCES: EPA 300.0, SW846 9056, SM18 4110

***Revised Sections:* 8.3.4, 8.4.3, 8.4.8, 11.8.1**

1.0 SCOPE AND APPLICATION

1.1 This method is for the measurement of anions such as bromide, chloride, fluoride, nitrate, nitrite, ortho-phosphate, and sulfate by ion chromatography. The method is applicable to potable and non-potable water, solids after extractions, and neutral leachates.

2.0 SUMMARY OF METHOD

2.1 A small volume of sample is introduced into an ion chromatograph. The anions of interest are separated and measured, using a system comprised of a guard column, and analytical column, a suppressor column, and a conductivity detector.

2.2 Detection limits vary with the instrument conditions and calibration levels used.

3.0 REPORTING LIMIT AND METHOD DETECTION LIMIT

3.1 Reporting Limit. The normal reporting limit for this method is normally established at or above the lowest non-zero concentration standard in the calibration curve. Detected concentrations below this concentration are not reported unless MDL reporting is being done.

3.2 Method Detection Limit. Experimentally determine MDLs using the procedure specified in 40 CFR, Part 136, Appendix B. This value represents the lowest reportable concentration of an individual compound that meets the method qualitative identification criteria.

3.2.1 Experimental MDLs must be determined approximately once per year for this method.

3.2.2 Process all raw data for the replicate analysis in each MDL study. Forward the processed data to the QA group for archiving.

4.0 DEFINITIONS

BATCH: A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

CALIBRATION CHECK STANDARD. The calibration check standard is a mid-range calibration standard. It is recommended that the calibration check standard be run at a frequency of approximately 10 percent. (For some methods this is mandatory and for some it is a recommendation only. Refer to individual method SOP's) For most methods, the mid-level calibration check standard criteria is ± 10 percent of the true value. The exception to this rule is if the recovery on the calibration check standard is high and the samples to be reported are less than the detection limit.

EXTERNAL CHECK STANDARD. The external check standard is a standard from a separate source than the calibration curve that is used to verify the accuracy of the calibration standards. An external check must be run a minimum of once per quarter for all analyses where a check is commercially available. The laboratory should initially assess laboratory performance of a check standard using the control limits generated by the external check supplier. In house limits should also be generated once sufficient external check standard data is available to generate limits (usually a minimum of 20 to 30 analyses). If the external check is outside of the control limits for a given parameter, all samples must be reanalyzed for that parameter after the problem has been resolved.

SPIKE BLANK OR LAB CONTROL SAMPLE. Digest and analyze a laboratory control sample or spike blank with each set of samples. A minimum of one lab control sample or spike blank is required for every 20 samples. Assess laboratory performance against the control limits specified in the SOP. In house limits should also be generated once sufficient external check standard data is available to generate limits (usually a minimum of 20 to 30 analyses). If the lab control is outside of the control limits for a parameter, all samples must be redigested or redistilled and reanalyzed for that parameter. The exception is if the lab control recovery is high and the results of the samples to be reported are less than the reporting limit. In that case, the sample results can be reported with no flag. Note: If control limits are not specified in the SOP, then default limits of 80 to 120 percent should be used.

MATRIX: The component or substrate (e.g., water, soil) which contains the analyte of interest.

MATRIX DUPLICATE: A duplicate sample is digested at a minimum of 1 in 20 samples. The relative percent difference (RPD) between the duplicate and the sample should be assessed. The duplicate RPD is calculated as shown below. Assess laboratory performance against the control limits that are specified in the SOP. In house limits are generated once sufficient duplicate data is available to generate limits (usually a minimum of 20 to 30 analyses). If a duplicate is out of control, flag the results with the appropriate footnote. If the sample and the duplicate are less than 5 times the reporting limits and are within a range of ± the reporting limit, then the duplicate is considered to be in control. Note: If control limits are not specified in the SOP, use default limits of ± 20% RPD.

$$\frac{(|\text{Sample Result} - \text{Duplicate Result}|) \times 100}{(\text{Sample Result} + \text{Duplicate Result})/2} = \text{Duplicate RPD}$$

MATRIX SPIKE: The laboratory must add a known amount of each analyte to a minimum of 1 in 20 samples. The matrix spike recovery is calculated as shown below. Assess laboratory performance against the control limits that are specified in the SOP. In house limits are generated once sufficient matrix spike data is available to generate limits (usually a minimum of 20 to 30 analyses). If a matrix spike is out of control, then the results should be flagged with the appropriate footnote. If the matrix spike amount is less than one fourth of the sample amount, then the sample cannot be assessed against the control limits and should be footnoted to that effect. Note: If control limits are not specified in the SOP, then default limits of 75 to 125 percent should be used.

$$\frac{\text{(Spiked Sample Result - Sample Result)}}{\text{(Amount Spiked)}} \times 100 = \text{Matrix Spike Recovery}$$

MATRIX SPIKE DUPLICATES: Intralaboratory split samples spiked with identical concentrations of target analyte(s). The spiking occurs prior to sample preparation and analysis. They are used to document the precision and bias of a method in a given sample matrix.

METHOD BLANK. The laboratory must digest and analyze a method blank with each set of samples. A minimum of one method blank is required for every 20 samples. For a running batch, a new method blank is required for each different digestion day. If no digestion step is required, then the method blank is equivalent to the reagent blank. The method blank must contain the parameter of interest at levels of less that the reporting limit for that parameter. If the method blank contains levels over the reporting limits, the samples must be redigested or redistilled and reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the method blank level. In addition, if all the samples are less than a client required limit and the method blank is also less than that limit, then the results can be reported as less than that limit.

METHOD DETECTION LIMITS (MDLS). MDLs should be established for all appropriate methods, using a solution spiked at approximately 3 times the estimated detection limit. To determine the MDL values, take seven replicate aliquots of the spiked sample and process through the entire analytical method. The MDL is calculated by multiplying the standard deviation of three replicate analyses by 3.14, which is the student's t value for a 99% confidence level. MDLs should be determined approximately once per year for frequently analyzed parameters.

REAGENT BLANK: The reagent blank is a blank that has the same matrix as the samples, i.e., all added reagents, but did not go through sample preparation procedures. The reagent blank is an indicator for contamination introduced during the analytical procedure. (Note: for methods requiring no preparation step, the reagent blank is equivalent to the method blank.) Either a reagent blank or a method blank must be analyzed with each batch of 20 samples or less. The concentration of the analyte of interest in the reagent blank must be less than the reporting limit for that analyte. If the reagent blank contains levels over the reporting limits, the samples must be reanalyzed. The exception to this rule is when the samples to be reported contain greater than 10 times the reagent blank level. In addition, if all the samples are less than a client required limit and the reagent blank is also less than that limit, then the results can be reported as less than that limit.

REAGENT GRADE: Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents which conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.

REAGENT WATER: Water that has been generated by any method which would achieve the performance specifications for ASTM Type II water. For organic analyses, see the definition of organic-free reagent water.

REFERENCE MATERIAL: A material containing known quantities of target analytes in solution or in a homogeneous matrix. It is used to document the bias of the analytical process.

STANDARD CURVE: A plot of concentrations of known analyte standards versus the instrument response to the analyte. Calibration standards are prepared by successively diluting a standard solution to produce working standards that cover the working range of the instrument. Standards should be prepared at the frequency specified in the appropriate section. The calibration standards

should be prepared using the same type of acid or solvent and at the same concentration as will result in the samples following sample preparation. This is applicable to organic and inorganic chemical analyses.

5.0 HEALTH & SAFETY

5.1 The analyst should follow normal safety procedures as outlined in the Accutest Laboratory Safety Manual which includes the use of safety glasses and lab coats. In addition, all acids are corrosive and should be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these reagents should be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets should be made available to all personnel involved in these analyses.

6.0 COLLECTION, PRESERVATION, AND HOLDING TIME

6.1 For bromide, chloride, and fluoride, no special preservations are required. For nitrite, orthophosphate, and sulfate, the sample must be cooled to 4 °C. For nitrate, the sample must be preserved to a pH of less than 2 with sulfuric acid unless it is analyzed within 48 hours.

6.2 Bromide, chloride, fluoride, and sulfate must all be analyzed within 28 days. If nitrate is preserved with sulfuric acid, it must also be analyzed within 28 days. Nitrite, orthophosphate, and unpreserved nitrate must be analyzed within 48 hours.

6.2.1 It is recommended that separate samples be collected and preserved for nitrate so that the longer holding time may always be applied.

7.0 APPARATUS AND MATERIALS

7.1 Ion Chromatograph with a guard column, an analytical column, a suppressor column, and a conductivity conductor. This SOP is written for the use with the Dionex ICS-2000 instrument. The ICS-2000 is run using the external water mode with the suppressor and using internally generated eluent. No manual eluent preparation is required for the ICS-2000 instruments. The columns used are listed below. Alternate columns may be used if all method requirements can be met.

7.1.1 Suppressor column, ASRS Ultra 4 mm. Dionex part number 061561

7.1.2 Guard Column, IONPAC AG18 4 mm. Dionex part number 060551.

7.1.3 Analytical Column, IONPAC AS18 4 mm. Dionex part number 060549,

7.2 Analytical balance, capable of weighing to 4 places.

7.3 Volumetric flasks, class A.

7.4 Volumetric pipets, class A or autopipeters. Note: If autopipeters are used, make sure that the calibration is checked before use as specified in the autopipeter SOP.

7.5 Helium tank and regulator. On the IC-2000 instruments, helium is used only for head pressure on the water reservoirs. The pressure should be set at approximately 6 psi.

7.6 Magnetic stirrers and stirring bars (for solid samples)

7.7 Nylon 0.20 um membrane filters or equivalent, that can be attached to the end of the syringe.

8.0 REAGENTS

All chemicals listed below are reagent grade unless otherwise specified. Deionized water must be used whenever water is required.

8.1 Stock Standard Solution, 4000 mg/L: The stock standard solution must be prepared for chloride and sulfate as listed below. If no other instructions are noted, then dry the salt at 104 deg. C for a minimum of 2 hours or to constant weight and store in a desiccator before use

8.1.1 Chloride and Sulfate stock: Dissolve 6.594g of sodium chloride (NaCl) and 7.256g of potassium sulfate (K_2SO_4) in 500 mL of DI water and dilute to 1 liter. This stock standard may be stored for 6 months.

8.1.1.1 Dry the sodium chloride for 1 hour at approximately 600 deg. C and cool and store in a desiccator before use.

8.2 Stock standard solutions, 1000 mg/l: Stock standard solutions must be prepared for each anion as listed below. If no other instructions are noted, then dry the salt at 104 deg. C for a minimum of 2 hours or to constant weight and store in a desiccator before use.

8.2.1 Bromide stock: Dissolve 1.2876 g of sodium bromide (NaBr) in reagent water and dilute to 1 liter. This stock standard may be stored for 6 months.

8.2.1.1 Dry the sodium bromide at 150 deg. C for a minimum of 6 hours. Cool and store in a desiccator before use.

8.2.2 Fluoride stock: Dissolve 2.2100 g of sodium fluoride (NaF) in reagent water and dilute to 1 liter. This stock standard may be stored for 6 months.

8.2.3 Nitrate stock: Dissolve 6.0679 g of sodium nitrate ($NaNO_3$) in reagent water and dilute to 1 liter. This stock standard may be stored for 6 months.

8.2.4 Nitrite stock: Dissolve 4.9257 g of sodium nitrite ($NaNO_2$) in reagent water and dilute to 1 liter. This stock standard may be stored for 1 month, but may be used for 3 months if the analyte is not to be reported.

8.2.5 Orthophosphate stock: Dissolve 4.3937 g of potassium phosphate (KH_2PO_4) in reagent water and dilute to 1 liter. This stock standard may be stored for 1 month,

but may be used for 3 months if the analyte is not to be reported.

8.3 Working standard and spiking solutions: Solutions containing bromide, chloride, fluoride, nitrate, and sulfate must be made fresh weekly. Solutions containing nitrite and orthophosphate should be made fresh daily but can be made weekly as long as they are not reported and used for retention time recognition only. Note: Levels shown below are suggested levels and may be changed to meet different reporting limit requirements.

8.3.1 Standard Intermediate "A" (100 mg/l chloride and sulfate). Using a volumetric autopipet, transfer 2.50 mL of 4000 mg/l stock into 75 ml DI and dilute to 100 ml.

8.3.2 Spiking Solution Intermediate. Pipette the following amounts of the stocks shown into a 100 ml volumetric flask. Dilute to final volume of 100 ml and mix well.

10ml of 1000 mg/l Fluoride = 100 mg/l
10ml of 1000 mg/l Nitrite = 100mg/l
25ml of 1000 mg/l Bromide = 250 mg/l
10ml of 1000 mg/l Nitrate = 100 mg/l
10ml of 1000 mg/l Orthophosphate = 100 mg/l

8.3.3 Standard Intermediate "B". Pipette 20 ml of Spiking Solution Intermediate into 100 ml volumetric flask, dilute to final volume of 100 ml and mix well.

8.3.4 Spiking Solution Soup. Using a class A pipette or cylinder transfer 50 ml of 4000 mg/L chloride and sulfate stock and 50 ml of spiking solution Intermediate together in a 100 ml volumetric flask. Use 2 ml of this solution to 50 ml DI for spike blank or sample for matrix spike. Final concentrations for this spiking solution are shown below.

Analyte	Conc. In spiking solution	Conc in spike blank
Fluoride	50 mg/l	2.00 mg/l
Nitrite	50 mg/l	2.00 mg/l
Bromide	125 mg/l	5.00 mg/l
Nitrate	50 mg/l	2.00 mg/l
Orthophosphate	50 mg/l	2.00 mg/l
Chloride	2000 mg/l	80.0 mg/l
Sulfate	2000 mg/l	80.0 mg/l

8.4 Calibration standard solutions: Solutions containing bromide, chloride, fluoride, nitrate, and sulfate must be made fresh weekly. Solutions containing nitrite and orthophosphate should be made fresh daily, but can be made weekly as long as they are not reported and used for retention time recognition only. Note: Levels shown below are suggested levels and may be changed to meet different reporting limit requirements.

8.4.1 Standard A. This is the blank and contains DI water only.

8.4.2 Standard B. Pipette the following amounts of stocks shown into a 100 ml volumetric flask: 0.50 ml of intermediate "A" and 0.25 ml of intermediate "B"

8.4.3 Standard C. Pipette the following amounts of stocks shown into a 100 ml volumetric

flask: 2.00 ml of intermediate "A" and 0.50 ml of intermediate "B"

8.4.4 Standard D. Pipette the following amounts of stocks shown into a 100 ml volumetric flask: 10.0 ml of intermediate "A" and 2.00 ml of intermediate "B"

8.4.5 Standard E. This standard level is normally also used as the CCV standard. Pipette the following amounts of stocks shown into a 100 ml volumetric flask: 50.0 ml of intermediate "A" and 10.0 ml of intermediate "B"

8.4.6 Standard F and CCV. Pipette the following amounts of stocks shown into a 200 ml volumetric flask: 10.0 ml of 4000 mg/l chloride and sulfate stock and 30.0 ml of intermediate "B" and 1.5 ml of 1000 mg/l bromide stock.

8.4.7 Standard G. Pipette the following amounts of stocks shown into a 100 ml volumetric flask: 10.0 ml of 4000 mg/l chloride and sulfate stock and 30.0 ml of intermediate "B" and 1.0 ml of 1000 mg/l of bromide stock.

8.4.8 The final concentrations in the suggested standards outlined above are shown in the table below. All units are in mg/l.

Anion	STDA	STDB	STDC	STDD	STDE	STDF	STDG
F	0.0	0.05	0.1	0.4	2	3	6.0
CHL	0	0.5	2	10	50	200	400
NO2	0.0	0.05	0.1	0.4	2	3	6.0
BRO	0.0	0.125	0.25	1	5	15	25
NO32	0.0	0.05	0.1	0.4	2	3	6.0
OPO4	0.0	0.05	0.1	0.4	2	3	6.0
SO4	0	0.5	2	10	50	200	400

8.8 ICV (External Check Solution.) The ICV can be made in the same manner as the standard curve from a separate source than the standards. It must be within the range of the curve. Alternatively, it can be purchased from a outside supplier.

8.9 0.2N H_2SO_4 for suppressor regeneration: Pipet 1.0 ml of concentrated H_2SO_4 into 100 ml DI and dilute to final volume of 200ml with DI.

8.10 0.1M Oxalic Acid for metals column clean-up: Dissolve 6.3 g of oxalic acid into approx. 300 mL of DI water. Bring to final volume of 500 mL with DI water.

8.11 10X eluent concentrate (300mM KOH) for hydrophilic ionic contamination clean-up.

8.12 Acetonitrile, reagent grade

8.13 1M HCl: Add 8.3 ml of concentrated hydrochloric acid to approximately 70 ml of deionized water. Dilute to a final volume of 100 ml and mix well.

9.0 INTERFERENCES

9.1 Interferences can be caused by substances with retention times that are similar to and overlap those of the anion of interest. This interference is especially important at low

concentrations.

9.2 The acetate anion elutes early during the chromatographic run and can cause elution times of other anions to vary when large amounts of acetate are present. High levels of acetate also can cause interference with the fluoride peak. Therefore, this method is not recommended for leachates containing acetic acid.

9.3 Large amounts of an anion can interfere with the peak resolution of an adjacent anion. High concentrations of an anion can also cause the peak to be misidentified on the chromatograph due to the large width of the peak.

10.0 SAMPLE PREPARATION PROCEDURE

10.1 For soil samples, follow the preparation outlined below.

10.1.1 Mix the sample well and remove any artifacts. Remove a 10.0 g aliquot of sample and add 100 ml of DI water. Mix the resulting slurry for 10 minutes using a magnetic stirring device. Filter the resulting slurry through a 0.45 um membrane filter before analysis.

10.1.2 For matrix spikes, make sure to spike the aliquot of the sample directly and then add the 100 ml of water and stir.

10.1.3 Check with the lab supervisor if there is insufficient sample to use a 10.0 g aliquot. Smaller aliquots may be used if a homogeneous portion of the sample can be obtained. The sample must always be extracted with 10 times the sample weight of DI water.

10.2 For water samples, filter all samples through 0.45 um filters before analysis. Matrix spikes must be spiked before filtration.

11.0 ION CHROMATOGRAPHY ANALYSIS PROCEDURE FOR THE ICS-2000 INSTRUMENTS

11.1 Check to make sure that the helium tank pressure is > 100 PSI and the pressure gauge by the eluent bottles is set at 6 PSI.

11.2 Fill the eluent generation bottle(s) that are to be used with deionized water (resistance of 18.2 megaohms or greater), making sure that they are pressurized with helium. On the instrument panel (or in the software) set the water volume at the level in the bottles and adjust the flow rate up to 1.0 ml/min. Check the KOH is set at the proper molarity. Normally it should be set at 30 mM. Also fill the external water bottle(s) for the supressor with deionized water.

11.3 Check the lines coming out of the supressor for air bubbles. Bubbles should be present. If not, then check to make sure the current is on and the suppressor is working properly.

11.4 Check the pump waste line and see if bubbles are present. If they are present, then prime the pump using the procedure described below.

11.4.1 Hit the prime button on the front panel.

11.4.2 Open up the pressure transducer on the pump by turning it 3 turns to the left. Then enter OK on the front panel.

11.4.3 Put the syringe in the syringe prime knob and turn it 3 times to the left and begin to slowly pull back on the syringe to 10 ml. Check for air bubbles on the pump waste line. If not air bubbles are present, then close the syringe priming knob and shut the pump off. Then close the pressure transducer knob.

11.4.4 Allow the instrument conditions to settle and then check the pressure and check for air bubbles. If there is still a problem, the priming procedure may be repeated.

11.5 In the software, go to the browser and go to the correct instrument panel (1 or 2). Then connect the instrument. Monitor the baseline until it is stable.

11.6 Go to the template sequences and edit a sequence for the samples in the run. If a calibration is being using from an earlier run, make sure to copy the calibration into the front of the sequence. After the sequence is generated, then save it using the file name (instrument, date, run). Refer to the instrument manuals or help screens in the program for help in using the software. A summary of the instrument conditions required for the analysis of perchlorate is shown below. Note: the retention time window for each anion must not exceed ± 10% of that anions retention time from the calibration. Refer to section 13.7 for more discussion of the proper application of retention time.

Column: IonPac AG18, AS18
Eluent: 30 mM KOH
Surpressor setting: approximately 100 mAmps. This setting will be autogenerated.
Flow Rate: 1.0 ml/min
Inj. Volume: 12.5 µl
Pump pressure – should be around 2000 psi
Detection: Suppressed conductivity, SRS Ultra II, external water mode

11.7 Load the autosampler and turn it on. The autosampler should then move to the first sample. A print-out of the autosampler table should be generated showing the order that the samples are loaded into the autosampler.

11.8 Start the run. Monitor the results as the run is going to make sure that problems are identified quickly. Note: the initial demonstration of capability, including instrument MDL's and linear calibration ranges, must be completed before samples can be run.

11.8.1 Data files should be saved using the naming scheme of instrument, year (last 2 digits), month, day, run number followed by the extension of .ic. For example, the first IC run on instrument 2 on May 20, 2005 would be named 205052001. This name should always be used in the workgroup description in the LIMS system.

11.8.2 It is recommended that a new calibration be run a minimum of once per month. (It is required that a calibration be run once per quarter.) Calibrations standards may be varied from the one stated in this SOP depending on the levels of each anion that are to be reported. A minimum of 5 standards and a blank are required and a low standard must be at or below the reporting limit for each anion. A correlation coefficient of 0.995 is required. If this correlation coefficient is not met, than the

instrument must be recalibrated.

11.8.3 After the calibration, a low check at the reporting limit must be run. This low check must have the levels in standard B or at the reporting limit for the calibration outlined in this SOP and recoveries must be in the range of 75 to 125. On a daily basis, it is recommended that an external check is analyzed and recoveries must be within a range of 90 to 110 percent. (This check must be analyzed at a minimum with each new calibration.) Continuing calibration checks and continuing calibration blanks must be run every 10 samples. The continuing calibration checks must have recoveries in the range of 90 to110 percent. Refer to the quality control section of this SOP for more detail on these quality control samples.

11.9 After the run is completed, review all of the chromatograms and check for overlapping peaks, dilutions, etc.

11.9.1 If the retention time of any anion in the ICV or CCV check standards has shifted more than 10% from the original calibration curve retention time, then no results can be reported for that anion. The column should be reconditioned, if necessary, and the instrument recalibrated before any more samples are reported for that anion. Affected samples are reanalyzed after the problem has been corrected.

11.9.2 If a sample peak has shifted significantly from the original retention time (and the ICV and CCV check standards are within the 10% retention time window), then verify the reported result using post-digest spike on that sample. Do not report results from peaks where the retention time has shifted more than 10 percent unless the peak can be verified using a post-digest spike.

11.9.3 For large or overlapping peaks, make dilutions. If at all possible, make dilutions and reruns on the same run as the original sample.

11.9.4 Refer to section 13.7 for information on how to determine the appropriate retention time window.

11.10 Review all data and update the appropriate tests in the LIMS system. A write-up including a run log, a calibration summary, batch quality control summary, and copies of all chromatograms should be turned into the area supervisor for each batch.

11.10.1 If edits are needed in the calibration after the data has been calculated, the run can be reprocessed using the batch function in the software. Refer to the instrument manuals or on-screen help for addition information.

12.0 INSTRUMENT MAINTENANCE

12.1 Whenever a new suppressor is put in place or when the baseline is unstable or very high, the suppressor should be regenerated. The procedure below is for the Ultra 4 mm suppressor.

12.1.1 Using a disposable plastic syringe, push approximately 3 mL if 0.2(200mN) H_2SO_4 through the ELUENT OUT port and 5 mL of 0.2N H_2SO_4 through the REGIN IN port respectively.

12.1.2 Allow the suppressor to sit for approximately 20 minutes to fully hydrate the suppressor screens and membranes.

12.1.3 Re-connect the suppressor to the system in the recycle mode.

12.2 Periodically, due to the matrix of samples, both guard and analytical columns become degraded and cleaning them becomes necessary. This is evidenced in changing retention times, round-shaped peaks, tailing peaks and overall poor integration. The metals cleanup should be done a minimum of once per month, while the others should be done a minimum of once per quarter.

12.2.1 There are 3 recommended cleanup solutions for the AS18A and AG18 columns.

12.2.2 Metal contamination column clean-up: Use 500 ml of 0.1M oxalic acid solution.

12.2.3 Low valency hydrophilic ionic contamination column clean-up. Use 500 ml of 10X eluent concentrate (300 mM KOH).

12.2.4 High valency hydrophobic ion 200mM HCl in 80% acetonitrile: The acetonitrile solution is stored in a separate eluent bottle because acetonitrile slowly breaks down in acidic aqueous solutions. Prepare 2 bottles (E1 and E2) with the following 500-mL solutions: E1: 100% Acetonitrile and E2: 1M HCl using DI water.

12.3 Column Clean-up Procedure.

12.3.1 Prepare 500 mL solution of the appropriate cleanup solution from 12.2.1

12.3.2 Disconnect the ASRS-ULTRA from the IonPac AS18A Analytical column. **Make sure to reverse the order of the guard and analytical column in the eluent flow path.** Contaminants that have accumulated on the guard column can be eluted onto the analytical column and irreversibly damage it. If in doubt, clean each column separately. Double check that the eluent flows in the direction designated on each of the column labels.

12.3.3 Set the pump flow rate to 1.0 mL/min for an AS18A 4-mm analytical or guard column.

12.3.4 Rinse the column for 15 minutes with DI water before pumping the chosen cleanup solution over the columns.

12.3.5 Pump the cleanup solution through the column for at least 60 minutes.

12.3.6 Rinse the column for 15 minutes with DI water before pumping eluent over the column.

12.3.7 Equilibrate the columns with eluent before resuming normal operation for at least 30 minutes.

12.3.8 Reconnect the ASRS-ULTRA and place the guard column in line between the injection valve and the analytical column.

13.0 QC REQUIREMENTS

13.1 A method detection limit study must be done approximately once per year. The MDL study is done following the procedure outlined in the Accutest laboratory MDL SOP. A minimum of seven replicates spiked at 3 to 5 times the MDL must be taken through the procedure for each anion. If instrument conditions (columns, etc.) are modified, then a new MDL must be done.

13.2 A batch is defined as a maximum of 20 samples in one day. A method blank and a spike blank are required to be run with every batch. Additionally two matrix spikes and a duplicate are required for every 20 samples. In some cases a matrix spike duplicate may be required in place of a duplicate.

13.2.1 The method blank must contain less than the reporting limit of each anion that is reported and this sample must be run with each set of samples in a batch. If the blank contains more than the reporting level, then all samples must be reanalyzed. If no sample volume remains to be reanalyzed, then the data must be flagged. (The exception is if the sample results are less than the reporting limit.)

13.2.1.1 Although the method states that values greater than the MDL should be suspect, this is not appropriate for the concentration levels being applied for this analysis. MDL's are generally at least 10 times lower than reporting limits for all analytes and values over the MDL do not impact data usability.

13.2.2 The recovery of the spike blank must be within the limits of 90 to 110% recovery for each anion that is reported and this sample must be run with each set of samples in a batch. If the recoveries are outside of this range, then all associated samples must be reanalyzed. If no sample volume remains to be reanalyzed, then the data must be flagged.

13.2.3 The matrix spike is spiked with all anions of interest. Method limits of 80 to 120 % recovery must be applied. Control limits must be generated from laboratory data to support method limits. If the recoveries are outside of this range, and all other method quality control is within limits, then matrix interference should be suspected.

13.2.4 For either duplicates or matrix spike duplicates, control limits of 20% RPD must be applied for all sample values within the calibration range (up to 10 times the reporting limit). If the RPD values are outside of this range, and all other method quality control is within limits, then sample non-homogeneity should be suspected.

13.3 An external source must be analyzed at least once per quarter and/or after every new calibration. This external must be within the range of 90 to 110 % recovery. If this criteria is not met, the problem must be corrected and the external source reanalyzed. It is recommended that the external source be analyzed with each batch to verify the standard values obtained.

13.4 It is recommended that a new calibration be run a minimum of once per month. (It is required that a calibration be run once per quarter.) Calibrations standards may be varied from the one stated in this SOP depending on the levels of each anion that are to be reported. A minimum of 5 standards and a blank are required and a low standard must be at or below the reporting limit for each anion. A correlation coefficient of 0.995 is required.

$$r = \frac{\Sigma(x - \bar{x})(y - \bar{y})}{\sqrt{\Sigma(x - \bar{x})^2 \Sigma(y - \bar{y})^2}}$$

Where r = correlation coefficient
x = amount of analyte
y = response of instrument

$\bar{x}$ = average of x values

$\bar{y}$ = average of y values

13.4.1 A new calibration is required when standard retention times shift by more than 10% from the original calibration.

13.5 A low check at the reporting limit for each anion must be run after each calibration. Recoveries for this low check must be in the range of 75 to 125 for all reported anions. If the recoveries are not in this range, the problem must be identified and the instrument recalibrated.

13.6 Continuing Calibration Verification (CCV) Checks at or near the mid-level of the curve must be run at the beginning and the end of the run and after every 10 samples throughout the run. Every CCV must be followed by a continuing calibration blank (CCB). The CCV must have results within 90 to 110 % of the true value. If the results are outside of that range for a CCV, then all bracketed samples must be reanalyzed. The results for the CCB must be less than the reporting limit for an analyte. If they are not, then all bracketed samples for that analyte must be reanalyzed.

13.7 Retention Time Windows. The width of the retention time window used to make identification should be based upon measurements of the actual retention time variations of standards over the course of an analysis. A suggested window would be three times the standard deviation of the retention time windows for an analysis day or period of time covered by a calibration. However, sample concentration and matrix can greatly affect retention time and the experience of the analyst and reviewer should weigh heavily in the interpretation of a chromatogram. Therefore, 10 % is defined as the widest window that can be used. If an analyte is outside of that window, it cannot be reported unless that peak is confirmed with a post-digest spike.

14.0 DOCUMENTATION REQUIREMENTS

14.1 All reagents must be recorded in a reagent logbook with manufacturers, lot numbers, and

expiration dates. All reagent information must be cross referenced on the sample worksheet.

14.2 All instrument data must be exported to the LIMS system and a copy of the run log must be included in the logbook by the instrument.

14.3 A data package consisting of a manual run log, a LIMS run log, a calibration summary, batch quality control summary, and copies of all chromatograms must be turned into the area supervisor for each batch. The analyst should also complete the preliminary review in the LIMS system.

14.4 Refer to EQA044 for procedures and documentation that must be followed when peaks are manually integrated.

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 15.2.

15.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the waste management SOP, ESM003. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

15.2.1 Non hazardous aqueous wastes.

15.2.2 Hazardous aqueous wastes

15.2.3 Chlorinated organic solvents

15.2.4 Non-chlorinated organic solvents

15.2.5 Hazardous solid wastes

15.2.6 Non-hazardous solid wastes

16.0 ADDITIONAL REFERENCES

16.1 Dionex Instrument and column manuals

16.2 EQA044 Manual Integration SOP.

Attachment 14

BCI Inc SOP 14 for Macrocosm Presence Absence test

Bioremediation Consulting Inc.

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Standard Operating Procedure

 # 14

Microcosm Studies

and

Presence-Absence Tests

October 21, 2008

Prepared by: _____ _____
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BCI SOP #14

Microcosm Studies

1 Scope and Application

The purpose of a Microcosm Study is to enhance the understanding of the microbial interactions at a site in order to document natural attenuation of organic contaminants, document methods for optimizing biodegradation, examine remedial options, or enhance the efficacy of a bioremediation program. Both anaerobic and aerobic Microcosm tests are discussed in this SOP.

2 Simple vs Complex Microcosm Studies

Presence/Absence Tests

Presence / Absence Tests are short term studies focused on documenting the presence of a specific microbial type. *Procedures for setup and analysis, as well as quality control considerations, are the same as those used in more extensive testes.* Only one microcosm is constructed for each well, amended to provide optimal conditions for biodegradation, and usually, only two time points are anayzed: start and finish.

Microcosm Studies, - Longer term, have more complex goals, including:

- Document on-going natural attenuation,
- Investigate the availability of natural electron donor.
- Determine amount and type of amendments needed to optimize biodegradation,
- Demonstrate the effect of pH on success of biodegradation.
- Examining site-specific contaminant interactions.
- Demonstrate successful Bioaugmentation.

3 Typical Microcosm Study Plan for each well sample

Negative Control: pH adjusted to 2 to kill bacteria. The purpose of the negative control is to detect abiotic degradation of contaminants, and to show evidence of containment (no leaks).

Positive Control. Amended ground water inoculated with BCI culture. The purpose of the positive control is to document that BCI techniques preserve anaerobic conditions and support activity of known biodegrading organisms.

(3 or more) Experimental Microcosms. The experimental microcosms address one experimental variable each, such as different donors for anaerobic studies, different pH, the requirement for added minerals, intra site bioaugmentation, bioaugmentation with BCI culture. For aerobic studies, separate microcosms might be set up to investigate co-metabolic vs direct oxidation of contaminant. For microcosms which include soil, the tests can also provide information on microbial / geochemical interactions.

4 Summary of Method

Selection of wells for microcosm testing is usually based on prior geochemical characterization of well samples. Ground water samples from a monitoring well, and if possible, corresponding soil core material, are collected in a manner which preserves the subsurface reducing conditions. A set of microcosms is constructed for each well sample, including controls and experimental bottles. Microcosms are sealed with septa through which small amounts can be transferred by syringe, and are amended by injecting amendments through the septum.

Microcosm bottles are monitored by removing headspace sub-samples by syringe through septum and analyzing by gas chromatography for contaminant reduction. For anaerobic microcosm tests, headspace samples are also analyzed for microbial H_2 production, and aqueous samples are analyzed by electrophoresis for microbial reduction of sulfate and utilization of electron donors. For aerobic microcosm tests, headspace samples are monitored for O_2 use and CO_2 generation. Anaerobic tests require 3 to 5 monthly analyses, to observe long-term processes, such as sequential degradation of a variety of contaminants, or bioaugmentation at the midpoint of the test period. For aerobic tests, analyses may be more frequent.

Results for anaerobic reductive dechlorination can be validated by comparing the molar amount of parent contaminant with the molar amounts of biodegradation products produced. Results for aerobic dechlorination can be supported by corellation with O_2 use and CO_2 generation. For aerobic co-metabolic dechlorination, results can be correlated with utilization of growth-supporting alkane (such as methane).

5 *Interferences*

For anaerobic microcosm studies, exposure of ground water to air during sampling can inhibit strict anaerobes that are of interest in characterizing a site's potential to dechlorinate certain contaminants. A reducing agent is provided in the sampling bottle to protect the sample from oxidation. Further reducing agent may be added upon arrival at the laboratory if necessary. Procedures to eliminate exposure of bacteria to oxygen during testing are described in BCI SOP #10, Anaerobic Procedures. For both anaerobic and aerobic tests, high concentrations of certain compounds may interfere with the activity of microorganisms.

6 *Sample Collection, Preservation, and Handling*

Ground Water Sampling, Shipment and Storage. For anaerobic microcosm testing, glass serum sample bottles are supplied which have been filled with Argon to exclude oxygen, and contain a small amount of black ferrous sulfide reducing agent. Groundwater samples are collected using a peristaltic pump with EPA low-flow procedures. The sampling tube is inserted a few inches into serum bottle and held in place. Argon is heavier than air, thus continues to blanket the groundwater during filling. The small (1 cm) opening of the bottle limits turbulence as Argon is forced out of the bottle.

The sulfide reducing agent removes traces of oxygen which might enter the water during transfer from the well. In addition, the reducing agent serves as an indicator, being black when reduced and orange when oxidized. Serum bottles are filled completely, stoppered, and secured with crimped caps. The samples are then packed in special foam protection in a BCI cooler. Ice (in warm seasons) is limited to maintain ground water temperature. Coolers are shipped *priority* overnight to BCI.

Soil Sampling, Handling, and Storage. For anaerobic microcosm tests, soil cores remain in brass tubes, which are immediately sealed, and shipped to BCI where they will be stored unopened to exclude oxygen. For soil preparation, cores will be opened in an anaerobic glove box, sieved and mixed, then placed in Argon-flushed glass jars with Teflon-lined lids.

6 *Methods for Microcosm Set-Up*

Microcosm Construction and Incubation. Microcosms are constructed in 160 ml serum bottles. For anaerobic microcosms, the bottles are initially flushed with argon, and techniques for the exclusion of oxygen during microcosm set-up and monitoring are carried out according to BCI SOP #10 "Anaerobic Procedures". Aerobic tests are set up under aerobic conditions. Microcosms are incubated inverted at 21°C in darkness, and shaken briefly 3 at least times/week.

Optional Addition of Soil to Microcosm Bottles: For anaerobic microcosms, soil cores are opened in an anaerobic glove box, the soil is sieved and composited, about 30 grams of soil composite are transferred to each microcosm bottle, and the bottle closed with a temporary stopper. Operation of the anaerobic glove box is described in BCI SOP #15.

Addition of Ground Water and Sealing of Anaerobic Microcosm Bottles. Anaerobic Transfer of 100 ml of ground water to the microcosm bottles will be performed outside of the glove box, using a proprietary transfer method involving Argon flushing. Microcosms are sealed with Teflon-lined gray butyl rubber septa affixed by crimped aluminum caps, then pressurized 5% by injecting additional gas.

Amendments. Anaerobic Microcosms may be amended with organic compounds as energy source for the bacteria, such as organic acids or carbohydrates. Amendment solutions for anaerobic microcosms are prepared using procedures which exclude oxygen, and will be injected into the sealed microcosm bottles through the septa. For both aerobic and anaerobic microcosms, mineral amendments such as ammonia and phosphate will be added, and in some cases, amendment with other minerals may be conducted. Some microcosms may be 'spiked' with chlorinated compounds.

Killed and Positive Control Microcosms One microcosm in each set will be converted to a 'killed control' by lowering the pH to 2.5. The killed control will be monitored to document stability of contaminant to abiotic losses. A Positive control microcosm will be inoculated with about 0.3 ml of BCI culture containing the appropriate bacteria.

7 *Microcosm Monitoring, Maintenance and Waste Handling*

Chlorinated Compounds, Dissolved Gasses and Molecular Hydrogen. Headspace samples, 100 μL, will be analyzed by gas chromatography for volatile compounds by direct injection into a HP5890 gas chromatograph with flame ionization detector, quantitated according to BCI SOP #12. For anaerobic microcoms, H_2 will be analyzed by removing 100 μL headspace samples and injecting into a Trace Analytical Reduction Gas Analyzer according to BCI SOP #13. For aerobic microcosms, headspace samples will be analyzed by gas chromatography (BCI SOP #12) for O_2 and CO_2, as well as for light hydrocarbons (methane, propane) which support co-metabolic degradation of contaminant.

Organic Acids and Sulfate, and pH. Aqueous subsamples, 150 μL, will be obtained by syringe through the septum. Organic acids and sulfate will be determined by capillary ion electrophoresis for anions according to EPA method 6500 and BCI SOP #11, suplemented by ion chromatograaphy, EPA Method 300. Microcosm subsamples will be analyzed by Orion pH meter with semi-micro electrode.

Microcosm Maintenance.

Data on pH will be used to determine when pH should be adjusted.

For anaerobic microcosms, information on donor utilization, H_2 concentrations, sulfate reduction, and dechlorination will be used to determine whether additional donor is required, or bioaugmentation should be carried out.

For aerobic microcosms, information on O_2 and light hydrocarbon use, and CO_2 production, will be used to determine when additional O_2 and light hydrocarbon should be added.

Waste Handling. Unused ground water and soil, as well as waste from completed microcosm studies, will be disposed of in barrels provided by licensed hazardous waste disposal company.

8 Data Treatment, Reporting and Quality Assurance.

Data Handling. Data is stored and recorded according to the BCI SOPs for gas chromatography (#12), electrophoresis (#11), and reduction gas analyzer (#13). Records of set-up procedures, amendments, spiking amounts pH and adjustments, and other information are recorded in a project notebook.

Reporting. Reports include all contaminant data in tabular and graphic form. Data on anaerobic dechlorination is presented in molar concentrations, so that recovery of daughter products can be ascertained easily. The results and interpretations are also described in a narrative.

Quality Assurance. Although microcosm bottles are occasionally set up and operated in duplicate, the cohesiveness of the entire data set is the best quality control. Killed control bottles which maintain relatively constant values for all analytes over many months are evidence for maintenance of airtight closure of bottles and re-sealing of septa. For anaerobic microcosms, the molar equivalence of degraded parent compound and appearance of each daughter product indicates appropriate quantitation of analytical data. For aerobic microcosms, simultaneous contaminant degradation, O_2 use and CO_2 production support validity.

9 References

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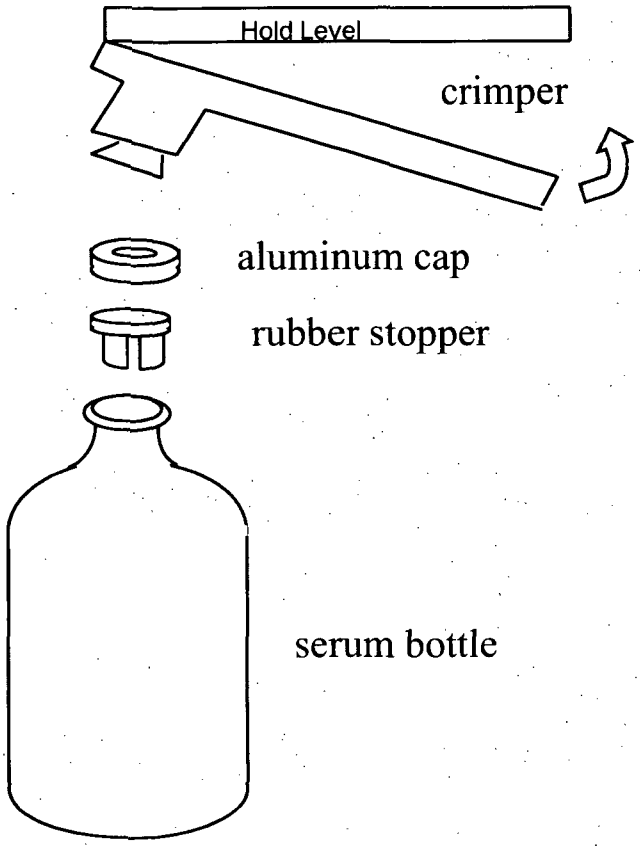

Before filling 1-Liter bottle
Practice with *Crimper Practice Kit*

Practice on 26 ml serum bottle.
Insert stopper.
Fit cap over stopper.
Fit crimper over cap.
Hold crimper horizontal.
Squeeze ***gently*** to crimp.
Test by trying to rotate cap.
<u>If rotates with difficulty, is OK</u>
If very loose, re-squeeze.

Filling and Capping 1-Liter Serum Bottles in the Field

1. 1-Liter bottles are filled with Argon and may contain a small amount of black reducing agent.

2. Remove old aluminum cap using pliers.
   Remove stopper and save.

3. Use Low-Flow Sampling Procedure.
   Insert tubing a few inches and hold in place

4. Fill bottle slowly. Argon is heavier than air so will continue to 'blanket' the groundwater during filling. Do not overflow. Fill completely.

5. Note that 1-Liter Serum Bottles are *not as strong* as the 26 ml and 160 ml bottles. Do not tilt crimper or squeeze too hard.

6. Apply stopper and cap, then crimp as described for practice.

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Oct 20 2008

Standard Operating Procedure for BCI Functional Test for *D.ethenogenes* (Presence-Absence Test)

This information is supplementary to that contained in BCI SOP #14, Microcosm Tests.

Purpose and Introduction

Purpose of the Presence-Absence Test.

The purpose of this BCI functional test is to provide a positive identification, in site ground water, of live, active cells of *D.ethenogenes* (DE), the only bacterial species capable of reductively dechlorinating DCE to VC, and further dechlorinating VC to ethene.

D. ethenogenes performs the following dechlorination under anaerobic conditions having strong negative reducing conditions, -180 mv or lower, and when molecular H_2 is provided.

$$DCE + 2\,H_2 \rightarrow VC$$
$$VC + 2\,H_2 \rightarrow \textbf{ethene}.$$

Live, active *D.ethenogenes* are detected by measuring the **production of ethene** in a sealed microcosm bottle containing the site ground water to be tested. Dead DE will not produc ethene.

Brief summary of microcosm construction and anlysis.

Ground water is collected using anaerobic technique. At BCI ground water is transferred to microcosm bottles using anaerobic technique, leaving an anoxic headspace, and sealing with a septa through which amendment can be added by syringe.

The ground water in the microcosm bottle is brought to optimal conditions for *D.ethenogenes* by amending with minerals required by bacteria for growth, and with a mixture of organic compounds that will be degraded by anaerobic bacteria to provide the H_2 needed by declorinating bacteria. In addition, vitamin B_{12} is added, and the pH is adjusted to 6.9.

The progress of dechlorination is determined by removing 100 µL headspace samples by syringe and injecting directly into a gas chromatograph and analyzing according to EPA Method 5021A. Data for chlorinated compounds and ethene are expressed on a molar basis. Comparing start and finish analyses, the mols of DCE dechlorinated must equal mols of ethene produced.

Other types of Dechlorinating Bacteria

PCE/TCE dechlorinators. In addition to *D.ethenogenes*, methanogens, sulfate-reducers, and acetogens can dechlorinate PCE and TCE to DCE, but can not dechlorinate DCE.

Defective strains of DE. This functional test is able to identifiy defective strains of DE which can not *completely* perform both of the steps shown above (e.g. strain 195). If an 'incomplete dechlorinator' is present, VC will accumulate in the microcosm.

Other Information gained from the functional microcosm Test.

The BCI P-A functional test will also give information about other important members of the microbial community associated with dechlorinating bacteria, such as:

Sulfate-reducing bacteria (based on SO_4 reduction in the microcosm bottle)
Methanogens (based on production of methane).

This information is important since methanogens and some SRB produce vitamin B_{12} which is needed by *D.ethenogenes*. The test also shows whether the site water contains active bacteria that can metabolize donor .

Limitations of the BCI Functional P-A Test.

The functional test is simplified to use as a screening test. For a P-A Test, only one killed control and one positive control are provided for every batch of 10 well samples.

A standard experimental microcosm test is conducted by constructing a minimum of three microcosms for every ground water sample:

(1) Experimental: groundwater amended as described above for the P-A test
(2) Negative Control: pH adjusted to 2 to kill bacteria
(3) Positive Control: Amended ground water inoculated with BCI *D.ethenogenes* culture.

The purpose of the negative control is to detect abiotic degradation of contaminants, as well as to show evidence of containment (no leaks). The purpose of the positive control is to document that our techniques preserve anaerobic conditions and support activity of *D.ethenogenes*.

PA Microcosm Functional Test for *D.ethenogenes*, Construction and Analysis

- Microcosms are constructed using ground water that has been collected according to strict procedures described by BCI. The ground water is transferred in the field to special proprietary containers which are filled at BCI with Argon to exclude air and contain a reducing agent to eliminate traces of air that might be introduced during sampling. These bottles are sealed with crimped caps and shipped to BCI with priority overnight delivery.

- At BCI, 100 ml ground water from each well is transferred to one 160 ml microcosm bottle using proprietary anaerobic techniques. The bottle is sealed with a septum which allows samples to be removed and added by syringe.

- Initial Analyses of Microcosm bottle and calculation of donor amount.
  - Analytical procedures are described at the end of this section.
  - 150 μL of ground water are removed from the microcosm bottle by syringe and analyzed by capillary ion electrophoreses for sulfate, nitrate, and native organic acids.
  - In addition, 100 μL of headspace is analyzed for ethene, methane, and chlorinated compounds by gas chromatography.
  - This information is used to calculate the amount of donor needed to reduce sulfate and nitrate and to dechlorinate the chlorinated contaminants.

- Addition of amendments and pH adjustment
  - Stock solutions of the three 'most successful' donors are added to the microcosm. These are sodium lactate (organic acid), emulsified soy oil (hydrocarbon type), and high fructose corn syrup (sugar type). The amounts added are such that if only one donor type is utilized by the site bacteria, there will be sufficient donor.
  - In addition, stock solutions of minerals needed by bacteria are added, including ammonium chloride, K and Na Phosphate, and other elements.
  - pH is adjusted to 6.9 by adding KOH or HCl
- Final analyses.
  - After at least one week, the microcosms are analyzed for sulfate, nitate, and organic acids by removing 150 μL and analyzing by capillary ion electrophoresis.
  - After 30 to 60 days, depending on the concentrations of chlorinated compounds and sulfate, the microcosm is analyzed for dissolved gasses by gas chromatography.

Reporting.

Reports will include a statement of methods, a table containing start and finish GC and anion results, and a brief discussion of implications for further sampling and testing.

Analytical Procedures.

Microcosms are monitored for chlorinated compounds by removing 100 μL samples of microcosm headspace and injecting into a HP5890 gas chromatograph according to EPA Method 5021A. Standards are prepared similarly, and analyzed in the same manner as samples. ChemStation software is used to calculate response factors and quantitate results. *Concentrations reported are those that would be present if each compound were completely in the aqueous phase* (not partially in the headspace). Standards are analyzed daily.

Nitrate, Sulfate and organic acids are determined by removing 150 μL aqueous samples and analyzing by capillary ion electrophoresis according to EPA Method 6500 (which does not separate lactate and propionate). Compounds are identified by retention time ratio in comparison with standards analyzed with each batch of samples. Response factors are calculated and results quantified by Waters Empower software. Selected samples are analyzed by Metrohm Peak Ion chromatography to obtain separate values for lactate and propionate.

<u>pH</u> is determined by removing 150 μL aqueous samples by syringe and measuring with a ThermoOrion model 290A pH meter with a Sure-flow Ross semi-micro electrode.

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Standard Operating Procedure

 # 10

Anaerobic Procedures

April 20, 2001

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BCI SOP # 10
Anaerobic Procedures

1 Scope and Application

Anaerobic procedures are designed to exclude oxygen from biological experiments involving anaerobic bacteria. In addition, these procedures are used to exclude oxygen from field samples to be analyzed for reduced analytes.

2 Summary of Method

Containers to be used for anaerobic procedures are flushed with oxygen-free gas. Traces of oxygen are removed from compressed gasses by passing through heated copper shavings. Ground water is treated with reducing agents to remove oxygen. Stock solutions are prepared using boiled water, sparged to remove residual oxygen, and protected with reducing agents. Stock solutions and experimental vessels are sealed with Teflon-lined gray butyl rubber stoppers affixed with crimped aluminum caps. Handling of soil is carried out in an anaerobic glove box.

3 Apparatus and Materials

- Copper turnings in glass tube wrapped in heat tape with voltage regulator.
- Thermister and flow meter.
- Anaerobic storage chamber and anaerobic glove box as per BCI SOP # 15
- Serum bottles, Teflon-faced butyl rubber septa, aluminum caps, crimper, de-crimper, gas tight syringes and needles, sodium sulfide and ferrous chloride

4 Procedures.

The procedures are modifications of Hungate (1950), and are also discussed in Breznak and Costilow (1994)

4.1 Removal of trace oxygen from compressed gasses.

- Argon (99.999 % oxygen-free) and 30% CO_2 / 70% N_2 (99.996 % oxygen-free) are used.
- Copper turnings are packed into a glass tube wrapped in heat tape at 350°C .
- Gas is passed through copper turnings.
- The temperature of copper is checked using thermister probe.
- To verify oxygen removal, 200 volumes of treated gas is passed through boiled water then tested for dissolved O_2 using micro-Winkler, or using a suitable redox indicator.
- Copper is exhausted when the color appears green/black. Copper is regenerated by passing a mixture of 5 % H_2 in Argon (99.999 % oxygen-free) through the heated tube until the original color is restored.

4.2 Soil Handling, Storage and Transfer to Microcosms.

- Soil cores will remain enclosed in brass tubes, which will be immediately sealed and shipped to the BCI Laboratory, where they will be stored in special anaerobic chambers maintained with 5% H_2 in argon.
- For soil preparation, the cores from a given boring will be opened in an anaerobic glove box, sieved, and mixed.
- Working in the anaerobic glove box, a weighed quantity of soil composite will be put into each microcosm bottle, and the bottle stoppered.
- Remaining soil will then be placed in argon-flushed glass jars with Teflon-lined lids and returned to the anaerobic storage chambers.

4.3 Storage of Ground Water Samples

- Ground water samples may become aerated during sampling, or may be shipped with an air bubble. In the laboratory, sulfide is added to react with the oxygen, then ferrous iron is added to identify residual sulfide by forming a black precipitate.
- Upon receipt, sodium sulfide is added as needed, from an (oxygen-free stock solution), to give about 3.5 ppm sulfide (0.1 mM).
- A black precipitate of FeS forms if oxygen has been removed and excess sulfide remains.
- If FeS does not form, additional sulfide is added.
- The sample container is inspected after 1 hr and additional sulfide is added if necessary.

4.4 Transfer of Ground Water to Microcosm Bottles

- This procedure is conducted in a fume hood.
- The field bottle is flushed with copper treated ultra pure Argon, directing the flow to the surface of the water in the field bottle.
- Simultaneously, a second source of argon is directed into a serum bottle, which has been flushed with three volumes of Argon.
- Argon is used because it is heavier than air and will stay in the bottles, excluding air.
- While flushing, 50 ml of ground water is drawn up slowly from the field bottle into a 60 ml syringe and transferred to the microcosm bottle. This step is repeated.
- Then both bottles are flushed with 3 headspace volumes of Argon and capped.

4.5 Sparging of Microcosms to Remove Chlorinated Volatiles

- This procedure is conducted in a fume hood.
- The microcosm is sealed with a butyl rubber stopper.

- Using a long needle through the stopper, ultra pure, copper treated, gas (30% CO_2 in N_2) is directed to the bottom of the serum bottle, at 100 cc/min.
- Tubing, 1 mm ID, is inserted 1/4" through the stopper to allow exit of the purge gas.
- Sparging time is determined by the concentration of the analyte having the lowest Henry's constant. To determine if sparging is sufficient, a septum cap is applied, the microcosm is shaken 5 min, and equilibrated by standing 1 hr, then analyzed by GC headspace method.
- Quality Control: If the sparging gas is not oxygen-free, the black ferrous sulfide precipitate in the microcosm will disappear as sulfide becomes oxidized to sulfate. If this occurs, more sodium sulfide and ferrous chloride will be added to the microcosm, and the oxygen removal system will be checked for malfunction.

4.6 Preparation of Stock Solutions.

- Boiled water is transferred to an argon-flushed serum bottle and sparged with 5 volumes of copper- treated argon.
- Reagents are added and sparged with 15 volumes of copper-treated argon.
- The bottle is sealed with Teflon-lined butyl rubber and crimped cap
- Na_2S and $FeCl_2$ are injected to give 0.4 mM each
- The headspace is over pressurized, bottle labeled with contents & date, and refrigerated.
- Details are entered into 'Media & Stock Solutions' Log Book

4.7 Preparation of Spiking Solutions.

- Magnetic stirring bar is placed in a 250 ml septum-cap bottle.
- Boiled, sparged DI water is added, leaving 5 ml headspace,
- The headspace is flush, and cap applied.
- 1 ml of neat compound is injected through the septum.
- Na_2S and $FeCl_2$ are injected to give 0.4 mM each.

- The mix is stirred overnight. The droplet of neat compound should be visible.
- To spike a microcosm, oxygen-free gas is injected, and an equal volume of spiking solution is withdrawn and injected into microcosm.

4.8 Addition of Stock Solutions to Microcosms.

- A syringe is flushed with oxygen-free gas
- The oxygen-free gas is injected into stock bottle.
- The same amount of stock solution is withdrawn.
- The stock solution is injected into the microcosm bottle.
- The needle is flamed

4.9 Removal of Sub-samples from Microcosms.

- Microcosms are stored on their sides, with septa submerged, but not contacting sediment.
- For Headspace Samples, the microcosm is placed in upright position. A 100 μL gas-tight syringe is flushed twice with O_2-free gas, then inserted thru the septum. The syringe is flushed twice with headspace, then filled and withdrawn.
- For Liquid samples; the microcosm remains on its side.
  A 1 ml plastic syringe with 26 gauge needle is used to withdraw 0.1 to 0.3 ml
  The syringe is flushed twice with oxygen-free gas as above.
  Gas is injected into the microcosm and liquid is withdrawn.

5 *References*

Breznak, J.A. , and R.N. Costilow. 1994. Physicochemical Factors in Growth. Chapter 6, in Methods for General and Molecular Bacteriology. P. Gerhardt, ed. American Society for Microbiology, Washington DC.

Hungate, R.E. (1950). The anaerobic mesophilic cellulolytic bacteria. Bacteriological Reviews. vol 14, page 1-49.

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Standard Operating Procedure

 # 11

Quantitation of Dissolved Anions

by

Capillary Ion Electrophoresis

EPA Method 6500

April 20, 2001

Prepared by: _____ _____
Margaret Findlay, Ph.D., President *Date*

Reviewed by: _____ _____
Donna Smoler, Laboratory Manager *Date*

Approved by: _____ _____
Samuel Fogel, Ph.D. Vice President *Date*

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BCI SOP # 11

Anions by Capillary Ion Electrophoresis

EPA Method 6500

1 Scope and Application.

Capillary Ion Electrophoresis is used to determine the concentration of inorganic and organic ions in aqueous solution. Analytes routinely determined at BCI include chloride, sulfate, nitrate, nitrite, formate, acetate, propionate, and butyrate. The method is applicable to ground water samples as well as sub-samples from laboratory microcosm tests.

2 Summary of Method.

A sample to be analyzed for anions is diluted with ultra pure water as necessary to obtain the analytes in the working range of the method, and is placed into the instrument, where the sample is drawn into one end of a capillary. The ends of the capillary are then placed in electrolyte solution and a high voltage is applied across the capillary, causing the anions to migrate toward the anode. During migration, the anions are separated mainly due to charge-to-size ratio. The separated anions pass by a UV source and detector. Identification of analytes is based on migration times, and quantitation is based on analysis of standards.

3 Interferences.

High concentrations of anions in the sample influence the electrophoretic mobility of other anions, making it necessary to dilute the sample so that the most concentrated anion is within instrument range.

Silicates originating in laboratory glassware migrate in the range of analytes, causing false positive results. Therefore, dilution water, stock solutions , and sample dilutions are placed in plastic containers, and transfers are made using plastic autopipet tips.

4 Apparatus and Materials.

Waters Capillary Ion Analyzer, (Waters, 34 Maple St, Milford, MA, 01757)
Waters Millenium 2010 (or later) Chromatography Software.
PC computer and printer.
Capillary column (Polymicro Technologies Inc, Phoenix AZ, part # 2000019)
Waters IonSelect™ High Mobility Anion Electrolyte
Inorganic Anion Standard Solutions, 1000 ppm (Analytical Products Group, Belpre, OH)
Organic Acid Standard Solutions, 1000 ppm (AccuStandard Inc. , New Haven CT)
Plastic vials, autopipet, Eppendorf vials
Ultrapure Water.

5 Sample Collection, Preservation, Handling, and Holding Time

Field Samples. Samples are collected in 40 ml vials and shipped to BCI with ice by priority overnight express. Samples are usually not preserved with acid or alkali because the electrophoresis separation is effected by the pH of the samples. In addition, adding HCl as a preservative results in incorrectly high chloride values, and may require dilution of the sample, which lowers detection limits. After receipt, samples are kept refrigerated. Since samples are not preserved, analysis is usually carried out on the day received, or within 48 hours.

Microcosm Samples. Samples are withdrawn from microcosm bottles using a 1 ml syringe through the septum, according to BCI SOP # 10, and analyzed immediately.

6 Procedure.

The Waters Capillary Ion Analyzer is operated according to the Waters Capillary Ion Analyzer Operator's Manual. Procedures are also described in EPA Method 6500.

Capillaries are prepared by cutting capillary wire, burning a UV 'window', and rinsing, according to the Owners Manual. The capillary is replaced approximately weekly.

Ultra pure water is stored under refrigeration.

Standard mixtures are prepared every 2 weeks by diluting the 1000 ppm stock standards with ultra pure water, so that each compound is within its linear range, as well as within the range of the (diluted) samples to be analyzed. The standards are stored under refrigeration.

Typical standard mixtures for a high-chloride site are: 30-80 ppm chloride, 5-15 ppm each sulfate, formate, acetate, propionate, and butyrate, and 2-5 ppm each nitrite, nitrate, and oxalate.

Routine analyses are conducted using chromate electrolyte (Waters IonSelect High Mobility Ion Electrolyte). The phosphate electrolyte is employed for samples containing benzoate, and to confirm identification of certain analytes.

A new standard curve is prepared daily, and whenever a capillary is replaced.

Samples are diluted with ultrapure water, using plastic vials and plastic autopipet tips. After analysis, if analyte interference is observed, samples are diluted to a greater extent and re-analyzed.

Analytes are identified by Migration Time Ratio, (the migration time of the analyte relative to the migration time of the fastest-migrating component, usually chloride).

Although the MTR are determined daily by analyzing the standard mixture, analyte migration times can be altered in a given sample by the presence of other analytes. Therefore, when analyte identity is in question, the sample is re-analyzed with an internal spike of known compounds.

Compounds are quantitated by the Millenium software, using the response factors generated by the analysis of the daily standards.

For every sample, the raw data and the quantitated data, are stored electronically. Information stored for every sample includes : date of analysis project name, sample designation, dilution, designation of the standards and their date of preparation.

7 *Quality Control*

- Working standards are made every 2 weeks by diluting purchased stock standard solutions.
- A Standard curve is prepared daily.
- A QC standard is analyzed with every batch of samples, and recorded in a QC standard log.
- A log book is kept into which each sample is entered, including project, sample designation, dilution and analyst, each standard preparation is detailed, newly purchased standards are noted, and electrolyte and capillary changes are noted.
- A DI water blank (and trip blanks and field blanks when provided) are analyzed.
- Duplicates are analyzed with every batch of 12 samples.

8 *Method Performance.*

Detection Limits: Practical Quantitation Limits using the chromate electrolyte (for analytes that are baseline-resolved) are 0.1 ppm. The PQL are increased proportionately if the sample is diluted. Thus, for a 10-fold dilution, the PQL is about 1.0 ppm.

Migration Time Ratios. The migration time of an analyte changes very slightly with the age of the capillary and of the electrolyte. Typical migration times are listed in Table 1.

Analyst Performance. An analyst must demonstrate the ability to obtain values for the QC standard within 5% of the running values. An analyst must also be able to analyze calibration standards, obtaining a correlation coffecient of at least 0.99 for each analyte.

Table 1
Typical Migration Times (Minutes) for Selected Analytes
Waters Capillary Ion Electrophoresis

	Chromate Electrolyte	Phosphate Electrolyte
Chloride	3.16	3.05
Bromide	3.24	3.02
Nitrite	3.28	3.13
Sulfate	3.40	not analyzable
Nitrate	3.51	3.22
Oxalate	3.61	3.36
Fluoride	3.74	-
Formate	3.79	3.78
Phosphate	3.92	not analyzable
Carbonate	4.20	4.75
Acetate	4.45	4.52
Prop/Lac	4.78	4.89
Butyrate	5.06	5.15
Benzoate	not analyzable	5.41

9 *References*:

Waters Capillary Ion Analyzer, Operator's Manual.(1995). Waters Corporation, 34 Maple Street, Milford MA 01757.

Krol, J. M. Benvenute, and J. Romano. (1998) Ion Analysis Methods for IC and CIA and Practical Aspects of Capillary Ion Analysis Theory. Waters Corporation, 34 Maple St, Milford MA 01757

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Standard Operating Procedure

 # 12

Measurement of Dissolved Gasses and Volatile Organic Compounds in Water Samples by Analysis of Headspace Samples Using Gas Chromatography with Flame Ionization and Thermal Conductivity Detection

Revised Aug, 2005

Prepared by: _____ _____
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Measurement of
Dissolved Gasses and Volatile Organic Compounds in Water by
Analysis of Headspace Samples Using
Gas Chromatography with Flame Ionization Detection
And Gas Chromatography with Thermal Conductivity Detection
EPA Method 5021A

1 Scope and Application.

1.1 EPA Method 5021A is applicable to volatile organic compounds in water. Using a flame ionization detector (FID) with a gas chromatograph, both chlorinated and non-chlorinated compounds can be detected. The method is applicable to low molecular weight compounds such as dissolved gasses (methane, ethene, acetylene, ethane, propene) as well as higher molecular weight volatile compounds such as xylene and trichlorobenzene. Using a Thermal Conductivity Detector (TCD) with the gas chromatograph, the method is applicable to CO_2 analysis.

1.2 The practical quantitation limit for samples analyzed by the 'Vial Method' at 21°C ranges from less than 1 μg/L for dissolved gasses, to 100 to 300 μg /L for compounds having low Henry's Law Constants. The headspace method can be applied to any water sample in a septum-capped filled container. At BCI, two procedures are routinely employed, – the Vial Method, and the Serum Bottle Method.

2 Summary of Headspace Method

2.1 *Theoretical Basis.* The method is based on the concept that in any closed container having a headspace and an aqueous portion, volatile compounds will distribute between the two matrices according to their Henry's Law Constants. A measurement of an analyte in a headspace sample can be used to determine the amount in the aqueous portion.

2.2 *Approach*. A standard is created in a closed container having both headspace and water by injecting a known mass of analyte through a septum, and allowing an equilibrium to develop. Analysis of a headspace sample from that container will produce an instrument response proportional to the mass of the compound injected. If an unknown sample is prepared in a similar container with the same headspace and liquid volumes, the mass of that compound can be determined by analyzing a headspace sample, and utilizing the response factor obtained by analyzing the standard.

2.3 *Gas Chromatography with Direct Injection*. A gas sample containing volatile compounds is injected into a capillary column in a gas chromatograph with flow conditions as described below, the compounds travel down the column with the carrier gas, partitioning between the column and carrier gas stream, and are separated roughly on the basis of molecular size with some sensitivity to polarity. Combustable compounds are detected by the FID transmitting a signal to the integrator which records a response (area) proportional to the mass of the compound.

2.4 *Application*. At BCI, 43 mL vials are used for field samples of groundwater, and 160 ml serum bottles are used for microcosm studies.

3 Interferences and Limitations.

3.1 *General Interferences*.

- In general, the headspace method is free from interferences from high molecular weight compounds because they do not volatilize from the water sample, and are not present in the headspace sample.
- The presence of non-aqueous phase liquid, either as droplets or as a free phase, will trap volatile compounds, removing them from solution and making them unavailable for volatilization.

3.2 *Interference in Measurement of Dissolved Gasses*

- For dissolved gasses, there are no other compounds that have similar retention times.
- However, large amounts of ethene or ethane can obscure small amounts of acetylene.

3.3 *Interference by Soil.*

The presence of sediment having high Koc will interfere with the analysis of higher molecular weight compounds by adsorbing both chlorinated volatiles (such as PCE) and non-chlorinated compounds such as benzene.

3.4 *Advantages.* The advantage of the method is that detection limits are lower for the lighter compounds than can be achieved by Mass Spectrometry, and mixtures having wide ranges of analyte concentrations can often be analyzed without dilution.

3.5 *Limitations.* Analytes are identified by retention time only, therefore when two compounds have the same retention time (and similar peak width), the identity of a peak must be determined by mass spectrometry. Pairs of analytes having the same retention times with the column type and temperature program used by BCI include: (1) carbon tetrachloride and 1,1,1-TCA, and (2) PCE and 1,3-Dichloropropane.

4 Apparatus and Materials

HP 5890 Gas Chromatographs, Series I or II,
equipped with Flame Ionization Detector and Thermal Conductivity Detector.

HP Chemstation with PC computer and Printer, or HP Integrator/ Printer HP3396A.

Appropriate Column.

Hamilton Gas Tight Syringe.

Standard Gas Mix, 1% ppmv each methane, ethene, acetylene, ethane, CO_2 . Scott Gas.
100 % CO_2 , 100 % methane, 100 % Helium, Scott Gas.

Standard Analytes (see Table 1) in methanol, 2 to 10 mg/ml. Restek.

5 Sample Collection, Preservation, and Handling.

Vials with septum caps, containing 250 µL 6N HCl, are shipped to the field. Ground water is added to overfull, and caps replaced. Samples are shipped to BCI, chilled, by overnight express, then refrigerated, and analyzed within 7 days.

6 Procedure.

6.1 GC Conditions

Temperature Program

- Initial temperature, 35°C
- Initial time, 5 min
- Ramp, 8 degrees per minute.
- Final Temperature, 240°C

GC gasses

All gasses are high purity (99.999% pure)

Carrier gas, helium

FID gasses, H_2 and air.

6.2 Vial Method.

- In the laboratory, the vials, containing 43 ml of ground water, are brought to room temperature, then using two syringes, 5 ml of water are displaced through the septa while 5 cc of Helium are injected. The vials are shaken for 20 minutes on a reciprocal shaker allowing the volatile compounds remaining in 38 ml of liquid to volatilize into the headspace portion.

- Standards are prepared in 43 mL vials with 5 cc headspace, in a similar manner.

- Headspace samples of the standards, 100 μL, are withdrawn with a syringe and injected into the gas chromatograph. A response factor R (concentration in mg /L per instrument response in area counts) is generated for each compound. This response factor applies only to this container and the stated volumes of gas and aqueous phase, and the stated injection volume. Concentrations are recorded in terms of the entire mass of analyte in the aqueous phase. For Each Analyte:

  A = Instrument response in terms of area counts

  R = Response Factor in terms of mg/L/A

- 100 μL of headspace from a field sample is injected into the GC. The area counts generated for each analyte are multiplied by the Response factor

  $$mg/L = (R)(A)$$

- This application of the headspace concept is based on one developed in the EPA laboratory in Ada, OK (Kampbell et al. 1998).

6.3 *Serum Bottle Method.*

- The Serum Bottle Method of headspace analysis is used for repeated analysis, over a time period of several months, of microcosms in which the biodegradation of selected analytes in ground warter is being evaluated.

- The bottles to be sampled have a volume of 160 ml, contain 100 ml of groundwater and 60 cc of headspace, and are sealed with a septum cap. The analytes in the serum bottle are distributed between the water and the headspace.

- Standards are prepared in 160 ml serum bottles in a similar manner, and analyzed daily. Headspace samples of the standards are analyzed by injection into the GC to generate response factors specific to this ratio of headspace to aqueous phase.

- Headspace samples are removed from the microcosms by syringe and injected into the GC, and quantitated using the response factors.

- Results are expressed in μM, as though the entire mass of analtye were dissolved in the 100 ml aqueous portion.

*7 **Quality Control.***

7.1 *Decontamination*. GC column is baked at 240°C for 20 minutes prior to every analysis. Syringe barrels and needles used for headspace sampling are baked at 120°C for 5 minutes prior to every analysis.

7.2 *Syringes*. Syringes used for concentrated standards or for neat compounds are kept in a separate location, and are not used for sampling headspace. These are rinsed repeatedly with methanol prior to baking.

7.3 *GC Log Book*. Every analysis is recorded in a log book, with project name, sample code, run number, date, and analyst. Maintenance events are also entered in the log book. Data are stored electronically, and a hard copy of the chromatogram and data are stored in a project binder.

7.4 *Standards Log Binder*. A log is kept on the preparation of standards, including the concentration, source, date purchased, and expiration date of every analyte. Standards received in ampules, when opened, are used only once. Chromatograms and data from analyses of standards are stored in the standards binder.

7.5 *QC for Vial Method (Dissolved Gases)*. Dissolved gas standards are prepared at three concentrations in the expected range of the samples for each batch of 10-15 samples. If the response factors are not within 10 % of the expected values, new standards are made and analyzed. Vial standards are used only on the day they are

prepared. Sample values which exceed the linear range for any analyte are "diluted" by analyzing a smaller volume of headspace. A Trip Blank, Field Blank, and duplicate are analyzed for each batch of samples.

7.7 *QC for Serum Bottle Method (Dissolved Gasses and Volatile Chlorinated Compounds).* A project-specific standard is analyzed once a month, with the monthly analysis of the project batch. Killed control microcosms for every 'set' of bottles (containing water from the same well) are analyzed with each project batch, and checked with previous analyses for consistency.

8 Method Performance

Method performance is described in Table 1, which lists Retention Times and typical Practical Quantitation Limits for a range of analytes from methane to trichlorobenzene for both the Vial Method and the Serum Bottle Method. If the analysis of standards results in retention times (RT) that differ by more than 0.05 minutes from the running RT, or response factors (RF) differ by more than 15% from the running RF, the standard will be re-analyzed and instrument flow parameters and temperature program will be checked.

9 Safety

Compressed gas tanks are chained and belted to tank brackets affixed to a wall in a garage/storage area on the other side of the laboratory wall. The gasses are plumbed into the lab via 1/8 ID copper tubing and connected directly to the GCs.

10 References

- Kampbell, D. H. and S. A. Vandegrift. 1998. Analysis of dissolved methane, ethane, and ethylene in ground water by a standard gas chromatographic technique. J. Chromatographic Science, vol 36, page 253-256.
- EPA Method 5021A.

Typical Retention Times of Selected Compounds.

Headspace method Samples at 21°C. FID

R.T. min	***name***
0.9	methane
1.4	ethane
1.8	ethene
3.2	acetylene
7.0	propene
4.1	propane
7.9	chloromethane
8.2	Chloroethene (VC)
11.4	butene
11.8	butane
10.6	chloroethane
11.0	dichloromethane
10.0	1,1-dichloroethene
11.2	t-dichlroethene
13.7	c-dichloroethene
13.9	chloroform
14.6	carbon tetrachloride
16.0	1,1-dichloroethane
16.6	trichloroethene
18.5	1,1,1-trichloroethane
19.4	1,2-dichloroethane
19.6	tetrachloroethene
19.7	benzene
21.2	1,2-dichloropropane
21.6	toluene
22.7	ethylbenzene
22.9	m&p xylene
23.1	o xylene

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Quality Assurance Plan

Approved _____

Margaret Findlay Ph.D.
Laboratory Director

Date _____

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1. INTRODUCTION, OBJECTIVE AND SCOPE

Introduction

Bioremediation Consulting Inc (BCI) is committed to consistent delivery of the highest quality data. To achieve this level of performance, BCI has developed a Quality Assurance (QA) program, which will ensure the timely production of data that are precise, accurate, complete and representative. This program is administered by the Laboratory Director.

QA. Quality Assurance is the management system which operates to ensure that the results and conclusions produced are accurate and reliable. QA includes such measures as the development of Standard Operating Procedures, documentation of sample history, and design of data review and approval procedures. The QA plan also ensures that the information produced is interpreted by qualified personnel who are trained in the appropriate disciplines.

QC. Quality Control refers to the specific system of analytical tests and checks to assure that the data being generated conform to the QA standards, and to document that fact for review. QC includes the establishment of criteria for calibration and for analysis of QC samples such as blanks, duplicates and spikes. QC activities are designed to ensure the accuracy, precision, representativeness, and comparability of results.

Objective and Scope

This document describes the specific procedural methods of Quality Assurance and provides written procedures and guidelines that allow the achievement of QC goals.
The objective and scope of the QA program include:

- Provision for review of all technical products to ensure that documentation provides accurate and acceptable information.
- Coordination of quality assurance and quality control (QA/QC) procedures which provide a documentable, consistent level of quality for analytical measurements.
- Coordination of QA project plan (QAPP) development to ensure the adequacy of the technical product specified by a client, as to both the technical content and the document control procedures that ensure its security.
- Identification of QA/QC deficiencies and coordination of expeditious resolutions.
- Assurance of systematic, timely revisions of QA/QC procedures as needed.
- Responsibility for documentation of precision, accuracy, and origin, to ensure that data are scientifically valid and defensible.

2. QUALITY ASSURANCE POLICY

The policy of BCI is to maintain an active QA program which provides the highest quality data and results to achieve the goals of each activity undertaken. To accomplish this goal, the following QA responsibilities are defined:

- The BCI Laboratory Director has technical and administrative responsibility for the overall QA program.
- The BCI Laboratory Director has the authority to implement action to document and correct any situation having a negative impact on the quality of data or on the documentation related to its intended use.
- The BCI Laboratory Director consults frequently with project and laboratory managers.
- The BCI Laboratory Director refers all QA issues or disputes that cannot be resolved within the Laboratory to the Vice President.
- The QA program is implemented by qualified personnel.
- Facilities, equipment, and services are routinely inspected and maintained.
- Specified quality control guidelines set for each laboratory task must be met.
- Analytical data is collected using Standard Operating Procedures (SOPs) developed by BCI.
- Work plans and work flow diagrams are developed to define the scope of major activities.
- The levels of documentation and QC are defined for all analytical measurements.
- Acceptance criteria for the quality of the data are specified in the BCI Quality Assurance Plan and in SOPs.
- The precision, accuracy, representativeness, and completeness of all data are defined and documentation is maintained on file.
- Data processing procedures are documented, routinely reviewed, and revised as necessary.
- QA management implements review of technical operations.
- A mechanism for identifying QA problems and expediting corrective actions is maintained.
- The Lab Director works with project managers to develop, revise, and implement mechanisms as needed.

3. QUALITY ASSURANCE MANAGEMENT

3.1 Organization and Assignment of Responsibilities

The Laboratory Director is responsible for the development and administration of the Quality Assurance Program and is responsible for the quality of all work performed. The Laboratory Director is also responsible for providing direction and guidance to each of the project managers, and for scheduling review of the technical efforts. The Laboratory Director's specific responsibilities include:

- Meeting with the project managers.
- Developing and revising the QA program, as required.
- Supervising the QA responsibilities of other members of the staff.
- Coordinating and auditing QA review of documentation, as required.
- Providing recommendations and orders, as required, for corrective action for all aspects of work that do not meet QA standards.
- Identifying nonconformance situations to management, as needed.
- Ensuring compliance with corrective action requests. Maintaining and updating qualification records for all laboratory personnel.
- Assigning senior personnel to train new personnel.

3.2 Sample History, Data Tracking and Calculations

Provision of Containers and Receipt of Field Samples

- Sampling containers will be provided with appropriate preservative or reducing agent as required for the requested analytical procedures. Upon arrival at BCI, sample label information will be checked against the Chain of Custody record, the condition of the samples will be noted, the record will be signed and dated, and the COC will be filed in the Project Binder. Copies of the COC, which includes the samples' testing requirements, will be distributed to the appropriate task managers.

Laboratory Data Tracking and Calculations

- For each sample, its unique sample code, sample preparation, analysis date, and analyst are recorded directly in method-specific logbooks. Such notebooks include those kept for the following procedures: GC analysis, Capillary Ion Electrophoresis, H_2 analysis, inorganic analysis by chemical kits, and Use of Compressed Gas. Sample code, preparation information and date are also entered into the computer associated with most analytical instruments. Associated blanks, standards, and spiked samples are recorded in the same manner.

- For most analyses, primary data printed by the instrument are placed in the Project Binder. For other analyses, primary data are recorded by the analyst in a log book, and photocopied for placement in the Project Binder. Accuracy and completeness of the primary data is then reviewed by the Project Manager, who then signs the primary data sheet. Corrections are made by crossing a line through the error and entering the correct information with date and initials.

- Primary data requiring data reduction and calculations are usually entered into a computerized data handling system. The computer-printed data worksheet and results are then submitted to a QA reviewer (assigned by the Project Manager) for QC check, signed and placed in the Project Binder. Hand calculations are recorded and reviewed similarly.

3.3 Data Validation and Reporting

Data Validation

Data Validation is the process by which analytical data are reviewed and either accepted or rejected. All data are validated by laboratory supervisors prior to being released for reporting. The persons responsible for validating the data will have sufficient knowledge of the technical work to identify questionable results. BCI personnel use the following criteria in the validation of laboratory and experimental data.

Validation of Analytical Data

- Use of published or approved analytical procedures.
- Use of properly operating and calibrated instrumentation.
- Precision and accuracy meeting project-specific criteria.
- Absence of contamination in blanks.
- Completeness of data set.

Validation of Experimental Results for Microcosms

- Lack of changes in killed control microcosms
- Conversion of parent compounds to daughter products in molar amounts
- Use of electron donor compounds concomitant with reduction of electron acceptors.
- Non-changes in a conservative analyte such as (high concentrations) of chloride.

Data Reporting

Data Reports include:

- Sample results
- Statement of methods for each parameter
- Signed chain of custody
- Practical quantitation limits
- Duplicate results
- Blank results
- Matrix spike, as requested

3.4 Quality Control Checks

Quality Control is the routine application of procedures for obtaining prescribed analytical standards. Quality control checks are performed by project analysts under the guidance of the Laboratory Director. As needed, the Laboratory Director meets with the Laboratory Manager to review QC data summaries and discuss corrective action.

The following types of QC samples are used routinely:

- *Blank Samples* – Blanks are used to assess contamination introduced in transit, storage, or in the laboratory.

- *Matrix Spike* – as needed, samples are spiked with analytes in order to evaluate any matrix effect of the samples on the analytical method.

- *Duplicate Samples* –a second aliquot of a sample is analyzed to verify the precision of the analytical method.

- *Blind Samples*. If requested, the BCI Quality Assurance manager will carry out internal quality assurance audits in which blind samples are submitted to the laboratory for analysis.

- *Control Microcosms*. Typically, killed control microcosms are constructed with each batch of microcosms for a given field sample, in which the bacteria are killed by lowering the pH to 2. In addition, as appropriate for a given research project, additional control microcosms may be constructed, such as unamended controls. Furthermore, positive controls may be provided which are inoculated with known bacteria capable of defined biodegradation processes.

3.5 Performance Audits

An audit consists of an on-site review of a laboratory's quality assurance system and physical facilities for sampling, calibration and measurements. BCI will cooperate in such audits as requested by the client. The audit may include components listed below:

- Personnel, facilities and equipment
- Chain-of-custody procedures
- Instrument calibration and maintenance
- Standards preparation and verification
- Analytical procedures
- Quality control procedures
- Data handling procedures
- Document control procedures

3.6 Routine Maintenance

BCI follows a well-defined program to prevent the failure of laboratory equipment or instrumentation during use. This preventive maintenance and careful calibration help to assure accurate measurements from laboratory instruments.

Preventive maintenance procedures are performed according to the procedures delineated in the manufacturer's instrument manual, as well as when instrument performance begins to decline as evidenced by the decrease in instrument response or failure to meet one of the quality control criteria.

Gas chromatographic carrier gas purification traps, injector liners, and injector septa are cleaned or replaced on a regular basis.

Capillary ion electrophoresis capillaries, lamps and electrolyte are replaced on a regular basis.

Reduction Gas Analyzer lamp is replaced at intervals and the mercury bed is baked at intervals to remove contaminants.

The gas purification system for anaerobic procedures is tested regularly and regenerated as needed.

Instrument logbooks maintained in the laboratory contain records of usage, calibration, maintenance and repairs.

Adequate supplies of spare parts and supplies are maintained in the laboratory so that they are available when needed.

3.7 Assessment of Data Precision, Accuracy and Completeness

Precision. Precision is a measure of the degree of agreement between repeated measurements of the same parameter under prescribed, similar conditions. Analytical precision will be monitored using results from duplicate analyses.

Accuracy. Accuracy is the degree of agreement between the true value of the parameter being measured and the observations made according to the test method. Accuracy will be evaluated by analyzing standards on a regular basis, and by comparing results for standards obtained from different sources.

Completeness. For Analytical work, BCI's QA objective is to obtain acceptable data for all the samples received. The procedures in Section 3.3 of this QA document for validating data will be used to determine which data are acceptable. For research projects, BCI's objective is to provide experimental results which answer the questions defined in a research workplan. BCI's scientists are knowledgeable regarding bioremediation processes, and are experienced in data interpretation.

3.8 Corrective Action.

While the entire quality assurance program is designed to avoid problems, it also serves to identify and correct those that may develop.

Immediate Corrective Action. When inconsistent data, or results that do not meet quality control criteria, are discovered and corrected immediately by analysts, the problem and correction will be noted in the instrument log book and reported to the supervisor.

Long Term Corrective Action. The need for long-term action may be identified by supervisor's inspection of QC results, review of project data or review of long term system data. The process of establishing and implementing corrective action will then be undertaken by the supervisor working with the QA coordinator, according to the following steps:

- Identify the problem.
- Assign responsibility for investigating the problem.
- Determine of the cause of the problem and necessary corrective action..
- Assign responsibility for implementing the corrective action.
- Verify that the problem has been eliminated

4 SUMMARY OF STANDARD OPERATING PROCEDURES AND QC REQUIREMENTS

Detailed SOP Instructions (SOPI) and Performance Tests

- Detailed SOP Instructions (SOPI) are provided in a 3-ring binder available to BCI Staff.
- Individual SOPI are posted at each work station as appropriate.
- Each employee is required to receive individual instruction in each SOPI.
- The instructor is the Laboratory Director, Laboratory Manager, or a Senior Technician.
- Certain Procedures require Performance Tests to be completed by new personnel.
- A record is kept of each completed instruction session and test for each employee.

The following pages give brief summaries of the SOPI together with description of the associated Performance Tests.

BCI SOP 10 Anaerobic Procedures

Method References:

BCI Anaerobic procedures are modifications of Hungate (1950), and are also discussed in Breznak and Costilow (1994).

Hungate, R.E. (1950). The anaerobic mesophilic cellulolytic bacteria. Bacteriological Reviews. vol 14, page 1-49.

Breznak, J.A. , and R.N. Costilow. 1994. Physicochemical Factors in Growth. Chapter 6, in Methods for General and Molecular Bacteriology. P. Gerhardt, ed. American Society for Microbiology, Washington DC.

Procedure Summary and Performance Tests

BCI Detailed SOPI-10 for Anaerobic Procedures comprises the following ten activities, three of which involve Performance Tests.

10-1 Design of Sparging System with use of Copper Catalyst.

Compressed gas is directed via copper tubing with Swage-lok fittings, through a flow meter, then through a glass column packed with copper turnings, then to use such as sparging groundwater in microcosms.

10-2 Compressed Argon and N_2:CO_2 Certification and Use.

Each new tank of compressed argon or N_2:CO_2 is required to be certified in writing and dated by the distributor. The use of compressed gas regulators and flow through the column of copper turnings is described.

10-3 Regeneration and Repacking of Copper Turnings

Instructions for disconnecting the column, repacking if necessary, and regeneration the copper with 4% H_2 in argon are given.

10-4 Test for O_2 in Compressed Gasses

De-oxygenated water is sparged with 15 volumes of compressed gas which has been passed through heated copper turnings to remove traces of oxygen. (If oxygen remains in the gas, it will be transferred to the water.) The water is immediately tested for the presence of oxygen

using a Hach spectrophotometer method for Low Range oxygen measurements using Winkler the test.

Analysts are required to perform the sparging and Winkler test 3 times under supervision of a senior analyst and to obtain a value of < 100 ppb oxygen in the test water.

10-5 Preparation of Oxygen-Free Gas Bottles.

Instructions are given for preparing serum bottles of argon, including a redox indicator, by flushing with copper treated compressed argon.

Analysts are required to prepare such bottles which maintain anoxic conditions for 1 month.

10-6 Boiled, Sparged DI Water in Serum Bottles.

Instructions are given for preparing anoxic deionized water in serum bottles (which is later used to make up stock anoxic solutions).

10-7 Overpressurizing and Adding Stock Solutions to Anaerobic Serum Bottles

Instructions are given for flushing syringes with argon, overpressurizing serum bottles, and transferring stock solutions from stock bottles to microcosms.

10-8 Preparation of Amendment Solutions

Instructions are given for flushing, weighing analyte, and sparging anoxic amendment solutions under anaerobic conditions.

Analysts are required to analyze dilutions of stock solutions of formate and lactate using capillary ion electrophoresis, to confirm the concentrations.

10-9 Preparation and Use of Anaerobic Spiking Solutions

Instructions are given for anaerobic preparation of anoxic saturated solutions of PCE, cDCE and CT.

Analysts are required to analyzed dilutions of these stock solutions to confirm the concentrations of the analytes.

10-10 Preparation of Bottles for Anaerobic Ground Water, and Receipt of Samples.

Instructions are given for flushing sample bottles with argon and adding reducing agent / redox indicator. Additional instructions are given for inspection of groundwater samples returned in these sample bottles, and adding additional reducing agent if necessary.

BCI SOP 11 Anions by Capillary Ion Electrophoresis

11.1 Scope and Application.

Capillary Ion Electrophoresis is used to determine the concentration of inorganic and organic ions in aqueous solution. Analytes routinely determined at BCI include chloride, sulfate, nitrate, nitrite, formate, acetate, propionate, and butyrate. The method is applicable to ground water samples as well as sub-samples from laboratory microcosm tests.

11.2 Summary of Method.

A sample to be analyzed for anions is diluted with ultra pure water as necessary to obtain the analytes in the working range of the method, and is placed into the instrument, where the sample is drawn into one end of a capillary. The ends of the capillary are then placed in electrolyte solution and a high voltage is applied across the capillary, causing the anions to migrate toward the anode. During migration, the anions are separated mainly due to charge-to-size ratio. The separated anions pass by a UV source and detector. Identification of analytes is based on migration times, and quantitation is based on analysis of standards.

11.3 Interferences.

High concentrations of anions in the sample influence the electrophoretic mobility of other anions, making it necessary to dilute the sample so that the most concentrated anion is within instrument range.

Silicates originating in laboratory glassware migrate in the range of analytes, causing false positive results. Therefore, dilution water, stock solutions , and sample dilutions are placed in plastic containers, and transfers are made using plastic autopipet tips.

11.4 Apparatus and Materials.

Waters Capillary Ion Analyzer, (Waters, 34 Maple St, Milford, MA, 01757)
Waters Millenium 2010 (or later) Chromatography Software.
PC computer and printer.
Capillary column (Polymicro Technologies Inc, Phoenix AZ, part # 2000019)
Waters IonSelect™ High Mobility Anion Electrolyte
Inorganic Anion Standard Solutions, 1000 ppm (Analytical Products Group, Belpre, OH)
Organic Acid Standard Solutions, 1000 ppm (AccuStandard Inc. , New Haven CT)
Plastic vials, autopipet, Eppendorf vials
Ultrapure Water.

11.5 Sample Collection, Preservation, and Handling.

Field Samples. Samples are collected in 40 ml vials and shipped to BCI with ice by priority overnight express. After receipt, samples are kept refrigerated. For samples that are not preserved, analysis is usually carried out on the day received, or within 48 hours.

Samples for electrophoresis are usually not preserved with acid or alkali because the electrophoresis separation is effected by the pH of the samples. Also, adding HCl as a preservative results in incorrectly high chloride values, and may require dilution of the sample, which lowers detection limits. Preservation with sulfuric acid interferes with sulfate analysis.

Microcosm Samples. Samples are withdrawn from microcosm bottles using a syringe through the septum, according to BCI SOP # 10, and analyzed immediately.

11.6 Procedure.

The Waters Capillary Ion Analyzer is operated according to the Waters Capillary Ion Analyzer Operator's Manual. Procedures are also described in EPA Method 6500. Capillaries are prepared according to the Owners Manual. The capillary is replaced every few months.

Standard mixtures are prepared every 2 weeks by diluting the 1000 ppm stock standards with ultra pure water, so that each compound is within its linear range, as well as within the range of the (diluted) samples to be analyzed. The standards are stored under refrigeration.

Routine analyses are conducted using chromate electrolyte (Waters IonSelect High Mobility Ion Electrolyte). The phosphate electrolyte is employed for samples containing benzoate, and to confirm identification of certain analytes.

Samples are diluted with ultrapure water, using plastic vials and plastic autopipet tips. After analysis, if analyte interference is observed, samples are diluted to a greater extent and re-analyzed.

Analytes are identified by Migration Time Ratio, (the migration time of the analyte relative to tat of the fastest-migrating component, chloride). Although MTR are determined daily by analyzing the standard mixture, analyte migration times can be altered in a given sample by the presence of other analytes. Therefore, when analyte identity is in question, the sample is re-analyzed with an internal spike of known compounds.

Compounds are quantitated by the Millenium "Empower" software, using the response factors generated by the analysis of the daily standards. A new standard curve is prepared daily, and whenever a capillary is replaced.

For every sample, the raw data and the quantitated data are stored electronically. Information stored for every sample includes : date of analysis project name, sample designation, dilution, designation of the standards and their date of preparation.

11.7 Quality Control

- Working standards are made every 2 weeks by diluting purchased stock standard solutions.
- A Standard curve is prepared daily.
- A QC standard is analyzed with every batch of samples, and recorded in a QC standard log.
- A log book is kept into which each sample is entered, including project, sample designation, dilution and analyst, each standard preparation is detailed, newly purchased standards are noted, and electrolyte and capillary changes are noted.
- A DI water blank (and trip blanks and field blanks when provided) are analyzed.
- Duplicates are analyzed with every batch of 12 samples.

11.8 Method Performance.

Detection Limits: Practical Quantitation Limits using the chromate electrolyte (for analytes that are baseline-resolved) are 0.2 ppm. The PQL are increased if the sample is diluted. For a 10-fold dilution, the PQL is about 1.5 ppm.

Migration Times. The migration time of an analyte changes very slightly with the age of the capillary and of the electrolyte. Typical migration times range from 3.1 minutes for chloride to 5.1 minutes for butyrate.

Analyst Performance. An analyst must demonstrate the ability to obtain values for the QC standard within 5% of the running values. An analyst must also be able to analyze calibration standards, obtaining a correlation coefficient of at least 0.99 for each analyte.

11.9 Method References:

Waters Capillary Ion Analyzer, Operator's Manual.(1995). Waters Corporation, 34 Maple Street, Milford MA 01757.

Krol, J. M. Benvenute, and J. Romano. (1998) Ion Analysis Methods for IC and CIA and Practical Aspects of Capillary Ion Analysis Theory. Waters Corporation, 34 Maple St, Milford MA 01757

BCI SOP 12
Headspace GC/FID for Dissolved Gasses and Volatile Organic Compounds

12.1 Scope and Application.

The method is applicable to volatile organic compounds in water. Using a flame ionization detector (FID) with a gas chromatograph, both chlorinated and non-chlorinated compounds can be detected. The method is applicable to chlorinated and non-chlorinated compounds over a large range of molecular weights, as follows:

- Dissolved gasses (methane, ethene, acetylene, ethane, propene)
- PCE, TCE, cDCE, 1,1-DCE, VC
- 1,1,1-TCA, 1,1-DCA, 1,2-DCA, Chloroethane
- CT (carbon tet), CF (chloroform), DCM (dichloromethane)
- BTEX and chlorobenzenes.

The practical quantitation limit for samples analyzed by the 'Vial Method' at 21°C ranges from less than 1 μg/L for dissolved gasses, to 100 to 300 μg /L for compounds having low Henry's Law Constants. The headspace method can be applied to any water sample in a septum-capped container. At BCI, two procedures are routinely employed, – the Vial Method, and the Serum Bottle Method.

12.2 Summary of Headspace Method

Theoretical Basis. The method is based on the concept that in any closed container having a headspace and an aqueous portion, volatile compounds will distribute between the two matrices according to their Henry's Law Constants. A measurement of an analyte in a headspace sample can be used to determine the amount in the aqueous portion.

Approach. A standard is created in a closed container having both headspace and water by injecting a known mass of analyte through a septum, and allowing an equilibrium to develop. Analysis of a headspace sample from that container will produce an instrument response proportional to the mass of the compound injected. If an unknown sample is prepared in a similar container with the same headspace and liquid volumes, the mass of that compound can be determined by analyzing a headspace sample, and utilizing the response factor obtained by analyzing the standard.

Gas Chromatography with Direct Injection. A gas sample containing volatile compounds is injected into a capillary column in a gas chromatograph. The compounds travel down the column with the carrier gas, partitioning between the column and gas stream, and are separated roughly on the basis of molecular size with some sensitivity to polarity. Combustible compounds are detected by the FID, which transmits a signal to the computer software, which records a response proportional to the mass of the compound.

Application. At BCI, 43 mL vials are used for field samples of groundwater, 160 ml serum bottles are used for microcosm studies, and 1 L serum bottles are used for culture growth.

12.3 Interferences, Advantages, and Limitations.

Interferences.

In general, the headspace method is free from interferences from high molecular weight compounds because they do not volatilize from the water sample, and are not present in the headspace sample.

Large amounts of one compound can obscure small amounts of another which has a similar retention time.

The presence of sediment having high Koc may interfere with the analysis of higher molecular weight compounds due to their adsorption to the solid matrix.

Advantages.

The advantage of the method is that detection limits are lower for the lighter compounds than can be achieved by Mass Spectrometry, and mixtures having wide ranges of analyte concentrations can often be analyzed without dilution.

Limitations.

Analytes are identified by retention time only, therefore when two compounds have the same retention time (and similar peak width), the identity of a peak must be determined by mass spectrometry.

12.4 Apparatus and Materials

HP 5890 Gas Chromatographs
GC Column (proprietary)

Flame Ionization Detector.

PC computer with HP Chemstation software and Printer.

Standard Gas Mixture, 1% ppmv each methane, ethene, acetylene, ethane. Scott Gas.

Standard Analytes in methanol, 2 to 10 mg/ml. Restek.

12.5 Sample Collection, Preservation, and Handling.

Vials with septum caps, containing 250 µL 6N HCl, are shipped to the field. Ground water is added to overfull, and caps replaced. Samples are shipped to BCI, chilled, by overnight express, then refrigerated, and analyzed within 7 days.

12.6 Procedure for headspace Analysis.

GC Temperature Program . Proprietary program 35°C to 240°C

GC gasses. All gasses are high purity (99.999% pure)
Carrier gas, helium FID gasses, H_2 and air.

Detector. TCD for CO_2
FID for all other analytes.

Typical Retention Times 1 min to 25 min.

Vial Method.

In the laboratory, the vials, containing 43 ml of ground water, are brought to room temperature, then using two syringes, 5 ml of water are displaced through the septa while 5 cc of air or helium are injected. The vials are shaken for 20 minutes on a reciprocal shaker allowing the volatile compounds remaining in 38 ml of liquid to volatilize into the headspace portion. Standards are prepared in 43 mL vials with 5 cc headspace, in a similar manner.

Headspace samples of the standards, 100 μL, are withdrawn with a syringe and injected into the gas chromatograph. A response factor R (concentration in mg /L per instrument response in area counts) is generated for each compound. This response factor applies only to this container and the stated volumes of gas and aqueous phase, and the stated injection volume. Concentrations are recorded in terms of the entire mass of analyte in the aqueous phase. For Each Analyte:

A = Instrument response in terms of area counts
R = Response Factor in terms of mg/L/A

100 μL of headspace from a field sample is injected into the GC. The area counts generated for each analyte are multiplied by the Response factor

$$mg/L = (R)(A)$$

This application of the headspace concept is based on one developed in the EPA laboratory in Ada, OK (Kampbell et al. 1998).

Serum Bottle Method.

The Serum Bottle Method of headspace analysis is used for repeated analysis, over a time period of several months, of microcosms in which the biodegradation of selected analytes in ground water is being evaluated.

The bottles to be sampled have a volume of 160 ml, contain 100 ml of groundwater and 60 cc of headspace, and are sealed with a septum cap. (1.1 L serum bottles contain 1.0 L aqueous and

100 m headspace. The analytes in the serum bottle are distributed between the water and the headspace.

Standards are prepared in 160 ml serum bottles in a similar manner. Headspace samples of the standards are analyzed by injection into the GC to generate response factors specific to this ratio of headspace to aqueous phase.

Headspace samples are removed from the microcosms by syringe and injected into the GC, and quantitated using the response factors.

Results are expressed in μM, as though the entire mass of analyte were dissolved in the 100 ml aqueous portion.

12.7 Quality Control.

Decontamination. GC column is baked at 240°C for 20 minutes prior to every analysis. Syringe barrels and needles used for headspace sampling are baked at 120°C for 5 minutes prior to every analysis.

Syringes. Syringes used for concentrated standards or for neat compounds are kept in a separate location, and are not used for sampling headspace. These are rinsed repeatedly with methanol prior to baking.

GC Log Book. Every analysis is recorded in a log book, with project name, sample code, run number, date, and analyst. Maintenance events are also entered in the log book. Data are stored electronically, and a hard copy of the chromatogram and data are stored in a project binder.

Standards Log Binder. A log is kept on the preparation of standards, including the concentration, source, date purchased, and expiration date of every analyte. Standards received in ampules, when opened, are used only once. Chromatograms and data from analyses of standards are stored in the standards binder.

QC for Vial Method. Dissolved gas standards are prepared at three concentrations in the expected range of the samples for each batch of 10-15 samples. If the response factors are not within 10 % of the expected values, new standards are made and analyzed. Vial standards are used only on the day they are prepared. Sample values which exceed the linear range for any analyte are "diluted" by analyzing a smaller volume of headspace. A Trip Blank, Field Blank, and duplicate are analyzed for each batch of samples.

QC for Serum Bottle Method (Dissolved Gasses and Volatile Chlorinated Compounds). Killed control microcosms for every 'set' of bottles (containing water from the same well) are analyzed with each project batch, and checked with previous analyses for consistency.

12.8 Method Performance

If the analysis of standards results in retention times (RT) that differ by more than 0.3 minutes from the running RT, or response factors (RF) differ by more than 15% from the running RF, the standard will be re-analyzed and instrument flow parameters and temperature program will be checked.

12.9 Safety

Compressed gas tanks are chained and belted to tank brackets affixed to a wall in a garage/storage area on the other side of the laboratory wall. The gasses are plumbed into the lab via 1/8 ID copper tubing and connected directly to the GCs.

12.10 References

Kampbell, D. H. and S. A. Vandegrift. 1998. Analysis of dissolved methane, ethane, and ethylene in ground water by a standard gas chromatographic technique. J. Chromatographic Science, vol 36, page 253-256.

EPA Method 5021A

BCI SOP # 13
Molecular Hydrogen by Reduction Gas Analyzer

13.1 Scope and Application

The reduction gas analyzer is used to determine the concentration of molecular hydrogen in gas samples.

13.2 Summary of Method

Samples (4 cc) are introduced, with a N_2 carrier gas, into a column which acts as a mole sieve to separate gasses according to molecular size.

Reduction gasses (H_2 or CO) react with mercuric oxide to produce mercury vapor, which is detected by UV photometer.

$$H_2 + HgO\ (solid) \rightarrow H_2O + Hg\ (vapor)$$

13.3 Interferences

To avoid interference from impurities in the carrier gas, it is passed through a trap to remove traces of water vapor and petroleum contaminants, (UOP Corp N_2 Purifier, Model P 300).

Sample constituents may cause interference in the proper operation of the analyzer. H_2S damages the HgO bed. Chlorinated solvents interfere with analysis, requiring up to 15 minutes instrument recovery before subsequent analysis. High contamination requires baking the bed.

13.4 Apparatus and Materials

The instrument is manufactured by Trace Analytical, Menlo Park CA., and incorporates Gas sample injection port, Gas Chromatograph Column, Reduction Gas Detector, and Integrator with screen display. The column is a J&W Scientific GS-Molesieve Capillary Column, 0.53 mm I.D. x 30 m.

Samples are introduced using a variety of luer-loc Syringes, glass and plastic, gas-tight, with and without shut-off valves. Dilutions are made by injecting a sample, through a septum adapter (Hamilton #31335), into a 10 cc syringe containing dilution gas, and mixing with an internal agitator.

13.5 Standards and Dilution Gas

Ultra pure (99.999%) argon is used for sample dilution and flushing of syringes and needles. This gas must NOT be passed through a copper catalyst.

H_2 Standards are purchased as certified gases from Messer Industries, Allentown PA.

Calibration standards are prepared by diluting the Messer standard in ultra-high purity argon, in a 60 cc syringe, using the septum adapter described above. Standards are prepared within the working range of the system, up to 9 ppmv.

13.6 Handling Gas Samples from Microcosms.

100 μL samples are withdrawn from the headspace of a microcosm (according to subsampling protocol in BCI SOP # 10) and injected into the dilution syringe which contains 5 to 10 cc of ultra pure argon. The dilution is then injected into the RGA. Argon blanks are analyzed with each batch.

13.7 Handling Aqueous Groundwater Samples.

Serum bottles, 160 ml, provided pre-filled with H_2-free gas, are filled with ground water during field sampling and sealed with Teflon-coated gray rubber septa affixed with crimped caps. At BCI, using a two-needle procedure, 10 ml of water are removed while adding 10 cc of Argon, then the bottle is shaken for 20 min to allow H_2 to transfer to the headspace. A headspace sample of 100 μL was removed and diluted 100-fold in Argon prior to injecting into the reduction gas analyzer. Subsequently, for low concentration samples, 5cc is removed from the 160 ml serum bottle (while injecting 5cc degassed water) and injected directly into the H_2 Analyzer. A response factor for H_2 is obtained by analyzing a 5 ppm H_2 gas standard. The H_2 concentration originally in the ground water is calculated from the measured headspace concentration assuming a Henry's constant of 50.3. The lab method blank is subtracted from the sample results prior to entering in data table.

13.8 Handling Gas Samples from Ground Water Stripping:

Preparation of sample bottles. 26 ml serum bottles are prepared at BCI by flushing with ultra pure argon and are sealed with grey Teflon-lined butyl rubber stoppers, affixed with crimped aluminum caps, and shipped to the site.

Sample Collection. Samples are collected by stripping a continuous stream of ground water into a gas sampling bulb. A portion of the gas (10 cc) is then injected into sealed 26 ml serum bottles (causing the contents to become pressurized). In the field, the 26 ml bottles should be protected from sun. The samples are shipped priority overnight in coolers without ice.

Sub-Sampling 26 ml Serum Bottles in the Lab. For analysis, a 10 cc syringe is inserted and allowed to expand, thus measuring the amount of pressure in the bottle. Those with low pressure are reported as potentially incorrectly prepared in the field. Then 4 cc is injected into the RGA.

Preparation of Trip Blanks and Determination of Detection Limit. Trip blanks are prepared by injecting 10 cc of argon into 26 ml serum bottles prepared as described above, then shipped to the site and returned. Such trip blanks gain H_2 from ambient air (which is approximately 0.5 ppmv) by diffusion through the septa, at the rate of about 0.01 ppmv/day. Since the value of the trip blank must be subtracted from the sample value, the length of time between preparation and return of the serum bottles determines the detection limit.

Calculation of H_2 concentration in Groundwater from Gas Sample data

The raw data area count for each sample is corrected by subtracting the area count for the trip blank, or in the case of 100-fold lab dilution, subtracting the area counts for the dilution gas.

The result in terms of ppmv is calculated by multiplying the corrected area count by the Response Factor and field dilution and lab dilution factors.

To obtain the concentration in the original ground water, the ppmv value is converted to micromolar and divided by the Henry's Law constant for H_2.

13.8 Operation and Calibration of H2 Analyzer

Operating Parameters. The manufacturer's instructions are followed for carrier gas flow rate and sample injection rate, and for setting retention time windows. The column and Hg bed temperatures are held at 105°C and 265°C respectively.

Contamination of the Mercury Bed. Contamination from samples is removed by raising the bed temperature to 300°C for 8 hr.

Calibration. The instrument response (area counts) for calibration standards made by diluting stock standards is corrected by subtracting the area counts for the argon blank. Corrected areas are plotted against concentration. For the linear range, a response factor (RF) is defined as the unit concentration per unit instrument response.

Response Factor for Each Batch of Samples. The working Response Factor is determined by analyzing a mid-range standard in duplicate for each batch of samples. If the duplicates differ by more than 10 %, or differ from the running RF by more than 15 %, preparation and analysis of the calibration standard is repeated.

Blanks. Blanks (argon) are prepared at the same time as the sample containers to be sent to the field, and are pressurized with 10 cc ultra pure argon. These blanks are either maintained in the laboratory or sent as trip blanks. Blanks are analyzed in duplicate with each batch of samples.

Retention Time. Retention time of the calibration standard is noted daily. If not within the established retention time window, the manufacturer is contacted.

13.10 Quality Control

- The mid-range standard is analyzed in triplicate with each batch.
- Trip Blanks (for gas samples) are analyzed in duplicate with every sample batch.
- Field Samples are analyzed in duplicate when requested by client.
- All field samples and blanks (26 cc serum bottles of gas) are checked, prior to analysis, for the expected 10 cc of over-pressure (resulting from injection of 10 cc of sample in the field) by allowing the syringe barrel to rise and noting the excess volume of gas.
- Raw data is entered into a H_2 Analyzer Log Book, including project name, sample designation, pressure measurement, dilution, occurrence of bed contamination and 'bake-out' time, date, and analyst.
- Calculations are entered in the Project Notebook, and checked by another analyst.
- After an incident of bed contamination and bake-out, a standard is analyzed to provide assurance that the bed has been regenerated satisfactorily and that instrument response has recovered.

13.11 Method Performance

Each analyst that performs the method must demonstrate the ability to obtain a value for the average of triplicate determination of response factor that agrees with the running value within 10%.

Each analyst must be able to prepare and analyze standards and obtain linear standard curve within the established linear range of the instrument.

13.12 References.

RGA5 Process Gas Analyzer Operating Manual. Trace Analytical, 3517A Edison Way, Menlo Park, CA 94025.

Appendix A

US Army Corps of Engineers®

U.S. Army Corps of Engineers, Kansas City District

CONTRACT NO. W912DQ-06-D-0006, TASK ORDER 0025

CORNELL-DUBILIER ELECTRONICS SUPERFUND SITE SOUTH PLAINFIELD, NEW JERSEY

OPERABLE UNIT 3: GROUNDWATER

QAPP APPENDIX A:

FINAL FIELD SAMPLING PLAN

December 2008

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1. PROJECT DESCRIPTION

1.1. Introduction

This Field Sampling Plan (FSP) describes in detail the field procedures for the collection, analysis, and evaluation of Remedial Investigation/Feasibility Study (RI/FS) data for Operable Unit 3 (OU3) at the Cornell-Dubilier Electronics (CDE) Superfund Site (Site) located in South Plainfield, Middlesex County, New Jersey. The purpose of the FSP is to ensure that samples are properly collected, handled, and transported to the laboratory, and that sampling and handling procedures are fully documented.

Compliance with the FSP will result in the collection of representative samples, minimize the potential for cross-contamination, and ensure both proper preservation of the samples and laboratory analysis of appropriate constituents. The sampling and data collection methods described in this FSP are consistent with the U.S. Environmental Protection Agency (USEPA), U.S. Army Corps of Engineers (USACE), and the New Jersey Department of Environmental Protection (NJDEP) guidance documents including:

- A Compendium of Superfund Field Operations Methods (USEPA, 1987),
- Region II CERCLA Quality Assurance Manual (USEPA, 1989),
- Contract Laboratory Program Guidance for Field Samplers, EPA-540-R-04-003 (July 2007),
- Requirements for the Preparation of Sampling and Analysis Plans (USACE, 2001), and
- Field Sampling Procedures Manual (NJDEP, 2005).

Each member of the field team will be required to read this document and to fully understand the procedures that he or she are to follow in the field before

beginning work at the Site. In addition, each field team member will be required to read, understand, and sign the Site-specific Health and Safety Plan (SSHP) before initiating field activities.

Throughout the course of the field activities field conditions may differ from those expected. As a result, it may be necessary to change some of the procedures described in this FSP. The Malcolm Pirnie Project Quality Control Officer and/or Field Team Leader will advise the Project Manager (PM) of the need to modify procedures. If major changes to this FSP are warranted, then they will be documented on the Field Modification Form provided as Attachment 5 of the QAPP and provided to the USEPA and USACE for their approval. Copies of this FSP and Field Modification Forms (when approved) will be kept at the former Cornell-Dubilier Electronics facility at all times during the field work.

1.2. Project Background

The CDE Superfund Site has been divided into four Operable Units (OUs) by the USEPA. Operable Unit 1 (OU1) addresses residential, commercial, and municipal properties in the vicinity of the former CDE facility. On September 30, 2003, the USEPA signed a Record of Decision (ROD) to address OU1. Operable Unit 2 (OU2) addresses contaminated soils and buildings at the former CDE facility. On September 30, 2004, the USEPA signed a ROD to address OU2. Operable Unit 3 (OU3) addresses contaminated groundwater and Operable Unit 4 (OU4) addresses the Bound Brook.

The former CDE facility is located at 333 Hamilton Boulevard, South Plainfield, Middlesex County, New Jersey. The former CDE facility, recently known as the Hamilton Industrial Park, consists of approximately 26 acres. CDE manufactured electronic components including, in particular, capacitors in South Plainfield from 1936 to 1962. Polychlorinated biphenyls (PCBs) and chlorinated organic solvents were used in the manufacturing process. CDE disposed of PCB-contaminated materials and other hazardous substances directly on the facility soils. These activities led to widespread chemical contamination at the

facility. Elevated levels of volatile organic chemicals (VOCs) and PCBs have been found in soils at the facility, in soils at adjacent properties (residential, commercial, and municipal), in groundwater beneath the former CDE facility, and in the surface water and sediments of Bound Brook. A topographic map showing the location of the former CDE facility is included as Figure 1-1 and a Plan View of the facility, showing the former buildings, is included as Figure 1-2.

1.2.1. Summary of Groundwater Data

To focus the field activities of the FSP on contaminated groundwater, available historical data were evaluated to identify data gaps. FSP tasks were organized to complement the historical data and fill in data gaps. A summary of historical soil boring data collected between 1994 and 2006 and groundwater data collected in 2000 is presented in Section 3.1.7 of the Work Plan. A summary of the results of groundwater samples collected using packer testing procedures by the USEPA in January 2008 is presented below.

2008 Investigation - During January 2008, eight deep bedrock boreholes were drilled by USEPA to assess the hydraulic properties of the fractured bedrock and water quality of the bedrock groundwater up- and down-gradient of the former CDE facility. The boreholes were completed to an average depth of 150 feet below ground surface (bgs). Following completion of the boreholes, groundwater samples were collected from multiple depths using packer sampling techniques, targeting discrete water-bearing zones. Additionally, groundwater samples were collected from the 12 existing shallow bedrock monitoring wells located at the former CDE facility. Groundwater samples were analyzed for VOCs only. Results of the groundwater sampling indicate the presence of chlorinated VOCs in 11 of the 12 shallow bedrock wells located at the former CDE facility. Trichloroethylene (TCE) concentrations ranged from 4 ug/L in MW-02A to 186,000 ug/L in MW-11. Results of groundwater samples collected from the eight deep bedrock boreholes showed the presence of TCE in all but one (ERT-8). TCE concentrations ranged from 1.5 ug/L in ERT-1 to 2,250 ug/L in

ERT-2. Figure 1-3 summarizes the results of groundwater sampling conducted in January, 2008. Figure 1-3 also summarizes the approximate groundwater flow direction in the shallow bedrock monitoring wells, as interpreted by Foster Wheeler Environmental Corporation, 'Data Evaluation Report' (March, 2001).

A list of preliminary chemicals of potential concern (COPCs) in groundwater was identified by comparing the chemical concentrations of groundwater samples to the lowest of the Federal and New Jersey Maximum Contaminant Levels (MCLs), the New Jersey Groundwater Quality Criteria, and the Risk-Based Screening Levels. Based on this evaluation, a preliminary list of groundwater COPCs was identified. Table 1-1 lists the preliminary COPCs, their minimum and maximum concentrations, and the frequency of detections.

The results of the previous investigations indicate that contaminated soils may be contributing VOCs, SVOCs, pesticides, PCBs, dioxins/furans, and metals to the groundwater beneath the former CDE facility. Furthermore, the detection of VOCs in the deep bedrock packer samples indicates that contaminated groundwater may be migrating from the former CDE facility.

2. SCOPE AND OBJECTIVES

2.1. Remedial Investigation Objectives

This FSP presents the technical approach for conducting data collection to support the OU3 groundwater RI/FS. Studies of groundwater contamination in fractured porous sedimentary bedrock, like that found beneath the facility, have shown that, over time, much of the VOC mass originally present in fractures is eventually transferred to the rock matrix as a result of diffusion. Therefore, characterizing the mass in the rock matrix is important to predicting the fate and transport of contaminants. This approach, called Discrete-Fracture Network (DFN) (Parker, 2007), will be used to guide the RI data collection efforts. The additional data, to be collected during the RI phase, will be used in conjunction with historical groundwater data available for OU3.

The purpose of the additional field activities proposed for OU3 is to:

- Delineate the horizontal and vertical extent of groundwater contamination.
- Evaluate the lateral and vertical distribution and the amount of contaminant mass in the rock matrix by drilling rock core boreholes ('core borings'), collecting rock core samples and measuring the concentrations of VOCs and PCBs in the matrix pore water.
- Evaluate rock matrix moisture content, porosity, bulk density, solid-phase organic carbon (TOC), and rock oxidant demand by collecting rock core samples from the core borings.
- Measure the potential for presence of either light or dense non-aqueous phase liquid (LNAPL or DNAPL).
- Define the potential hydraulic connection between the water table aquifer in the Passaic Formation and Bound Brook.

- Identify water bearing zones and zones within the Passaic Formation that could be identified as confining units.
- Measure the Hydraulic Conductivity (K) of the Passaic Formation water bearing zones.
- Further evaluate exposure pathways and potential receptors to contaminants in groundwater underlying or migrating from the former CDE facility.

The field activities proposed to fill the data gaps discussed above includes the installation of monitoring wells and collection of groundwater samples, and the determination of hydrogeologic parameters. A detailed discussion of the proposed field activities is provided in Sections 3 and 4 of this FSP.

2.2. Quality Assurance/Quality Control

This FSP was developed to address data collection necessary to complete the RI/FS of OU3 groundwater. The collection of this data supports the Data Quality Objectives (DQOs) provided in Attachment 1.1 of the Quality Assurance Project Plan (QAPP).

The data generated during field activities and by the laboratory must be carefully collected and analyzed. The field methods and sampling procedures described in this FSP are based on established protocols published by the USACE and USEPA (USACE, 1994; USEPA, 1987b). The Quality Assurance/Quality Control (QA/QC) portions of the FSP and QAPP were prepared in accordance with USEPA Requirements for Quality Assurance Project Plans, USEPA QA\R-5, March 2001, USEPA Guidance for the Data Quality Objectives Process, QA/G-4, August 2000, and Uniform Federal Policy for Quality Assurance Project Plans, Final Version March 2005.

The QAPP describes the policy, organization, functional activities and QA/QC protocols necessary to achieve the DQOs dictated by the intended use of

the data. The QAPP provides detailed procedures for each QC-related field activity including:

- Site-specific standard operating procedures (SOPs) for sample management, sample preservation, and Technical System Field Audits.
- Chain of custody procedures.
- Sample packaging and shipment procedures.
- Decontamination procedures.
- QA/QC of field sampling and procedures for field changes and corrective action.
- Responsibilities of personnel.
- Parameters to be analyzed and analytical methods.

The QAPP presents information regarding DQOs, detection limits, preservation techniques, laboratory testing protocols, and laboratory accuracy and precision goals. It also provides details on the proposed analytical program including QA/QC samples, such as field duplicates and field equipment rinsate blanks. The QAPP also presents information on data validation.

2.3. Subcontractors

The following is a list of services to be subcontracted in order to complete the additional investigation activities:

- A drilling subcontract for bedrock drilling and coring.
- A subcontract for fabrication and installation of temporary borehole liners and multi-port monitoring wells (i.e., Flexible Liner Underground Technologies, Ltd. Co.)
- A geophysics subcontract for a non-invasive survey (*e.g.*, magnetic, resistivity, and gravity) to mark-out utilities at proposed drilling locations on private properties.

- Laboratory subcontracts for non-CLP analytical services (e.g., rock matrix VOC analyses).
- A waste disposal subcontract to remove all wastes (solid and liquid) generated during the investigation.
- A geophysics subcontract for down-hole high resolution temperature logging of the boreholes.
- A surveying subcontract for the survey of all newly installed monitoring wells and staff gauges.
- A subcontract for data validation services for validation of non-CLP data.

2.4. Site Access

It is anticipated that USEPA will obtain access to all properties that need to be entered for performance of the work. Accordingly, Malcolm Pirnie will coordinate with USEPA and provide all necessary information to facilitate the access agreement process. Should it be determined that Malcolm Pirnie is to obtain access, the approach outlined below will be implemented.

All affected property owners will be sent a request for access letter, with an attached property access permission form, by mail. Two attempts will be made to contact a property owner by telephone (if a phone number can be obtained) if no response to the mailing is received after 30 days. After verbal contact, a second access letter may be mailed out at the discretion of the USEPA or, if required by the property owner.

Visits to properties will be made on up to two occasions, if owners have not responded to the mailing and telephone access attempts. The property visits will be attempted in order to obtain a signed property access form and to record/answer any questions and concerns the owner may have.

No field investigation activity will be initiated until a signed copy of the access agreement form has been received by the USEPA. Upon receipt of a signed access agreement, affected parties will be contacted by telephone

approximately one week in advance of field activities to schedule the date of proposed field investigation. Once a date and time are agreed upon, a confirmation letter will be sent to the owner citing the relevant information. Property contacts will be limited to owners unless requested by the owner to contact the tenant to schedule appointments. The field crew will obtain a copy of the signed access form so that it can be presented upon visiting a property.

3. SAMPLE LOCATIONS, FREQUENCY AND DESIGNATION

3.1. Introduction

This section identifies each sampling event and includes the locations and number of samples to be collected and the parameters to be analyzed. Rock core samples will be collected from rock core extracted from cored bedrock borings ('core borings') in order to measure the concentration of VOCs and PCBs (Aroclors) contained within the rock matrix, and to determine physical properties of the rock matrix. A mobile laboratory will provide analytical results of the rock core VOCs within approximately 30 minutes of core sample collection. Monitoring wells will be constructed in all new bedrock boreholes, including existing boring ERT-7 (drilled and temporarily lined by USEPA in January 2008). Following completion of the wells, groundwater samples will be collected to further delineate the nature and extent of groundwater contamination.

Up to four bedrock 'core borings' will be continuously core drilled to a minimum of 250 feet bgs to obtain rock core samples and to install monitoring wells. The minimum depth of 250 feet assumes: bedding plane fractures dip at eight degrees to the northwest; fractures dipping at eight degrees to the northwest exist under the former CDE facility and extend northwest to MW-20 and MW-21; the distance from the former CDE facility to MW-20 and MW-21 is approximately 1,800 feet. Therefore, if the dominant pathway for contaminated groundwater is bedding plane fractures, then contaminated groundwater may be encountered as deep as 250 feet near MW-20 and MW-21.

Actual completion depths of the 'core borings' will be determined in the field based on the results of chlorinated VOCs in the rock core samples. The following criteria will be used to determine completion depths: concentrations of TCE in rock (ug/kg) will be charted; from the chart a trend, expected to be

asymptotic, toward a baseline or non-detect will be estimated; if over five consecutive samples below 250 feet, results are at or near baseline or non-detect, then the 'core boring' will be terminated.

These 'core borings' will be converted into monitoring wells by reaming to approximately 6-inches in diameter followed by installation of surface casings, riser pipes (assumed at 25 feet bgs), and Flexible Liner Underground Technologies, Ltd, L.C. (FLUTe™) 'Water FLUTe™' multi-port sampling systems (multi-ports). Figure 3-1 shows the locations of the proposed core borings and multi-port monitoring wells identified as "Proposed Rock Core & Multi-Port Water FLUTe Wells". Table 3-1 summarizes the rationale for all proposed monitoring wells.

An additional five borings will be drilled to an estimated minimum depth of 250 feet bgs using rotary tricone methods ('rotary borings') to allow for the installation of multi-port monitoring wells. These borings are not intended to be used for the collection of rock core samples and rock coring will not be conducted. The completion depths of the five 'rotary borings' will be based on the results of chlorinated VOCs in the 'core borings'. Since the core borings will be located within the centerline of the plume, Malcolm Pirnie has assumed that the maximum depths of detected VOCs in these core borings will represent the maximum depths anywhere else in the plume. The rotary borings will be located approximately along strike from the cored borings. Therefore the maximum depths of VOCs detected in rock core samples will be a reasonable guide for completion depths of the rotary borings. Rotary borings will be terminated at the completion depths of the nearest core boring located approximately along strike.

As will be conducted for the core borings, the rotary borings will be completed as monitoring wells, utilizing surface casings, riser pipes, and the 'Water FLUTe™' multi-port sampling systems. Figure 3-1 shows the locations of the proposed rotary borings/multi-port monitoring wells identified as "Proposed Multi-Port Water FLUTe Wells". Table 3-1 summarizes the rationale for all proposed monitoring wells.

Following the installation of the nine proposed bedrock wells equipped with multi-port sampling systems and the fitting of existing boring ERT-7 with a multi-port sampling system, Malcolm Pirnie will perform two full rounds of groundwater sampling, as well as a third limited round (for PCB Congeners/Dioxins/Furans only). Each full round of groundwater sampling will also include a synoptic round of water level measurements. Data from pressure transducers installed in some of the multi-port wells will also be downloaded during the synoptic sampling events.

3.2. Sample Locations and Frequency

3.2.1. Rock Core Sampling

The concentration of VOCs in the rock matrix will be measured by collecting rock core samples at two-foot intervals from three 'core borings' (MW-14, MW-16, and MW-20). A contingent core boring, MW-19, will only be core drilled and sampled for matrix diffusion parameters if VOCs in rock samples from MW-20 are not detected. The VOC results from MW-20 will be provided to the USEPA and USACE for review. The decision to core drill and sample for matrix diffusion parameters at MW-19 will be made in concert with the USEPA and USACE.

In addition, rock core samples will be collected for PCBs (Aroclors) every two feet from MW-14 and MW-16 (to be located at the former CDE facility). Samples for rock physical parameters will be collected at a frequency of every 20 feet from each core borings. The core borings will be drilled using an HQ-sized (2.5-inch diameter) triple-tube core barrel in five-foot runs to obtain the rock core samples. The core borings will be converted to multi-port monitoring wells as described in Section 3.2.5. Additional details on drilling procedures are provided in Section 4.4.2. Details of rock core sample collection procedures are provided in Section 4.6.

3.2.2. Sampling of Rock Matrix for VOCs and PCBs

This section describes the collection of rock core primary samples for VOCs and PCBs (Aroclors). 'Primary' samples are those collected specifically to determine concentrations of VOCs and PCBs (Aroclors). QA/QC samples, such as duplicates, are not considered 'primary' samples.

Volatile Organic Compounds (VOCs)

Rock core samples will be collected at approximately two foot intervals (detailed in Section 4.6) for analysis of VOCs by Stone Environmental, Inc. ('Stone Environmental'). The number of estimated samples is shown in Table 3-2. The samples will be prepared for analysis using a rock core crushing technique developed by the University of Waterloo and Stone Environmental. Rock core samples will be analyzed using Stone Environmental's mobile data acquisition laboratory to obtain turn-around times of approximately 30 minutes.

Samples will be ranked according to high or low priority. High priority samples are those whose results are needed to determine the final depths of the coreholes. High priority samples will be analyzed for chlorinated VOCs in the field by Stone Environmental. Lower priority samples (e.g., PCB Aroclors in rock matrix) will be crushed in the field by Stone Environmental and shipped to a fixed-based laboratory. The method of selection and preparation of rock core samples is discussed in Section 4.6. Details on the analytical method of rock core samples is provided in the QAPP.

The use of high priority samples to terminate a core boring is as follows:

- A minimum depth of 250 feet was assumed as described in Section 3.1;
- VOC sample results will be reviewed in the field as they are received from the mobile laboratory; and
- When TCE reaches baseline or non-detect in five consecutive samples (as estimated from a chart of results) then the boring will be terminated.

Polychlorinated Biphenyls (PCBs Aroclors)

Rock core samples to be analyzed for PCBs (Aroclors) will only be collected from the two core borings located at the former CDE facility (MW-14 and MW-16). Rock core samples for PCBs (Aroclors) will be collected from the same intervals and at the same frequency (one sample every two feet) as rock core samples for chlorinated VOCs. Stone Environmental will crush the rock sample, preserve a portion of it for analysis of VOCs by their mobile laboratory, and containerize the remaining crushed rock sample for shipment to an outside laboratory for analysis of PCBs (Aroclors). The number of estimated samples is shown in Table 3-2.

3.2.3. Sampling of Rock Matrix for Physical Parameters

Rock core samples will be collected every 20 feet for measurement of the following matrix physical parameters will be collected from all the proposed core borings, as detailed below and summarized in Table 3-2:

- Moisture content and porosity;
- Dry and wet bulk density; and
- Solid-phase organic carbon (TOC).

These parameters are necessary to estimate groundwater concentrations and determine phase partitioning of contaminant mass between rock matrix and groundwater (Hurley and Parker, 2002). The objectives of sampling rock matrix for physical parameters are:

- Acquire sufficient samples to determine variations of rock physical parameters of the Passaic Formation;
- Evaluate differences due to lithological variations, and;
- Acquire sufficient samples to determine average values of these parameters.

Rock core samples for moisture content, porosity, wet and dry density, and TOC analyses will be collected every 20 feet commencing at the base of the riser pipe (assumed at 25 feet bgs) from all of the core borings. In addition, Malcolm Pirnie will attempt to obtain samples selected to be representative of lithological variability.

Core borings MW-14 and MW-16, located at former CDE facility, are expected to intersect mostly Passaic Formation siltstones. Core borings MW-19 and MW-20, located in the presumed down-gradient direction, are expected to intersect mostly Passaic Formation mudstones to approximately 110 and 225 feet bgs respectively. These depths are estimated assuming an eight degree bedding dip to the northwest. Below these depths, Passaic Formation siltstones may be dominant as they underlie the mudstones.

Malcolm Pirnie anticipates that three additional samples per boring may be required to assess lithological variability. These additional samples should be biased toward mudstone, when present. This approach yields 12 primary samples and three additional lithological variability samples per boring. The number of estimated samples and rationale of collection depths are shown in Table 3-2.

3.2.4. Sampling of Rock Matrix for Natural Oxidant Demand

Separate rock core samples for natural oxidant demand will be collected below the riser pipe (assumed at 25 feet bgs) from proposed core borings MW-14 and MW-16 and will be selected to be representative of lithology and variability. Malcolm Pirnie anticipates that two samples per boring will be sufficient to assess rock oxidant demand in the Passaic Formation. The number of estimated samples and rationale for collection depths are shown in Table 3-2.

Rock oxidant demand testing will be conducted using a bench scale test as described in the QAPP. Malcolm Pirnie anticipates that the oxidants to be tested will include potassium permanganate (permanganate) and sodium persulfate (persulfate).

3.2.5. Detection of NAPL in Cored Boreholes

A temporary NAPL absorbent liner (NAPL FLUTe™ System) will be installed in four proposed deep bedrock wells MW-14, MW-15, MW-16, and MW-17. The liner will be used to evaluate whether NAPL is still present in the bedrock fractures through the use of a reactive covering on a blank liner. Installation of the liner is described in the FLUTe™ document entitled 'Installation Procedure for NAPL Blank Liner in Open Borehole below the Water Table' provided in Appendix A-A. The temporary liner will be installed in a 6-inch diameter reamed borehole, and left in place for approximately three hours. The liner will be retrieved and the reactive covering removed and examined for the presence of NAPL. The liner will be re-installed into the reamed borehole to conduct a FLUTe™ Liner Test as described in Section 3.2.8, and to seal the borehole prior to construction of a multi-port monitoring well. Flexible Liner Underground Technologies, Ltd. L.C. (FLUTe™) will supply, install, retrieve the NAPL absorbent liner, and assist in interpretation of the results. A FLUTe™ document entitled 'Guide for the use of FLUTe Hydraulic Conductivity Profiler Results' is provided in Appendix A-A.

3.2.6. Multi-Port Monitoring Well Construction

Three deep bedrock multi-port monitoring wells (MW-14, MW-15, and MW-16) will be installed at the former CDE facility to supplement the existing well network. This network currently includes 12 shallow bedrock wells (MW-1A, MW-2A, MW-3, MW-4, MW-5, MW-6, MW-7, MW-8, MW-9, MW-10, MW-11, MW-12) and one existing deep bedrock well (ERT-1). Table 3-3 shows construction details of the existing shallow and deep multi-port monitoring wells. As shown on Figure 3-1, proposed multi-port wells MW-14 and MW-15 will be located along Transect-A. MW-14 will be located near MW-11 which historically has contained the highest TCE results of the shallow bedrock wells (Table 3-1). MW-15 will be located at the southwest end of Transect-A. Proposed monitoring well MW-16 will be located along the presumed centerline of the plume, down-

gradient from MW-11. The proposed multi-port wells (MW-14, MW-15, and MW-16) will each contain 10 sampling ports as listed in Table 3-4; however, the actual number of ports will be based on hydraulic data and field observations during drilling.

Following the RI field program, the monitoring well network at the former CDE facility will include the following:

- Twelve existing single screen shallow bedrock wells;
- One existing multiport well (ERT-1) with eight sampling ports;
- Three proposed multi-port wells with 10 sampling ports each;
- A total of 50 existing and proposed sampling ports

The 12 existing shallow bedrock monitoring wells located at the former CDE facility were installed in 2000 to an average depth of 51 feet. These wells were installed with varying screen lengths ranging from 15 to 25 feet (Table 3-3). In order to confirm their integrity, an inspection of each shallow monitoring well will be conducted during the RI field program. A well assessment checklist, which includes an assessment of well conditions and recommendations for restoring the well, if necessary, will be completed for each well and will be provided to the USEPA and USACE. Upon approval, Malcolm Pirnie and its drilling subcontractor will perform the necessary well restoration. A copy of the well assessment checklist to be used is provided in Appendix A-B.

Six deep bedrock multi-port monitoring wells (MW-13, MW-17, MW-18, MW-19, MW-20, and MW-21) will be installed outside the boundaries of the former CDE facility to supplement the existing well network. This network currently includes six deep multi-port bedrock wells (ERT-2, ERT-3, ERT-4, ERT-5, ERT-6, and ERT-8); ERT-7 (drilled and temporarily lined by USEPA in January 2008) will be completed as a multi-port well during the RI field program conducted by Malcolm Pirnie. The proposed multi-port monitoring wells are intended to laterally and vertically delineate contamination outside the boundaries of the former CDE facility:

- Proposed multi-port wells MW-19 and MW-20 will be located north and down-gradient of the former CDE facility, along Transect-B, the presumed centerline of the plume;
- Proposed multi-port well MW-13 will be located east of the former CDE facility, along strike from ERT-2, ERT-5 and proposed wells MW-16, MW-17, and MW-18 on Transect-C;
- Proposed multi-port well MW-21 will be located northwest of existing well ERT-4, to characterize groundwater in the presumed lateral distal plume fringe or boundary zone;
- Proposed monitoring well MW-17 will be located near ERT-2. The purpose is to vertically delineate VOCs in this location. Further, together with proposed wells MW-13 and MW-16, MW-17 will provide a means to estimate mass discharge from the former CDE facility, through Transect-C; and
- Proposed multi-port well MW-18 will be located near ERT-5 at the western end of Transect-C to vertically delineate VOCs in this location.

Since proposed multi-port monitoring wells MW-17, MW-18, and MW-19 will be located near to existing wells ERT-2, ERT-5, and ERT-3 respectively (Table 3-4), the existing sampling ports will not be duplicated in the proposed wells. Therefore, the proposed multi-port wells will only be installed with sampling ports below the deepest port in nearby ERT wells. As shown in Table 3-4, the number of ports for the monitoring wells located outside the boundaries of the former CDE facility will be:

- Seven existing ERT multi-port wells (including ERT-7) with a total of 45 sampling ports;
- Proposed multi-port wells MW-13, MW-20, and MW-21 with 10 ports each for a total of 30 ports;

- Proposed multi-port wells MW-17, MW-18, and MW-19 with 5 sampling ports each for a total of 15 ports; and
- A total of 90 existing and proposed sampling ports

Note: Malcolm Pirnie has assumed that seven ports will be installed in ERT-7 based on down-hole geophysics performed by USGS.

Following completion of rock coring activities at proposed wells MW-14, MW-16, MW-19, and MW-20, the cored borehole will be reamed (opened out) to approximately 6-inches in diameter and completed with an impermeable temporary FLUTe™ liner to prevent vertical cross contamination of the borehole. As described in Section 3.2.8, FLUTe™ Liner Tests will be conducted during installation of the temporary blank liners and temperature profiling will follow installation of the liners.

Proposed multi-port monitoring wells MW-13, MW-15, MW-17, MW-18, and MW-21 will be constructed in rotary borings, drilled using rotary tricone methods. As outlined in Section 3.1, these wells are assumed to be completed to a minimum of 250 feet bgs. Also as noted in Section 3.1, rock core will not be collected from these borings. Each rotary borehole will be drilled to approximately 6-inches in diameter and completed with an impermeable temporary FLUTe™ liner to prevent vertical cross contamination of the borehole. As described in Section 3.2.8, FLUTe™ Liner Tests will be conducted during installation of the temporary liners and temperature profiling will follow installation of the liners.

The proposed wells together with the eight existing deep multi-port wells (including ERT-7) and the 12 shallow bedrock wells provide an anticipated 140 unique sampling points as shown in Table 3-4.

3.2.7. Groundwater Sampling

The groundwater sampling program includes two full synoptic events to collect groundwater analytical samples from each well and multi-port sampling port ('sampling port') within the monitoring networks. These events will be spaced approximately three months apart and will commence a minimum of two weeks following completion of the last deep bedrock monitoring well. A limited extent sampling event for PCBs (Congeners) and dioxins/furans will be conducted approximately three months after the second synoptic event. During the two synoptic events, groundwater samples will be analyzed for the following chemical parameters as listed in Table 3-4:

- TCL VOCs;
- TCL SVOCs;
- TCL Pesticides;
- PCBs (Aroclors);
- TAL Metals (unfiltered); and
- Cyanide

During the second synoptic event, PCBs (Congeners) and dioxins/furans will also be collected; however, only from those wells and sampling ports in which PCBs (Aroclors) were detected in the first synoptic event. A third limited extent sampling event will be conducted to collect samples for PCBs (Congeners) and dioxins/furans from only those wells and sampling ports in which PCBs (Aroclors) were detected in the first synoptic event.

In addition, baseline groundwater geochemical characterization will be conducted to support the evaluation of remedial options during the FS process. Baseline groundwater geochemical characterization parameters will be collected during the first and second synoptic events. Wells to be sampled are listed in Table 3-5. Chemical and hydraulic data collected by the USEPA in January 2008 was reviewed to aid in selecting sample points that represent up-gradient, down-gradient, and mid-plume conditions.

Baseline groundwater microbiological modified microcosm 'Presence/Absence' ('Functional Screening Tests') samples will be collected to assess the presence or absence of microbes or groups of microbes that may be involved with bioremediation or other biological processes. The likely active microbe in the anaerobic zone/s of the groundwater system is *Dehalococcoides spp* (DHC). Functional screening will utilize a modified microcosm test by Bioremediation Consulting, Inc. termed the 'Anaerobic Presence/Absence Test'. The purpose is to determine the presence or absence of DHC and possible activity levels. Functional screening samples will be collected during the second synoptic event.

While the anticipated wells to be sampled for functional screening are listed in Table 3-5, final selection of sample points will be based on analytical results obtained from the first groundwater sampling event. Available data from physical and hydraulic parameter tests will also be reviewed to aid in selecting sample points that represent up-gradient, down-gradient, and mid-plume conditions.

Existing Shallow Bedrock Wells

Groundwater samples from the 12 shallow bedrock wells installed in 2000 at the former CDE facility (MW-1A, MW-2A, MW-3, MW-4, MW-5, MW-6, MW-7, MW-8, MW-9, MW-10, MW-11, MW-12) will be collected according to the NJDEP low flow groundwater sampling protocol (NJDEP, 2003) as given in Appendix A-B. Physical parameters to be measured include:

- Depth of well from top of inner casing;
- Depth to water from top of inner casing;
- Depth to non-aqueous phase liquids (NAPLs) from top of inner casing;
- Volume purged;
- pH;
- Temperature (deg C.);

- Specific Conductance (SC), (micro Siemens/centimeter, uS/cm);
- DO (milligrams/liter, mg/l), during purging, before and after sample acquisition);
- ORP (millivolts, mV), during purging, before and after sample acquisition);
- Eh is calculated by adding a correction factor to ORP specific to the electrode used;
- Turbidity (nephalometric turbidity units, NTU); and
- Purge flow rate.

Analytical samples will not be collected if either light non-aqueous phase liquids (LNAPLs) and/or dense non-aqueous phase liquids (DNAPLs) are detected. Following measurement of physical parameters, analytical samples will be collected from the shallow bedrock wells. For the first and second synoptic events, each groundwater sample will be analyzed for TCL VOCs, TCL SVOCs, TCL pesticides, TAL metals and cyanide, and PCBs (Aroclors). As noted above, in addition to PCBs (Aroclors), a subset of groundwater samples will be collected and analyzed for PCBs (Congeners) and dioxins/furans during the second synoptic event. During the third limited extent groundwater sampling event, samples for PCBs (Congeners) and dioxins/furans will only be collected from those wells in which PCBs (Aroclors) were detected during the first round of sampling. The number of estimated groundwater samples is given in Table 3-4.

Deep Bedrock Multi-Port Wells

Groundwater samples from the eight existing (including ERT-7) and nine proposed deep bedrock multi-port wells will be collected according to 'Sampling Guidelines for Water FLUTe™ Systems', as given in Appendix A-A. The method allows flow rates at or less than 0.2 liter per minute. In order to achieve and maintain low rates of flow, FLUTe™ will supply a spreadsheet for each multi-port system (existing and proposed deep bedrock wells) designating the sampling

pressure and discard volume for each sampling port. Physical parameters will be collected following purging and prior to the collection of analytical samples. Physical parameters to be measured include:

- pH;
- Temperature (deg C.);
- Specific Conductance (SC), (micro Siemens/centimeter, uS/cm);
- DO (milligrams/liter, mg/l), during purging, before and after sample acquisition);
- ORP (millivolts, mV), during purging, before and after sample acquisition);
- Eh is calculated by adding a correction factor to ORP, specific to the electrode used; and
- Turbidity (nephalometric turbidity units, NTU).

Additional data specific to the FLUTe™ procedures will include:

- FLUTe™ Multi-Port 'Sampling Volume Spreadsheet' with recommended purge pressures specific for each port in each well;
- Depth to water inside the FLUTe™ liner via the 'TAG' tube, using a bead type electric water level meter or standard slim-line electric water level meter. The intent is to ensure that water inside the liner is approximately 10-feet above that of the highest water levels in the formation;
- Depth to water at each port (formation water) prior to purging as measured inside the pump tube for each port, using a bead type electric water level meter;
- Record formation pressure at each port using the pressure transducer, where installed;
- Perform at least three purge strokes for each port;
- *Document purge time and volume for each port; and*
- Record depth to water at each port at the end of sampling.

Following collection of physical parameters, analytical samples will be collected from the deep multi-port bedrock wells. For the first and second synoptic events, each groundwater sample will be analyzed for TCL VOCs, TCL SVOCs, TCL pesticides, TAL metals, cyanide, and PCBs (Aroclors). As noted above, in addition to PCBs (Aroclors), a subset of groundwater samples will be collected and analyzed for PCBs (Congeners) and dioxins/furans during the second synoptic event. During the third limited groundwater sampling event, samples for PCBs (Congeners) and dioxins/furans will only be collected from those wells in which PCBs (Aroclors) were detected during the first synoptic event. The number of estimated groundwater samples is given in Table 3-4.

In addition, baseline geochemical groundwater characterization will be conducted. The aim is to select wells that may be situated up-gradient and near the core of the plume. Table 3-5 lists the deep bedrock multi-port wells proposed for baseline characterization and the rationale for their selection. Locations for baseline samples may be modified following the results of rock matrix sampling and assessment of existing groundwater sampling results. Table 3-5 indicates the number of characterization samples.

The characterization parameters will be:

- Methane, ethane, ethene;
- TOC/DOC;
- Fe^{+2};
- Nitrate, sulfate, chloride;
- Alkalinity; and
- Hardness.

CLP/DESA or a private subcontracted laboratory will be utilized for these analyses as described in the QAPP.

To aid in assessment of microbiological activity baseline Functional Screening Tests samples will be collected. Table 3-5 lists the deep bedrock

multi-port wells proposed for this task; five samples per well are currently proposed. The aim is to acquire a vertical and horizontal distribution of samples. Samples will be collected from ERT-1, a presumed up-gradient background well, from MW-14 and MW-16 located in the presumed core of the plume, and from MW-20 located in the presumed distal plume core nearest to the Spring Lake water supply wells. The number of samples given in Table 3-5 is an estimate and will be finalized following review of all analytical data from the first synoptic event.

3.2.8. Groundwater Hydraulic Head Measurements

As with groundwater quality, monitoring for hydraulic heads is a critical component in determining how contaminants enter a system. Water levels will be measured in shallow bedrock wells by the use of manual water level meters. Hydraulic heads will be measured in multi-port monitoring wells by both manual water level meters, and pressure transducers installed within the ports. In addition to permanent pressure transducers installed in ERT-2, ERT-3, and ERT-4, permanent pressure transducers will also be installed in selected proposed multi-port wells. Hydraulic heads may change over time due in part to: changes in pumping from public water supply wells; an aquifer pumping test (Section 3.2.8); and changes in recharge. Further, hydraulic heads in monitored fractures or fracture zones may also exhibit temporal changes with respect to each other, such as changes in vertical gradients due to outside influences. Such temporal changes cannot be recognized by manually recording single water level measurements during synoptic events. Pressure transducers cannot be retroactively installed in Water FLUTe™ System multi-port wells; they must be installed during completion. Therefore, data downloaded from pressure transducers will be used, as needed, to assess background temporal changes in addition to the specific requirements of the aquifer pumping test.

Prior to initiation of the proposed cored boreholes and construction of proposed multi-port monitoring wells, water levels will be obtained from the

existing monitoring wells (MW-1A through MW-12, and ERT-1 through ERT-6 and ERT-8). The purpose is to establish whether upward or downward gradients are present, prior to final planning and installation of proposed monitoring wells. This information may be critical in evaluating the likelihood of cross-contamination during well design and installation.

Water levels will be measured in all monitoring wells and multi-port monitoring wells using manual methods during the two synoptic groundwater sampling events. Pressure transducer readings will also be recorded from those multi-port monitoring wells equipped with permanent pressure transducers.

Manual water level measurements will be recorded from monitoring wells by using electric water level meters. In the case of the multi-port monitoring wells, the formation water level for each port is recorded via the pump tube for each port as described in 'A guide for the use of pressure transducers with the Water FLUTe™ System' located in Appendix A-A. Purging of the ports is required prior to recording water levels. FLUTe, Ltd recommends using a slim-line or bead type electric water level meter as the pump tube is equipped with a stainless steel quick-connect (for the nitrogen gas driver line), and may also be somewhat curved.

Pressure transducers have been permanently installed at all ports in existing multi-port monitoring wells ERT-2, ERT-3, and ERT-4 (Table 3-6). Pressure transducers will be installed in existing single screen monitoring wells (MW-1A, MW-2A, MW-3, MW-4, MW-5, MW-6, MW-7, MW-8, MW-9, MW-10, MW-11, MW-12) for the purpose of the aquifer test (Section 3.2.8).

Each pressure transducer installed in multi-port deep bedrock monitoring wells is a permanent device, installed during the construction by FLUTe™ of a Water FLUTe™ System multi-port, and connected directly to the formation. FLUTe™ currently uses In-Situ, Inc. 'Level TROLL' pressure transducers and associated communication cables. The expected battery life of the In-Situ, Inc. pressure transducers is approximately five years. Pressure transducers will be installed:

- In all ports in proposed bedrock monitoring wells MW-13, MW-14, and MW-15.
- In MW-19 only below 139 feet bgs since existing well ERT-3 is equipped with seven pressure transducers in various ports above that depth.
- In all ports in proposed well MW-20, since this well will be located near the Spring Lake water supply wells.

A guide for the use of pressure transducers with the Water FLUTe™ System is provided in Appendix A-A.

The number and location of pressure transducers in each of the proposed multi-port wells will be finalized following review of available data, including FLUTe™ Liner Tests and borehole temperature profiling.

3.2.9. Physical and Hydraulic Properties of the Aquifer

Understanding the physical and hydraulic properties of the Passaic Formation will aid in the analysis of groundwater velocities and flow directions beneath the former CDE facility and surrounding area. The following tasks will be conducted:

- Water table aquifer/Bound Brook interaction;
- FLUTe™ Liner Tests;
- Borehole temperature profiling, and;
- Aquifer pumping test.

Water Table Aquifer/Bound Brook Interaction

To define the potential hydraulic connection between the Passaic Formation water bearing zones and Bound Brook, three staff gauges will be installed in Bound Brook. Water level measurements in the Bound Brook will be recorded manually during the two synoptic rounds of groundwater measurements in wells. Water levels will be measured manually in each shallow bedrock well,

from each port of the multi-port wells during the two synoptic groundwater sampling events. Assuming an equivalent porous media, comparison of hydraulic head from Bound Brook to the hydraulic head in the Passaic Formation water bearing units will evaluate if Bound Brook is hydraulically connected to the bedrock fractures.

FLUTe™ Liner Tests

The geophysical information collected in January 2008 will be used in conjunction with the FLUTe™ Liner Tests ('liner drop tests') in proposed deep bedrock monitoring wells to identify water bearing zones and zones that could be considered as confining units in the Passaic Formation. FLUTe™ will supply and install the temporary blank liners and assist in interpretation of the liner drop tests.

The Hydraulic Conductivity (K) of the Passaic Formation water bearing zones will be defined using liner drop tests to confirm the water bearing zones and estimate the hydraulic conductivity of each zone. Combined with matrix diffusion testing data and borehole temperature profiling, data from liner tests will be used to locate sampling and hydraulic head ports for the multi-port wells.

Liner drop tests will be conducted in all proposed boreholes that will be completed as multi-port Water FLUTe™ System monitoring wells. In addition, a liner drop test will also be conducted in existing borehole ERT-7 which is currently equipped with a blank liner. In ERT-7, the results of the liner drop test, borehole temperature profiling, and geophysical data collected in January 2008 will be used to refine the proposed number and placement of sampling and hydraulic head ports for the Water FLUTe™ System multi-port wells.

The liner drop tests will be conducted in the six-inch diameter reamed core borings and rotary boreholes, and the liners temporarily left in place until installation of the multi-port Water FLUTe™ systems. The technique is described in the FLUTe™ document entitled 'Guide for the use of FLUTe Hydraulic Conductivity Profiler Results' provided in as Appendix A-A.

Borehole Temperature Profiling

High-resolution temperature logging will be performed inside the temporary FLUTe™ liners. The technique is described in Pehme, Greenhouse, and Parker (2007). The data, in combination with liner drop tests and analytical results from rock core samples, will be used to determine depths and relative significance of water-bearing fractures. These data will be used to locate sampling and hydraulic head ports for the Water FLUTe™ System multi-port wells.

In addition, high-resolution temperature logging will be performed in the temporary FLUTe™ liner installed in existing borehole ERT-7. In ERT-7, the results of the liner drop test, borehole temperature profiling, and geophysical data collected in January 2008 will be used to refine the proposed number and placement of sampling and hydraulic head ports for the Water FLUTe™ System multi-port wells.

Aquifer Pumping Test

Note: The need for an aquifer pumping test is still being determined by USACE, USEPA, and Malcolm Pirnie.

An aquifer pumping test will utilize a pumping well installed at the former CDE facility. The specific design, including total depth and diameter will be specified following review of all available data, including; water level measurements from shallow and deep multi-port monitoring wells, liner drop tests, borehole temperature profiling, analytical results from rock core samples, and results from the two synoptic groundwater sampling events. A detailed plan for the integrated pumping test will be submitted following the data review. This FSP therefore does not contain any specific procedures for performance of the test.

For the purposes of this FSP, Malcolm Pirnie assumes that one zone within the pumping well will be isolated as necessary by the use of inflatable

packers. Isolation of the pumping zone is intended to limit in-hole cross contamination and more closely target pumping to a specific zone.

The test will consist of a background monitoring period, a step-rate pumping period, a constant rate pumping period of a minimum of 72 hours, and a recovery period. Pressure transducers and manual water level meters will be used to record aquifer hydraulic head data from each shallow bedrock well, and each port in the multi-port wells, where equipped. Surface water levels will also be manually acquired from the three staff gauges installed in Bound Brook. Barometric pressure and precipitation data will also be collected during the entire test.

Further details will be presented in an Addendum to this FSP following completion of other phases of the work.

3.3. Quality Control Samples

Quality control procedures will be employed to check that sampling, transportation and laboratory activities do not bias sample chemical quality. Trip blanks, field blanks, duplicate samples, matrix spike samples and matrix spike duplicate samples will provide a quantitative basis for validating the analytical data. A summary of the anticipated QA/QC samples for groundwater is provided in the QAPP.

3.4. Sample Designation

A sample numbering system will be used to identify each sample. This system will provide a tracking procedure to allow retrieval of information about a particular sample and will assure that each sample is uniquely numbered. The sample identification will consist of at least three components as described below:

- Project Identification: The first component consists of a three letter designation which identifies the Site name. For this project, the three letter designation will be CDE.

- Sample Type: The second component, which identifies the sample type or event, will consist of a two letter code as follows:
    1. MD - Matrix Diffusion (rock core samples);
    2. LF - Low Flow Groundwater Sample; and
    3. MP - FLUTe™ Liner Multi-Port Groundwater Sample.
- Sample Location: The third component either identifies the sample location or identifies the sample as a trip blank or field blank. The monitoring well number will be used to identify groundwater sample locations. A two digit number will be used to identify each concrete core/matrix diffusion sampling location. TB will be used to identify a trip blank and FB will be used to identify a field blank.
- Sample Identification: The fourth component will be used for monitoring well samples, rock core samples, trip blanks, and field blanks as described below:
    1. Rock Core Samples: A two-digit number will be used to indicate the interval (feet below ground) from which the sample was collected;
    2. Low Flow Well Samples;
    3. Multi-Port Well Samples: A two-digit number will be used to indicate which port the sample was collected from; and
    4. Trip Blanks and Field Blanks: A two-digit number will be used to indicate the sequence of the sample.
- Quality Assurance/Quality Control Samples will be labeled with the following suffixes:
    1. MS - Matrix Spike;
    2. MSD - Matrix Spike Duplicate.
- Duplicate samples will be numbered uniquely as if they were samples. A record of identification for duplicate samples will be maintained.
- Examples of identification numbers are given below:

1. CDE-MD-13-6-9: Matrix diffusion sample collected from core hole MW-13 at a depth 6-9 feet below grade; and
2. CDE-LF-FB-02: The second field blank collected during the low-flow groundwater sampling event.

4. FIELD EQUIPMENT AND PROCEDURES

4.1. Introduction

The following sections provide instructions for each type of field activity to be conducted during the groundwater RI. These procedures will be followed in order to achieve the necessary DQOs. SOPs that are applicable to each field activity are referenced in the following sections and included as Appendices and/or Attachments to the FSP and QAPP.

4.2. Project Planning and Support Activities

A survey will be conducted using a photoionization detector (PID) in order to determine if a hazard due to chemical materials exists. Prior to any field work, a field team member will walk over all areas where field activities will occur. At each location where there is a reading above background, the operator will place a numbered stake in the ground and record the stake number and reading in the field book. If the initial survey determines there may be a hazard, the USACE will be notified immediately.

4.3. Mobilization and General Procedures

Mobilization will include field personnel orientation, equipment mobilization, marking/staking sampling locations, utility mark-outs (New Jersey One Call System), and checking the mark-out ticket against the utility markings (flags) at each drilling location. Equipment mobilization will include renting, purchasing, and transporting the equipment to the field. Each field team member will attend an orientation meeting to become familiar with the history of the Site, health and safety requirements, and field procedures.

The locations of proposed groundwater monitoring wells and matrix diffusion borings on public and private property will be marked in white paint one

week before the start of work. New Jersey's One Call System will be called and asked to mark the location of all of the utilities near each proposed drilling location. A copy of the Mark-Out ticket will be obtained and kept by the field team leader during drilling activities. Three full working days after the call, each proposed drilling location will be visited to make sure that each utility on the Mark-out ticket has identified their utilities in the area. Any utilities identified on the Mark-Out ticket but not identified at each location will be contacted to confirm they do not have any utilities in the area. However, New Jersey's One Call System will not identify utilities on private property. A geophysics subcontractor will perform a non-invasive survey (including pipe locators, magnetic, and resistivity methods) to mark-out utilities at the proposed drilling locations on private properties.

Field activities will be described in as much detail as possible so that one can reconstruct a particular situation without reliance on memory. At the beginning of each day, the date, start time, weather, and names of all sampling team members present will be entered. The names of visitors and the purpose of their visit will also be recorded. All field measurements as well as the instrument(s) used (including serial number) will be noted. The equipment used to collect samples will be noted, along with the time of sampling, sample description, depth at which the sample was collected, volume collected, and number of containers. Observations such as sampling conditions or any problems will also be recorded.

4.4. Groundwater Well Installation

4.4.1. Installation

A geologist will be in the immediate vicinity of drilling operations when drilling is being performed. Each new multi-port monitoring well will be drilled and installed according to ASTM D 2113-06, ASTM D5784-95, ASTM D 5783-95, ASTM D 5876-95, and ASTM D 2113-06 as appropriate. Monitoring wells MW-14, MW-16, MW-19, and MW-20 will first be core drilled using triple-tube wire-line

coring methods to retrieve HQ-sized cores (2.5-inch/6.35-cm diameter). The cored borings will then be reamed to 6-inch diameter boreholes using rotary tricone roller bit methods. This will allow for the installation of FLUTe™ liners and Water FLUTe™ System multi-port devices.

Monitoring wells MW-13, MW-15, MW-17, MW-18, and MW-21 will then be drilled using rotary tricone roller bit methods to 6-inch diameter to allow for the installation of FLUTe™ liners and Water FLUTe™ System multi-port devices.

Drilling fluid additives to increase the weight or viscosity of water will not be permitted. Drilling water will be obtained from a fire hydrant near the former CDE facility. One sample will be obtained from the water supply and analyzed for:

- TCL VOCs;
- TCL SVOCs;
- TCL PCBs (Aroclors);
- TCL Pesticides;
- TAL Metals;
- Cyanide; and
- Trip blank for VOCs.

Responsibility for core sampling, logging, and analytical procedures are as follows:

- Malcolm Pirnie and Stone Environmental are jointly responsible for selecting VOCs, PCBs, and physical parameters (moisture, density, porosity), and TOC sample locations;
- Stone Environmental is responsible for collecting, management, crushing, and analyzing rock core VOCs samples. Stone Environmental will also ensure that all QA/QC procedures, including splits for duplicate VOC samples are followed.;
- Stone Environmental is responsible for containerizing and preserving a portion of the crushed rock sample for analysis of

PCBs (Aroclors). Stone Environmental will also ensure that all QA/QC procedures, including splits for duplicate PCBs (Aroclors) samples are followed.;

- Stone Environmental is responsible for collecting, wrapping, sealing, and management of samples for physical parameters. Stone Environmental will also ensure that all QA/QC procedures, including split samples for duplicate physical parameters are followed.;
- Stone Environmental will split the physical parameters samples to provide a sub-section for TOC analysis.
- Stone Environmental will crush samples for TOC analysis, and deliver the crushed samples to Malcolm Pirnie personnel;
- Malcolm Pirnie personnel are responsible for all TOC sample handling, selection of duplicate TOC samples, packaging, and shipment to an outside laboratory;
- Stone Environmental is responsible for shipment of samples for physical parameters to an outside laboratory;
- Malcolm Pirnie personnel are responsible for sample management of crushed PCBs (Aroclors) samples (provided by Stone Environmental) and shipment to an outside laboratory; and
- Malcolm Pirnie personnel are responsible for collection, management, and shipment of rock oxidant demand samples.

Core logging procedures for MW-14, MW-16, MW-19, and MW-20, based on Sara (2003) are modified as follows since removal of core samples as soon as possible prevents full logging of the entire retrieved HQ core. A template core log is presented in Appendix A-B. Rock core sample collection and logging should be conduction in conjunction with each other:

- Log depth to bedrock, soil and rock types, weathering, fracturing, and changes in lithology on the log sheet;

- Note zones of water loss/gain and approximate water quantities lost or gained during each core run;
- When noting breaks on the rock core logs, differentiate between natural and mechanical breaks;
- Prior to retrieval of core, label directly on the core box; boring number, depths, run numbers, and date;
- Place labeled core run blocks and core loss blocks as appropriate;
- Photograph the core;
- Record sample names and depth intervals on the rock core log; the priority is rock type and distance to nearest fracture/s;
- Stone Environmental personnel will then remove samples from the core for VOC and PCBs (Aroclors) analyses. Sample lengths will be approximately two inches (5 cm) in length, sufficient to provide approximately 100g of crushed rock for both VOC and PCBs (Aroclors) analyses. Remove and submit for processing as soon as possible following retrieval of the HQ core. Further details on the selection and handling of samples are presented in Section 4.6;
- Insert labeled core blocks to replace samples;
- Stone Environmental personnel will then remove samples from the core for moisture, density, and porosity (physical parameters) analyses. Sample lengths will be approximately three inches (8 cm);
- Stone Environmental personnel will remove a sub-section from each physical parameters sample for analysis of TOC. This ensures that the physical parameters and TOC are obtained from the same section of rock core. This will be accomplished by splitting the sample length-wise as noted in Section 4.6.
- Wrap, seal, bag, and label the physical parameters samples for shipment as soon as possible following retrieval of the HQ core. Further details on the selection and handling of physical parameters samples are presented in Section 4.6;

- Insert labeled core blocks to replace physical parameters and TOC samples.
- Label the core box and photograph the core;
- Log the core for rock type, rock quality, RQD, presence, fracture frequency and dip, removed samples;
- Rock quality designation (RQD) will follow ASTM D 6032-02 and Sara (2003). However, this indication may be compromised since core sub-samples will immediately be removed for VOC analyses, requiring the geologist to 'factor' the removed lengths in to calculations of RQD;
- Remove additional approximately one-foot long samples for ROD tests;
- Insert labeled core blocks to replace natural oxidant demand samples; and
- Log the natural oxidant demand samples.

Monitoring well drilling locations will first be located and labeled with their respective identification number. However, conditions encountered in the field may necessitate changes in the proposed drilling methods. The drill rig platform, drill rods, drill bit, and any other equipment that will enter the boreholes will be decontaminated by steam cleaning before drilling the first borehole and between holes. Decontamination water will be collected in a polyethylene-lined decontamination pad and transferred to a large capacity storage tank ('Frac Tank') located at the former CDE facility. Sampling equipment will be decontaminated as included in SOP-9 Procedures for Decontamination as presented in Appendix A-B. More specific decontamination procedures for rock processing tools are given in Stone Environmental's standard operating procedures 'SEI-6.37.0', presented in Appendix A-C.

The following procedure will be followed for the installation of proposed monitoring wells:

- Use a hollow stem auger to drill through any unconsolidated soil and fill into the top of weathered bedrock. The diameter of the drilled hole will be sufficient to allow installation and grouting of 10-inch diameter steel surface casing. ASTM D 5784-95 covers hollow stem auger drilling methods;
- Install a nominal 10-inch diameter steel surface casing set into weathered bedrock and grouted into place. The grout will be allowed to set and cure for approximately 24-hours prior to further work on the borehole;
- Use air hammer methods to drill an approximately 10-inch diameter borehole into competent bedrock to an assumed depth of 25 feet bgs. ASTM D 5872-95 (2006) provides a guide for the use of air hammer drilling with water based drilling fluids;
- Only potable water shall be used to assist any drilling operations;
- Use a mud tub, cyclone, drill rod and collar wipers and other applicable measures to control and collect dust, over-spray, and cuttings;
- Install a nominal 6-inch diameter steel casing to the base of the drilled borehole and grout into place. The grout will be allowed to set and cure for approximately 24-hours prior to further work on the borehole;
- Core drill (MW-14, MW-16, MW-19, and MW-20) to retrieve HQ-size core using five feet long runs. Use swivel-type triple barrel wire-line methods as referenced in ASTM D 2113-06. Ream the core boring to approximately 6-inch diameter using rotary tricone roller bit methods, as referenced in ASTM D5783-95; and
- Use rotary tricone roller bit methods (MW-13, MW-15, MW-17, MW-18, and MW-21) to directly drill a 6-inch diameter borehole, as referenced in ASTM D5783-95.

- Develop each boring to remove introduced drilling water and excess drill cuttings.

Development will include:

- No water shall be added to the wells to assist development;
- Acceptable development methods include over-pumping and mechanical surging; airlifting and air-surging is not permitted;
- Record the initial pH, temperature, turbidity, color, and specific conductivity/conductivity (physical parameters) of the discharged water at commencement of development and at 10 minute intervals thereafter;
- Development will commence near the top of the water column and proceed systematically to near the bottom of borehole;
- All down-hole development tools such as pumps, bailers, and swabs will be used over the entire length of the borehole to ensure that all fracture zones are developed and that water introduced during drilling is removed;
- Continue to over-pump until stabilization of physical parameters is reached;
- Development stabilization will be achieved when three consecutive readings are; +/- 0.1 (pH); +/- 3% (specific conductance); < 50 NTU (turbidity); and
- All development water will be contained and handled as IDW.

The depth of each cored hole will be determined by the VOC results provided by Stone Environmental's mobile laboratory following the criteria outlined in Sections 3.1 and 3.2.2. Rock cores will not be retrieved from those borings directly drilled using rotary tricone roller bit methods. Instead, the total depths of MW-13, MW-15, MW-17, MW-18, and MW-21 will be estimated from the finished depths of nearby cored holes as described in Section 3.1.

Immediately upon the completion of development of proposed monitoring wells MW-18 through MW-21, FLUTe™ will install a temporary FLUTe™ liner in the 6-inch diameter reamed borehole to seal the borehole and prevent vertical cross-contamination. Installation is conducted with an inverted liner on a spool that is turned right side out while the liner is filled with water and descends into the well. FLUTe™ will record the descent of each blank liner; this is the 'liner drop test' which will aid in defining groundwater flow zones along the length of a borehole. The technique is described in the FLUTe™ document entitled 'Guide for the use of FLUTe Hydraulic Conductivity Profiler Results' provided in Appendix A-A. The time taken to install the liner depends upon the hydraulic conductivity of the surrounding fractures and the depth of the well. The higher the hydraulic conductivity, the more quickly the liner will drop into the open borehole.

Immediately upon the completion of development of proposed monitoring wells MW-14, MW-15, MW-16, and MW-17, a temporary NAPL FLUTe™ System (blank liner with a NAPL reactive cover) will be installed in the approximately 6-inch diameter borehole. Installation and retrieval is identical to blank liners without a reactive covering. The blank liner is equipped with a nylon covering, impregnated with a hydrophobic dye which changes color in contact with NAPL. If stains are not produced, this does not necessarily mean that NAPL is not present; it simply means that dye may not have contacted NAPL, if present. The dye is a non-toxic replacement for Sudan IV. The blank liner with reactive cover will remain installed in the wells for approximately three hours. The liner and cover will then be removed from the borehole.

Following removal of the reactive cover, the blank liner (now without a reactive covering) will be immediately installed. A liner drop test will be conducted during the installation of the blank liner. The reactive covering will be inspected for stains and the results photographed and recorded while the blank liner is being installed. Appendix A-A contains the guide 'Installation procedure for NAPL blank liner in open borehole below the water table'. FLUTe™ will

supply, install, retrieve all blank liners and NAPL reactive cover, and assist in interpretation of the liner drop tests and NAPL stain results.

Once the liner is in place, high-resolution temperature logging inside the FLUTe™ liner to further define groundwater flow zones will be conducted. The technique is described in Pehme, Greenhouse, and Parker (2007).

Well vaults will be completed with the minimum dimensions to accommodate WaterFLUTe™ systems as indicated in attached Figures 4-1 and 4-2. Well vaults will be traffic rated and water tight. The covers will be equipped with bolts to the vault frame to discourage theft and equipped with lifting lugs to enable removal for future groundwater sampling.

Alternatively, according to field conditions, the 10-inch diameter steel surface casing and the 6-inch diameter riser pipe will be extended above grade to form a 'stickup'. Enclose the surface casing with an approximately 18-inch diameter steel casing seated a minimum of two-feet into the ground. Fill the annulus between the two casings with concrete, to at least 6-inches above ground surface. Top up the grout between the 10-inch diameter steel surface casing and the 6-inch diameter riser pipe to at least 6-inches above ground surface. Complete the 18-inch diameter steel casing with a lockable steel cap to provide a permanent protective cover for the WaterFLUTe™ system, as indicated in Figure 4-2. A minimum of two vehicle barrier bollards will be installed in concrete adjacent to each stickup completion.

Data Use

Data collected from rock core sample results, FLUTe™ liner drop tests, NAPL FLUTe™ System, and high-resolution temperature logging will be used to select the Water FLUTe™ System multi-port sampling intervals. The advantage of a multi-port FLUTe™ system is that it protects groundwater integrity while isolating sections of a well. More specifically, a multi-port Water FLUTe™ System allows for direct sampling of fractures or groundwater flow zones within the well by isolating specific depths with a liner and tubing brought to the surface.

Installation for the Multi-Port Water FLUTe™ System is an identical process for the installation of the blank liner. Appendix A-A contains a guide for the use of the Water FLUTe™ System.

4.5. Groundwater Sampling

Two full groundwater sampling events will be spaced approximately three months apart to capture seasonal variation in groundwater quality and water level elevations. All monitoring wells will be sampled. In addition, a subset of groundwater samples will be collected and analyzed for PCBs (Congeners) and dioxins/furans during the second synoptic event. During a third limited extent groundwater sampling event, groundwater samples for PCBs (Congeners) and dioxins/furans will only be collected from those wells in which PCBs (Aroclors) were detected during the first synoptic event. The third limited extent groundwater sampling event will be conducted approximately three months after the second synoptic event.

The twelve shallow monitoring wells will be sampled using NJDEP Low Flow techniques (Appendix A-B). Common to both single screen shallow monitoring wells, and multi-port wells are the use of photoionization detectors (PIDs) and instruments to measure inorganic physical parameters. The use of PIDs is covered under the standard operating procedure SOP-8 PID Operation/Calibration in Appendix A-B. The use of instruments to measure pH, temperature, turbidity, specific conductance and dissolved oxygen ('listed parameters') prior to collecting groundwater samples is presented in Malcolm Pirnie's July 2008 Quality Assurance Manual (QAM) in Appendix A-B. The QAM contains standard operating procedures for instruments used to measure groundwater inorganic parameter (nutrients and demands), and 'analyze-immediately' listed parameters. The QAM presents procedures for collection of listed parameters, and decontamination of groundwater sampling equipment used for this purpose. Standard operating procedures for use of other instruments, such as a PID and water level meters are also attached to the QAM.

4.5.1. FLUTe™ Liner Multi-Port Well Sampling Procedures

A total of 140 primary groundwater samples per synoptic event will be collected from the nine newly installed multi-port wells, eight ERT multi-port wells, and 12 existing shallow bedrock wells as shown in Table 3-4. This total assumes that 10 sampling ports will be installed in six of the nine new multi-port wells and five ports will be installed in the remaining three wells. Detailed sampling procedures using the Water FLUTe™ System are provided in Appendix A-A. Decontamination procedures are summarized in Section 4.8.

Upon arrival at each well, the well identification number will be entered into a field logbook, noting any damage to the well. New plastic sheeting will be placed over and around the monitoring well so that a 5 x 5 foot clean surface is created for the sampling equipment. All materials, tools and equipment will be decontaminated prior to the placement on the plastic sheeting. A summary checklist for sampling from the multi-port Water FLUTe™ System is included as follows.

Water FLUTe™ System Sampling Checklist.

1. Check and restore the water level in the liner, by measuring the depth of water in the liner via the 0.5-inch diameter 'TAG' line; if necessary restore water in the liner to approximately 10 feet above the highest formation water level if necessary.
2. Measure the water level in the "pump tube" for depth to water table, using a bead type electric water level meter.
3. Connect the gas driver source to the gas drive tube for the port.
4. Set the regulator to the recommended purge pressure.
5. Expel the tube water at purge pressure. Collect the purged water volume for the verification of a good purge. Note the water flow time of the purge stroke.

6. Allow the tubing to refill. Repeat the purge. Collect the purge volume to assure the amount removed is at least the "port tube volume".
7. Purge a third time.
8. Allow the tubing to refill for the sample stroke.
9. Reduce the driving pressure to the "sampling pressure". Apply the pressure and collect the first flow to measure the discard volume. Discard that water into a container dedicated for investigation derived waste (IDW).
10. Reduce the pressure, if needed, to slow the flow to between 100 and 250 milliliters per minute (ml/min) and collect the samples.
11. Perform a final purge of the water out of the sampling lines by raising the driving pressure to the purge pressure value.
12. Upon sampling system refill, measure the water level in the "pump tube" to confirm depth to water table. If a port system is refilling very slowly, record the water level at a later time.

Geochemical and microbiological samples (Table 3-5) will be collected during the second synoptic event. Microbiological samples will be collected for Bioremediation Consulting Inc. (BCI) Functional Screening Tests. Groundwater from a dedicated discharge tube in a sampling port of a multi-port well is run directly into a laboratory supplied bottle filled with argon, displacing the argon. The bottles are then capped and sealed then placed in coolers cooled to 4°C with ice, for shipment under chain of custody to BCI of Watertown, MA. The cooler(s) will be sealed and secured in preparation for overnight delivery. QAPP Attachment 14 contains specific details from BCI on collection of microbiological samples.

Equipment needed to sample Water FLUTe™ System multi-port wells include:

- FLUTe™ Multi-Port 'Sampling Volume Spreadsheet' with recommended purge pressures specific for each port in each well;
- 100-foot bead type electric water level meter;
- Nitrogen gas canister, FLUTe™ supplied nitrogen regulators, valves, and pressure gages;
- NPT (0.25-inch) connectors;
- Nitrile Gloves;
- Flow through cell and manifold;
- Water quality meters including but not limited to; Oakton Model 11 (pH, temperature, conductivity, ORP) and Horiba U10 for additional listed parameters. SOPs LCP-006, 007, and 010 are attached to the QAM in Appendix A-B;
- Teflon lined sample tubing and connections;
- Purge/sampling logs and field notebook; and
- Dedicated containers for purged water.

4.5.2. Low Flow Sampling Procedures and Equipment

The 12 existing shallow monitoring wells will be sampled in accordance with the NJDEP low-flow groundwater sampling procedures provided in Appendix A-B and summarized below. Decontamination procedures are summarized in Section 4.8.

Upon arrival at each well, the well identification number will be entered into a field logbook, noting any damage to the well. New plastic sheeting will be placed over and around the monitoring well so that a 5 x 5 foot clean surface is created for the sampling equipment. All materials, tools and equipment will be decontaminated prior to the placement on the plastic sheeting. The depth to water below the reference point (top of casing) will be measured to the nearest hundredth of a foot (0.01 ft) using an electronic water level meter. The measuring device will be cleaned with phosphate-free detergent and rinsed with distilled water prior to collecting water level measurements.

A properly decontaminated low-flow sampling pump will be lowered into the well until the pump intake is in the middle of the well screen. The pump will be placed at the same depth during each sampling event. The well will be purged at a rate between 200 and 500 milliliters per minute (ml/min). Water levels and water quality parameters (i.e., pH, specific conductance, and temperature) will be monitored at five minute intervals during well purging, and recorded on Malcolm Pirnie groundwater purging/sampling logs. All purged water will be contained in 55-gallon drums. Once the water quality parameters have stabilized, groundwater samples will be collected into the appropriate sample containers. Samples will be collected from the pump discharge at a flow rate between 100 and 250 ml/min.

Equipment needed to sample shallow monitoring wells will include:

- 100-foot electric/electronic water level meter;
- 100-foot oil/water interface meter;
- Photoionization Detector (PID);
- Flow through cell;
- Teflon lined sample tubing and connections;
- Polyethylene or polypropylene tubing;
- Grundfos pump, manifold, and controller;
- Generator;
- Water quality meters including but not limited to; Oakton Model 11 (pH, temperature, conductivity, ORP) and Horiba U10 for additional listed parameters. SOPs LCP-006, 007, and 010 are attached to the QAM in Appendix A-B;
- Purge/sampling logs and field notebook; and
- Dedicated containers for purged and decontamination water.

Following the completion of the sample collection at any well or multi-port well, the well cap will be replaced, and the protective casing or manhole cover will be locked. The plastic sheeting and all used expendable materials will be

properly discarded. Prior to overnight shipment to the laboratory, samples will be packed on ice and placed inside a cooler along with the chain-of-custody (COC) forms. The cooler(s) will be sealed and secured in preparation for overnight delivery.

4.6. Bedrock Core Sampling Procedures and Equipment

The following procedures for collection of VOCs samples are based on 'Rock Core Investigation of DNAPL Penetration and TCE Mobility in Fractured Sandstone' (Hurley and Parker, 2002). Specific methods to collect and analyze rock core samples are presented in Stone Environmental's standard operating procedures SEI-6.37.0, in Appendix A-C. General decontamination procedures, in addition to procedures specific to rock core sample collection, are summarized in Section 4.8.

Rock core samples (approximately 2 inches, or 5 cm long) will be taken from the HQ core for analysis of VOCs and PCBs (Aroclors), based on the following criteria.

1. Adjacent to (above and below) fractures and bedding plane partings, and less than 6-inches from the fracture or parting. These are termed "fracture pairs" and emphasis will be made on fractures with secondary coatings, staining, slickensides, changes in use of drilling water, and changes in coring penetration rates.
2. Within 12 to 18 inches from a fracture, both above and below the fracture. These are also "fracture pairs".
3. At distinct changes in lithology/mineralogy that is not represented within the regular sampling interval. Collect within 6-inches from both sides of the boundary. These are termed "lithology pairs".
4. Additional samples where there is no continuous section of core greater than two (2) feet without a sample. These are termed "two-feet" frequency samples.

5. Duplicate samples will be collected at a frequency of 1/20. "Duplicates" will be collected from the same length of core split along the core axis.

The sample interval of 'every two feet' in Item (4) is considered by Hurley and Parker (2002) a reasonable spacing to provide adequate characterization of rock matrix.

The samples for VOCs will be immediately crushed and preserved in methanol for laboratory analysis. The samples for PCBs (Aroclors) (MW-14 and MW-16 only), will be crushed by Stone Environmental and preserved at 4°C for analysis by an outside laboratory.

As the cores are extracted, they will be placed in aluminum-foil lined PVC core-trays and removed from direct sunlight (to minimize volatilization of organic compounds). To analyze for VOCs and provide samples for PCBs (Aroclors) by an outside laboratory, approximately two inches (five centimeters) long samples of the core will be removed from the core tray, wrapped in aluminum foil, sealed in re-closeable plastic bags, and placed in coolers in preparation for crushing. To analyze for PCBs (Aroclors), Stone Environmental will split approximately 100g of the crushed rock, containerize the split in four ounce glass jars, seal in re-closeable plastic bags, and placed in coolers for sample management by Malcolm Pirnie. The geologist will log the remaining rock core.

Stone Environmental will provide a mobile laboratory at each core borehole location to conduct the VOCs analyses. Stone Environmental will remove the core samples and process them one at a time. The outer 0.5 cm of the core will be stripped off (to eliminate potential error introduced from drilling fluids). The samples will then be crushed and a minimum of 15 grams transferred to sample jars containing a known amount of methanol. During the crushing process, the sample should only be exposed to the outside atmosphere for five to ten seconds. The entire process must be completed in less than two

minutes to minimize volatile losses. Stone Environmental will then use the crushed samples preserved in methanol to test for VOCs.

Stone Environmental will split the crushed core to provide approximately 100 grams for analysis of PCBs (Aroclors). The crushed split will then be transferred to sample jars, sealed in re-closeable plastic bags, and placed in coolers cooled to 4°C with ice for shipment under chain of custody to an outside laboratory.

Samples for physical parameters, including TOC, will be collected at the frequency described in Section 3.2.3. After the sample is broken from the HQ core, Stone Environmental will remove a sub-section from the sample for TOC, by splitting the sample length-wise. The TOC sub-section will be sealed in re-closeable plastic bags, labeled, and placed in coolers cooled to 4°C with ice in preparation for crushing by Stone Environmental. Following crushing, the TOC sample will be placed in a container. Malcolm Pirnie will place the container in coolers cooled to 4°C with ice, in preparation for shipment under chain of custody to an outside laboratory.

Following removal of a TOC sub-section, Stone Environmental will wrap the physical parameters sample in aluminum foil twice around the circumference, followed by plastic wrap and tape. Stone Environmental will then vacuum bag and label the sample and place in a cooler cooled to 4°C with ice, in preparation for shipment under chain of custody to an outside laboratory.

Rock core samples for natural oxidant demand testing will be collected at the desired depths from borings MW-14 and MW-16. Approximately one-foot long core lengths will be collected from the HQ core, following collection of all other samples (Section 4.2.2). The samples will be collected and, wrapped in aluminum foil twice around the circumference, followed by plastic wrap and taped, the samples will be labeled placed in coolers cooled to 4°C with ice. The selected samples will be placed in coolers cooled to 4°C with ice for shipment under chain of custody to an outside laboratory.

4.7. Water Level Measurements

Two rounds of synoptic water levels will be measured in the proposed wells, existing wells, and at the three Bound Brook staff gauges that will be installed during the RI. The water level measurements will coordinate with the two synoptic groundwater sampling events. The purpose of measuring the water levels is to evaluate the direction of groundwater movement, vertical hydraulic gradients, seasonal or temporal fluctuations, and to evaluate the hydraulic connection between bedrock water table aquifer and Bound Brook.

The following equipment will be used to collect surface water level measurements at the staff gages, and groundwater level measurements in the 12 shallow bedrock monitoring wells:

- 100-foot electric/electronic water level meter;
- 100-foot Oil/water interface meter;
- PID;
- Nitrile Gloves;
- Decontamination equipment, including 5-gallon buckets, spray bottles with Alconox, acetone, tap water, deionized water, and paper towels;
- Dedicated containers for decontamination water;
- Field notebook; and

At each location, the locking steel protective cap and internal well cap will be removed. The organic vapor level escaping from the well will be monitored using a PID and the reading recorded in the field notebook. The product/water interface meter will be grounded to the steel casing and the probe slowly lowered into the well. When the probe enters the water a single light goes on. The date, time, well number, and depth to water will be recorded in the field notebook. The cable and probe of the water level meter will be decontaminated between use at each well by washing with Alconox and tap water, followed by a rinse with deionized water.

Three staff gauges will be installed along the length of Bound Brook. At each location, a staff gauge will be attached to a 2-by-6-inch board secured to an angle-iron post or to a permanent structure (example: bridge) within the flow of Bound Brook. The staff gauges will be near the former CDE facility at locations along Bound Brook, as shown on Figure 3-1. When reading the staff gauge, the distance from the surveyed mark at the top of the staff to top of the surface water will be recorded, to the nearest one-hundredth of a foot. The date, time, staff gauge number, and depth to water will be recorded in the field notebook.

The following equipment will be used to collect water levels from the Water FLUTe™ System multi-port wells:

- 100-foot bead type electric water level meter;
- Nitrogen gas canister, FLUTe™ supplied nitrogen regulators, valves, and pressure gages;
- NPT (0.25-inch) connectors;
- Nitrile gloves;
- PID; and
- Purge/sampling logs and field notebook.

4.8. Decontamination Procedures and Investigation-Derived Waste

General decontamination procedures are presented in SOP-9: Decontamination of Sampling Equipment presented in Appendix A-B. Decontamination procedures to be used on equipment required to collect 'analyze-immediately' inorganic (physical) parameters during groundwater sampling are presented in SOP LCP 001 which is part of the QAM in Appendix A-B. Decontamination procedures specific to rock core sub-sampling is presented in Stone Environmental's SOP SEI-6.37.0 (Appendix A-C).

All decontamination solutions, drilling derived dust, drill cuttings, soil cuttings, slurries and excess grout, recirculation water, and well development purge water (investigation derived waste – 'IDW') resulting from the installation and development of monitoring wells, and groundwater sampling will be transported

from each drilling location to the former CDE facility by a NJDEP licensed waste hauler. All liquid IDW will be transferred to and stored in a 10,000 gallon 'Frac Tank' or equivalent. Dust, slurries and excess grout, drill and soil cuttings will be stored in DOT-approved 55-gallon drums. Drums containing cuttings or other solids will be permanently numbered and labeled with the date and contents.

Drums will be stored at the former CDE facility in a temporary drum storage area identified by the USACE. The IDW subcontractor (licensed waste hauler) obtained by Malcolm Pirnie will complete the work necessary for the appropriate disposal of all drummed water, wastes and drill cuttings generated from this investigation. The contents of the drums will be sampled by the IDW subcontractor for waste characterization to determine proper disposal.

Composite samples will be collected and analyzed for Resource Conservation and Recovery Act (RCRA) Characteristics and Toxicity Characteristic Leaching Procedure (TCLP) VOCs. The method of disposal will be determined by the licensed waste hauler after all analytical results have been obtained. The waste hauler will dispose of the IDW according to federal, state, and local regulations. Malcolm Pirnie will retain copies of the waste manifests in the project files.

4.9. Surveying

A NJ licensed land surveyor will be contracted to conduct a survey of the multi-port monitoring wells following installation of all Water FLUTe™ systems. A survey will be conducted on each of the 17 multi-port wells to determine the elevation of the top of each well casing, protective casing, and sampling ports relative to Mean Sea Level (MSL) datum (NAVD 1929). The locations of the new wells will also be surveyed and tied to the USGS datum (NAD1988) and the New Jersey coordinate system using a benchmark located near the former CDE facility. Additionally, the measuring point elevation and location of each staff gauge installed in Bound Brook will also be surveyed.

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6. GLOSSARY OF ABBREVIATIONS

ANSETS	Analytical Services Tracking System
AOC	Administrative Order on Consent
ARARs	Applicable or Relevant and Appropriate Requirements
BGS	Below Ground Surface
BHHRA	Baseline Human Health Risk Assessment
cis-1,2-DCE	Cis-1,2-Dichloroethylene
CDE	Cornell-Dubilier Electronics
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act of 1980
CERCLIS	Comprehensive Environmental Response, Compensation and Liability Information System
CFR	Code of Federal Regulation
CKE	Currently Known Extent
CLP	Contract Laboratory Program
COPCs	Chemicals of Potential Concern
CSL	Comprehensive Site List
CSEM	Conceptual Site Exposure Model
1,1-DCE	1,1-Dichloroethylene
1,2-DCE	1,2-Dichloroethylene
DESA	Division of Environmental Science and Assessment
DFN	Discrete-Fracture Network
DNAPL	Dense Non-Aqueous Phase Liquid
DQO	Data Quality Objectives
EDR	Environmental Data Resources Inc.
ERNS	Emergency Response Notification System
FASTAC	Field and Analytical Services Teaming Advisory Committee

FS	Feasibility Study
FSP	Field Sampling Plan
gpd/ft	Gallons Per Day / Foot
GWQC	Ground Water Quality Criteria
HASP	Health and Safety Plan
IGWSCC	Impact to Groundwater Soil Cleanup Criteria
IDW	Investigation Derived Waste
K	Hydraulic Conductivity
MCLs	Maximum Contaminant Levels
MCLGs	Maximum Contaminant Level Goals
MSL	Mean Sea Level
MTBE	Methyl tert-Butyl Ether
NAPL	Non Aqueous Phase Liquid
NCEA	National Center for Environmental Assessment
NCP	National Contingency Plan
NJDEP	New Jersey Department of Environmental Protection
NJMCL	New Jersey Maximum Contaminant Level
NPDES	National Pollutant Discharge Elimination System
NPL	National Priorities List
OSHA	Occupational Safety and Health Administration
OU	Operable Unit
OU-1	Operable Unit-1
OU-2	Operable Unit-2
OU-3	Operable Unit-3
OU-4	Operable Unit-4
PAH	Polycyclic Aromatic Hydrocarbon
PCB	Polychlorinated Biphenyl
PCE	Tetrachloroethylene
pg/g	Picogram / Gram
pg/L	Picogram / Liter

PM	Project Manager
PPM	Parts Per Million
PRP	Potentially Responsible Party
QA/QC	Quality Assurance / Quality Control
QAM	Quality Assurance Manual
QAPP	Quality Assurance Project Plan
QCP	Quality Control Plan
RAS	Routine Analytical Services
RD	Remedial Design
RI	Remedial Investigation
RI/FS	Remedial Investigation / Feasibility Study
RME	Reasonable Maximum Exposure
RPM	Remedial Project Manager
ROD	Record of Decision
RSCC	Regional Sample Control Center
RTC	Response to Comments
SARA	Superfund Amendments and Reauthorization Act of 1986
SMO	Sample Management Officer
SOPs	Standard Operating Procedures
SSHP	Site-Specific Safety and Health Plan
SVOC	Semi Volatile Organic Chemical
TCA	1,1,1-Trichloroethane
TCE	Trichloroethylene
TCLP	Toxicity Characteristics Leaching Procedure
µg/Kg	Microgram / Kilogram
µg/L	microgram / Liter
USACE	U.S. Army Corps of Engineers
USEPA	U.S. Environmental Protection Agency
USGS	U.S. Geological Survey
VOC	Volatile Organic Chemical

Tables

TABLES

TABLE 1-1
PRELIMINARY CHEMICALS OF POTENTIAL CONCERN
CORNELL-DUBILIER ELECTRONICS SUPERFUND SITE
SOUTH PLAINFIELD, NEW JERSEY

CAS #	Chemical	Frequency of Detection	Range of Detected Concentrations µg/L	Risk-Based Screening Levels[1] µg/L		Potential ARARs Screening Levels[2] µg/L	Preliminary COPC[3]
Volatile Organics							
67-64-1	Acetone	6 / 79	4 -- 147	2,200	nc	6,000	NO
71-43-2	Benzene	3 / 79	1.59 -- 2.11	0.41	ca	1	YES
108-90-7	Chlorobenzene	5 / 79	12.8 -- 68.3	9.1	nc	50	YES
67-66-3	Chloroform	1 / 79	1.65	0.19	ca	70	YES
95-50-1	1,2-Dichlorobenzene	4 / 79	2.17 -- 5.99	37	nc	600	NO
541-73-1	1,3-Dichlorobenzene	4 / 79	2.8 -- 21.2	NA		600	YES
106-46-7	1,4-Dichlorobenzene	4 / 79	12.2 -- 48	0.43	ca	75	YES
75-35-4	1,1-Dichloroethylene	5 / 79	3.86 -- 22.5	34	nc	1	YES
156-59-2	cis-1,2-Dichloroethylene	50 / 79	1.36 -- 242000	37	nc	70	YES
156-60-5	trans-1,2-Dichloroethylene	3 / 79	21.5 -- 55.7	11	nc	100	YES
594-20-7	2,2-Dichloropropane	1 / 50	1.63	NA		NA	YES
591-78-6	2-Hexanone	1 / 50	3.29	NA		300	YES
1634-04-4	Methyl tert-butyl ether	10 / 50	2.79 -- 116	12	ca	70	YES
108-10-1	4-Methyl-2-Pentanone	1 / 50	2.14	200	nc	400	NO
127-18-4	Tetrachloroethylene	14 / 79	1.26 -- 3420	0.11	ca	1	YES
108-88-3	Toluene	23 / 79	1 -- 59.5	230	nc	600	NO
87-61-6	1,2,3-Trichlorobenzene	3 / 64	1.25 -- 26.4	NA		NA	YES
120-82-1	1,2,4-Trichlorobenzene	8 / 79	1.91 -- 1200	0.82	ca	9	YES
79-00-5	1,1,2-Trichloroethane	1 / 79	24.2	0.24	ca	3	YES
79-01-6	Trichloroethylene	70 / 79	1.27 -- 186000	1.7	ca	1	YES
75-01-4	Vinyl chloride	10 / 79	1 -- 209	0.016	ca	1	YES
--	p,m-Xylene	4 / 50	1.47 -- 1.72	20	nc	1,000	NO
Semi Volatile Organics							
117-81-7	Bis(2-ethylhexyl)phthalate	1 / 15	1	4.8	ca	3	NO
87-68-3	Hexachlorobutadiene	2 / 50	1.48 -- 9	0.86	ca	1	YES
91-20-3	Naphthalene	2 / 79	4 -- 5	0.14	nc	300	YES

TABLE 1-1
PRELIMINARY CHEMICALS OF POTENTIAL CONCERN
CORNELL-DUBILIER ELECTRONICS SUPERFUND SITE
SOUTH PLAINFIELD, NEW JERSEY

CAS #	Chemical	Frequency of Detection	Range of Detected Concentrations µg/L	Risk-Based Screening Levels[1] µg/L		Potential ARARs Screening Levels[2] µg/L	Preliminary COPC[3]
Pesticides							
309-00-2	Aldrin	11 / 15	0.022 -- 1.3	0.004	ca	0.04	YES
319-85-7	beta-BHC	1 / 15	0.016	0.037	ca	0.04	NO
319-86-8	delta-BHC	1 / 15	0.074	NA		100	YES
76-44-8	Heptachlor	1 / 15	0.13	0.015	ca	0.05	NO
PCBs and Dioxins/Furans							
11141-16-5	Aroclor 1232	10 / 15	0.53 -- 80	0.0068	ca	0.5[4]	YES
11097-69-1	Aroclor 1254	4 / 15	4.1 -- 9.2	0.034	ca	0.5[4]	YES
--	Total PCB congeners[5,6]	3 / 3	0.0038 -- 0.37	0.0068	ca	0.5[4]	YES
--	Total 2,3,7,8-TCDD TEQ[7]	2 / 4	0.0000025 -- 0.00043	0.00000052	ca	0.00001	YES
Inorganics							
7429-90-5	Aluminum	15 / 15	37.3 -- 747	3700	nc	50	NO
7440-36-0	Antimony	1 / 15	3	1.5	nc	6	NO
7440-38-2	Arsenic	2 / 15	3.4 -- 5.6	0.045	ca	3	YES
7440-39-3	Barium	15 / 15	79.4 -- 1610	730	nc	2,000	YES
7440-41-7	Beryllium	11 / 15	0.21 -- 0.33	7.3	nc	1	NO
7440-70-2	Calcium	30 / 30	19500 -- 128000	NA		NA	NO
7440-47-3	Chromium	13 / 15	3.8 -- 18	NA		70	YES
7440-48-4	Cobalt	8 / 15	0.66 -- 2.7	1.1	nc	100	YES
7440-50-8	Copper	15 / 15	2.1 -- 36.9	150	nc	1,300	NO
57-12-5	Cyanide	10 / 15	0.98 -- 5.6	73	nc	100	NO
7439-89-6	Iron	15 / 15	76.7 -- 1210	2600	nc	300	YES
7439-95-4	Magnesium	30 / 30	7570 -- 29000	NA		NA	NO
7439-96-5	Manganese	15 / 15	36.4 -- 2570	88	nc	50	YES

TABLE 1-1
PRELIMINARY CHEMICALS OF POTENTIAL CONCERN
CORNELL-DUBILIER ELECTRONICS SUPERFUND SITE
SOUTH PLAINFIELD, NEW JERSEY

CAS #	Chemical	Frequency of Detection	Range of Detected Concentrations µg/L	Risk-Based Screening Levels[1] µg/L		*Potential* ARARs Screening Levels[2] µg/L	Preliminary COPC[3]
Inorganics (Continued)							
7439-97-6	Mercury	2 / 15	0.11 -- 0.16	0.063	nc	2	YES
7440-02-0	Nickel	15 / 15	3.2 -- 42	73	nc	100	NO
7440-09-7	Potassium	15 / 15	1080 -- 7490	NA		NA	NO
7782-49-2	Selenium	1 / 15	4.5	18	nc	40	NO
7440-23-5	Sodium	15 / 15	13100 -- 43800	NA		50,000	NO
7440-62-2	Vanadium	14 / 15	1.3 -- 8.5	26	nc	NA	NO
7440-66-6	Zinc	10 / 15	1.5 -- 44.6	1100	nc	2,000	NO

1 = Risk-Based Screening Levels are the USEPA Region 3 Regional Screening Levels for tapwater (12 Sep 2008) accessed online at www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/index.htm. Consistent with USEPA Region 2 guidance, screening levels based on non-cancer health effects were reduced by one-tenth to represent a target hazard quotient (THQ) of 0.1.

2 = Potential ARARS, selected as the lowest of the Federal MCLs (40 CFR 141), State MCLs (N.J.A.C. 7:10-5.2 & 7.2), and the NJ Groundwater Quality Criteria (N.J.A.C. 7:9C) .

3 = All chemicals exceeding either screening level or without screening levels were selected as preliminary COPCs except the essential nutrients (*i.e.* , calcium, magnesium, potassium, and sodium).

4 = The Potential ARAR is for Total PCBs.

5 = Excludes the PCB congeners with dioxin-like toxicity included in the total 2,3,7,8-TCDD TEQ (*i.e.* , PCB-77, -81, -126, -169, -105, -114, -118, -123, -156, -157, -167, -189, -170, and -180).

6 = The screening level is for Aroclor 1232.

7 = Includes dioxins, furans, and PCBs listed in Section 2.3.5 of the Regional Screening Levels.
NA = Not Available.
nc = non cancer.
ca = cancer.

TABLE 3-1
RATIONALE FOR ROCK CORE BORINGS AND MULTI-PORT MONITORING WELLS
CORNELL-DUBILIER ELECTRONICS SUPERFUND SITE
SOUTH PLAINFIELD, NEW JERSEY

Proposed			Rationale
Well	Method	Test/s	
MW-13	Rotary tricone	Groundwater	Approximately in the direction of bedrock strike along Transect-A. Characterize groundwater in lateral plume fringe or boundary to the east of the Facility. Also provide eastern limit for mass discharge estimates along plane perdendicular to the assumed direction of groundwater flow (Transect C).
MW-14	Core and ream	Rock matrix & groundwater	Mid-Transect-A near MW-11 (TCE = 186,000 ppb, January 2008). Characterize rock matrix and groundwater from location of highest TCE in shallow groundwater.
MW-15	Rotary tricone	Groundwater	Southeast end of Transect-A. Characterize groundwater from near MW-08 (TCE = 19,000 ppb, January 2008).
MW-16	Core and ream	Rock matrix & groundwater	Transect-B along possible centerline of plume. Characterize rock matrix and groundwater to aid estimates of mass discharge. Also provides rock matrix and groundwater data for mass discharge estimates across Transect-C.
MW-17	Rotary tricone	Groundwater	Near ERT-2. Characterize groundwater immediately downgradient of north-western part of the Facility, and together with MW-13, MW-16, and MW-18 (Transect-C), enable estimates of contaminant mass discharge (i.e., along plane perpendicular to the assumed direction of groundwater flow).
MW-18	Rotary tricone	Groundwater	West of the Facility along strike. Characterize groundwater in lateral distal plume fringe or boundary zone, and deeper than currently sampled in ERT-5. Also provide western boundary for mass discharge estimates along plane perdendicular to the assumed direction of groundwater flow (Transect C).
MW-19	Core and ream	Rock matrix & groundwater	Transect-B along possible center of plume downgradient from the Facility. Characterize rock matrix (only if chlorinated VOCs are not detected in MW-20 bedrock samples) and groundwater.
MW-20	Core and ream	Rock matrix & groundwater	Transect-B, distal from the Facility, along possible center of plume. Characterize groundwater downgradient from the Facility. Closest well to Spring Lake water supply wells.
MW-21	Rotary tricone	Groundwater	Northwest of Facility and ERT-4. Characterize groundwater in lateral distal plume fringe or boundary zone.

Note: Transects A through C are shown on Figure 3-1.
Transect-A. Oriented approximately parallel to strike, NE-SW through MW-11 (the highest TCE values in shallow bedrock).
Transect-B. Oriented approximately down-dip, NW-SE through MW-11.
Transect-C. MW-13, MW-16, MW-17, and MW-18. This will be used to estimate mass discharge from the Facility.
'Facility' refers to the former CDE facility.
'Method' refers to drilling by either coring and reaming (using rotary tricone method), or by rotary tricone method only without core extraction.

TABLE 3-2
ROCK MATRIX CHARACTERIZATION SAMPLES
CORNELL-DUBILIER ELECTRONICS SUPERFUND SITE
SOUTH PLAINFIELD, NEW JERSEY

Boring	Analytical and Physical Parameters					Comment
	VOCs	PCBs (Aroclors)	Moisture, Porosity & Densities	TOC	Rock Oxidant Demand	
Estimated Number of Rock Matrix Physical Parameters Samples, from Deep Bedrock Borings for Water FLUTe Multi-Port wells						
MW-13	0	0	0	0	0	N.E. end Transect-A. No rock core samples. Water FLUTe multi-port monitoring well only.
MW-14	120	120	15	15	2	Facility COCs. Area of shallow groundwater high TCE. Facility Physical samples & TOC.
MW-15	0	0	0	0	0	Facility, S.W. end Transect-A. No rock core samples. Multi-port monitoring well only.
MW-16	120	120	15	15	2	Down gradient of area with high TCE in shallow groundwater. Facility Physical samples & TOC.
MW-17	0	0	0	0	0	Transect C. No rock core samples. Multi-port monitoring well only.
MW-18	0	0	0	0	0	Western end of Transect C. No rock core samples. Multi-port monitoring well only.
MW-19	120	0	15	15	0	Down-gradient, Transect-B. Possible plume core. Physical samples & TOC.
MW-20	120	0	15	15	0	Distal down-gradient, Transect-B. Possible distal plume core. Physical samples & TOC.
Total Primary Samples	480	240	60	60	4	
For QA/QC samples, see QAPP Worksheet 20.						

Note:
This table displays the anticipated number of primary analytical and physical parameter rock core samples.
Number of samples assumes: borings are 250 feet deep, depth to water of 10 feet, bottom of surface casings of 25 feet.

Rock Matrix Physical Parameters

Moisture & Porosity: Refers to moisture and porosity analyses of rock matrix.
Density: Refers to wet and dry bulk density of rock matrix.
TOC Total organic carbon in rock matrix.

Rock Oxidant Demand: Refers to rock oxidant demand bench test. Assumes one bench test per reagent. Assumes two reagents, therefore two tests per sample for a total of 2*4 = 8 tests.

TABLE 3-2
ROCK MATRIX CHARACTERIZATION SAMPLES
CORNELL-DUBILIER ELECTRONICS SUPERFUND SITE
SOUTH PLAINFIELD, NEW JERSEY

Rationale For Collection Depths of Rock Matrix Physical Parameters					
Boring	On or Off the former CDE facility	Physical Parameter			
		Moisture, Porosity & Densities	TOC	Rock Oxidant Demand	Rationale, Assuming Termination Depths of 250 feet, bgs.
MW-14	On	12	12		Siltstone is expected to dominate. Collect samples every 20 feet from top; 12 samples. If mudstone is present collect 3 more where available.
		3	3		Three contingency samples for lithological variation.
				2	Collect in siltstone and mudstone (if present). Biase toward intervals of lowest or non-detect VOCs and PCBs.
MW-16	On	12	12		Siltstone is expected to dominate. Collect samples every 20 feet from top; 12 samples. If mudstone is present collect 3 more where available.
		3	3		Three contingency samples for lithological variation.
				2	Collect in siltstone and mudstone (if present). Biase toward intervals of lowest or non-detect VOCs and PCBs.
MW-19	Off Down-gradient	12	12		Mudstone may dominate to 110 feet, siltstone deeper. Collect samples every 20 feet from top; 12 samples. Three additional contingency samples for variation in rock type.
		3	3		Three contingency samples for lithological variation. Two samples in mudstone from 25 to about 110 feet, at approximately 45 and 85 feet bgs. Third sample in siltstone, below mudstone/siltstone contact at about 110 feet bgs.
MW-20	Off Distal down-gradient	12	12		Mudstone may dominate to 225 feet, siltstone deeper. Collect samples every 20 feet from top; 12 samples. Three additional contingency samples for variation in rock type.
		3	3		Three contingency samples for lithological variation. Two samples in mudstone from 25 to about 225 feet bgs, at approximately 65 and 165 feet bgs. Third sample in siltstone below mudstone/siltstone contact at about 225 feet bgs.

TABLE 3-3
MONITORING WELL CONSTRUCTION DATA
CORNELL-DUBILIER ELECTRONICS SUPERFUND SITE
SOUTH PLAINFIELD, NEW JERSEY

Well ID	Top of Bedrock (Feet Below Surface)	Bedrock Elevation (Feet)	Screen Top (Feet Below Surface)	Screen Bottom (Feet Below Surface)	Screen Length (Feet)	Depth to Water (Feet Below Surface)	Surface Elevation (Feet)
Existing Shallow Bedrock Wells							
MW01A	6	64.48	24	49	25	15.66'	70.48
MW02A	12	59.22	24	49	25	14.29'	71.22
MW03	8	63.11	17	32	15	12.84'	71.11
MW04	13	59.61	29	49	20	13.26'	72.61
MW05	11	59.79	25	45.5	20.5	13.94'	70.79
MW06	6	68.83	29	44	15	17.36'	74.83
MW07	6	76.16	43	58	15	24.44'	82.16
MW08	7	68.47	42	57.5	15.5	20.34'	75.47
MW09	14	60.83	29	54	25	17.40'	74.83
MW10	4	68.82	37	52	15	15.89'	72.82
MW11	14	61.18	34	59	25	19.42'	75.18
MW12	10	65.92	35	60	25	18.28'	75.92

Existing Deep Bedrock Wells

Well ID	Top of Bedrock (Feet Below Surface)	Depth of 10" Casing (Feet)	Depth of 6" Casing (Feet)	# of Ports	Depth to Water (Feet Below Surface)
ERT-1	Approximately 15	12	25	8	8.05
ERT-2	Approximately 15	12	25	7	19.37
ERT-3	Approximately 15	12	25	6	6.11
ERT-4	Approximately 15	12	25	7	0.65
ERT-5	Approximately 15	12	25	6	8.45
ERT-6	Approximately 15	15	25	5	5.05
ERT-7	3	5.5	25	Note 1	17.31
ERT-8	2	12	25	7	17.16

Note 1: ERT-7 not yet equipped with a Water FLUTe multi-port System. The number of ports, depths, and lengths to be finalized during the RI field program.

TABLE 3-4
GROUNDWATER SAMPLE INTERVALS AND NUMBER OF ANALYTICAL SAMPLES
CORNELL-DUBILIER ELECTRONICS SUPERFUND SITE
SOUTH PLAINFIELD, NEW JERSEY

Existing Well	Single Screen or Water FLUTe port #	Sample Interval Length (Screen or Port), (ft)	Sample Interval (ft, bgs)		Sample Method	Primary Analytical Samples, per Synoptic Event				
			Top	Bottom		TCL VOC	TCL SVOC	PCBs (Aroclors)	TCL Pesticide	TAL Metals (unfiltered) and Cyanide
Existing Shallow Bedrock Monitoring Wells, Equipped With Single Screens										
MW-01A	1	25	24	49	EPA Region II Low flow	1	1	1	1	1
MW-02A	1	25	24	49	EPA Region II Low flow	1	1	1	1	1
MW-03	1	15	17	32	EPA Region II Low flow	1	1	1	1	1
MW-04	1	20	29	49	EPA Region II Low flow	1	1	1	1	1
MW-05	1	20.5	25	45.5	EPA Region II Low flow	1	1	1	1	1
MW-06	1	15	29	44	EPA Region II Low flow	1	1	1	1	1
MW-07	1	15	43	58	EPA Region II Low flow	1	1	1	1	1
MW-08	1	15.5	42	57.5	EPA Region II Low flow	1	1	1	1	1
MW-09	1	25	29	54	EPA Region II Low flow	1	1	1	1	1
MW-10	1	15	37	52	EPA Region II Low flow	1	1	1	1	1
MW-11	1	25	34	59	EPA Region II Low flow	1	1	1	1	1
MW-12	1	25	35	60	EPA Region II Low flow	1	1	1	1	1
					Total	12	12	12	12	12
Existing Deep Bedrock Multi-Port Monitoring Wells										
ERT-1	1	5	24	29	Water FLUTe method	1	1	1	1	1
	2	10	33	43	Water FLUTe method	1	1	1	1	1
	3	10	46	56	Water FLUTe method	1	1	1	1	1
	4	5	59	64	Water FLUTe method	1	1	1	1	1
	5	10	67	77	Water FLUTe method	1	1	1	1	1
	6	5	100	105	Water FLUTe method	1	1	1	1	1
	7	5	112	117	Water FLUTe method	1	1	1	1	1
	8	5	135	140	Water FLUTe method	1	1	1	1	1
ERT-2	1	10	25	35	Water FLUTe method	1	1	1	1	1
	2	10	40	50	Water FLUTe method	1	1	1	1	1
	3	5	54	59	Water FLUTe method	1	1	1	1	1
	4	5	70	75	Water FLUTe method	1	1	1	1	1
	5	10	97	107	Water FLUTe method	1	1	1	1	1
	6	10	113	123	Water FLUTe method	1	1	1	1	1
	7	10	127	137	Water FLUTe method	1	1	1	1	1
ERT-3	1	10	27	37	Water FLUTe method	1	1	1	1	1
	2	10	55	65	Water FLUTe method	1	1	1	1	1
	3	15	90	105	Water FLUTe method	1	1	1	1	1
	4	10	110	120	Water FLUTe method	1	1	1	1	1
	5	10	124	134	Water FLUTe method	1	1	1	1	1
	6	10	138	148	Water FLUTe method	1	1	1	1	1

TABLE 3-4
GROUNDWATER SAMPLE INTERVALS AND NUMBER OF ANALYTICAL SAMPLES
CORNELL-DUBILIER ELECTRONICS SUPERFUND SITE
SOUTH PLAINFIELD, NEW JERSEY

Existing Well	Single Screen or Water FLUTe port #	Sample Interval Length (Screen or Port), (ft)	Sample Interval (ft, bgs)		Sample Method	Primary Analytical Samples, per Synoptic Event				
			Top	Bottom		TCL VOC	TCL SVOC	PCBs (Aroclors)	TCL Pesticide	TAL Metals (unfiltered) and Cyanide
ERT-4	1	10	27	37	Water FLUTe method	1	1	1	1	1
	2	10	46	56	Water FLUTe method	1	1	1	1	1
	3	5	61	66	Water FLUTe method	1	1	1	1	1
	4	5	83	88	Water FLUTe method	1	1	1	1	1
	5	15	91	106	Water FLUTe method	1	1	1	1	1
	6	5	111	116	Water FLUTe method	1	1	1	1	1
	7	10	128	138	Water FLUTe method	1	1	1	1	1
ERT-5	1	10	24	34	Water FLUTe method	1	1	1	1	1
	2	10	37	47	Water FLUTe method	1	1	1	1	1
	3	10	50	60	Water FLUTe method	1	1	1	1	1
	4	10	77	87	Water FLUTe method	1	1	1	1	1
	5	5	93	98	Water FLUTe method	1	1	1	1	1
	6	10	120	130	Water FLUTe method	1	1	1	1	1
ERT-6	1	10	26	36	Water FLUTe method	1	1	1	1	1
	2	10	75	85	Water FLUTe method	1	1	1	1	1
	3	10	93	103	Water FLUTe method	1	1	1	1	1
	4	10	107	117	Water FLUTe method	1	1	1	1	1
	5	10	128	138	Water FLUTe method	1	1	1	1	1
ERT-7 (to be equipped with Water FLUTe)	1		Existing boring not yet equipped with a Water FLUTe (multi-port) System. The number of ports is an estimate only. Number, depths, and lengths of sample interval ports to be determined following the liner drop test.		Water FLUTe low flow	1	1	1	1	1
	2				Water FLUTe method	1	1	1	1	1
	3				Water FLUTe method	1	1	1	1	1
	4				Water FLUTe method	1	1	1	1	1
	5				Water FLUTe method	1	1	1	1	1
	6				Water FLUTe method	1	1	1	1	1
	7				Water FLUTe method	1	1	1	1	1
ERT-8	1	10	17	27	Water FLUTe method	1	1	1	1	1
	2	10	31	41	Water FLUTe method	1	1	1	1	1
	3	10	44	54	Water FLUTe method	1	1	1	1	1
	4	5	57	62	Water FLUTe method	1	1	1	1	1
	5	10	87	97	Water FLUTe method	1	1	1	1	1
	6	5	107	112	Water FLUTe method	1	1	1	1	1
	7	10	135	145	Water FLUTe method	1	1	1	1	1
Total for all existing MW & ERT wells; assuming 7 ports in ERT-7						65	65	65	65	65

TABLE 3-4
GROUNDWATER SAMPLE INTERVALS AND NUMBER OF ANALYTICAL SAMPLES
CORNELL-DUBILIER ELECTRONICS SUPERFUND SITE
SOUTH PLAINFIELD, NEW JERSEY

Proposed Deep Bedrock Water FLUTe Multi-Port Monitoring Wells										
Adjacent Existing Well	Proposed Multi-Port Well	Water FLUTe Port	Sample Interval (ft, bgs)		Sample Method	Primary Analytical Samples, per Synoptic Event				
			Top	Bottom		TCL VOC	TCL SVOC	PCBs (Aroclors)	TCL Pesticides	TAL Metals (unfiltered) and Cyanide
	MW-13	Assume 10	Water FLUTe System number, depths, and lengths of sample intervals ports to be determined following analyses of rock matrix samples, FLUTe liner drop tests, and high resolution temperature logging.		Water FLUTe method	10	10	10	10	10
	MW-14	Assume 10			Water FLUTe method	10	10	10	10	10
	MW-15	Assume 10			Water FLUTe method	10	10	10	10	10
	MW-16	Assume 10			Water FLUTe method	10	10	10	10	10
ERT-2	MW-17	Assume 5			Water FLUTe method	5	5	5	5	5
ERT-5	MW-18	Assume 5			Water FLUTe method	5	5	5	5	5
ERT-3	MW-19	Assume 5			Water FLUTe method	5	5	5	5	5
	MW-20	Assume 10			Water FLUTe method	10	10	10	10	10
	MW-21	Assume 10			Water FLUTe method	10	10	10	10	10
Total for all Proposed Multi-Port Wells						75	75	75	75	75
SUMMARY OF WELLS										
Number of Sampling Ports (Existing and Proposed) Located at the Former CDE Facility						50	50	50	50	50
Number of Sampling Ports (Existing and Proposed) Located Outside the Former CDE Facility Boundaries						90	90	90	90	90
Total Number of all Existing and Proposed Multi-Port Wells and Existing Shallow Bedrock Wells						140	140	140	140	140
Total for Two (2) Synoptic Events						280	280	280	280	280
Water Supply Sample (Prior to Commencement of Drilling)						1	1	1	1	1
For Number of QA/QC samples, see QAPP Worksheet 20.										

Note:
1. Assumes 2 (two) synoptic events.
2. PCBs (Congeners) and dioxins/furans will only be analyzed in the second synoptic event and only from those screens and ports where PCBs (Aroclors) were detected in the first synoptic event. Therefore the number of samples cannot be pre-determined.
3. Only PCBs (Aroclors & Congeners), dioxins/furans will be analyzed in a third limited extent sampling event from those screens and ports from which PCBs (Aroclors) were detected in the first synoptic event. Therefore the wells and number of samples cannot be pre-determined.

TABLE 3-5
BEDROCK BASELINE GROUNDWATER GEOCHEMICAL CHARACTERIZATION
CORNELL-DUBILIER ELECTRONICS SUPERFUND SITE
SOUTH PLAINFIELD, NEW JERSEY

Existing Well	Proposed Well	Geochemical Parameter						Microbiological	Purpose
		TOC/ DOC	Fe^{+2}	NO_3^-, Cl^-, SO_4^{2-}	methane, ethane, ethene	alkalinity	hardness		
Existing Deep Bedrock (all ports of multi-port well)									
ERT-1		8	8	8	8	8	8	5	Background up-gradient monitoring well. Microbiological samples evenly distributed throughout well.
Proposed Deep Bedrock Well (all ports of each multi-port well)									
	MW-13	10	10	10	10	10	10		Lateral (side gradient), and along strike from Transect-A, and east end of mass discharge Transect-C.
	MW-14	10	10	10	10	10	10	5	Area of highest shallow bedrock TCE in plume core. Microbiological samples evenly distributed throughout well.
	MW-16	10	10	10	10	10	10	5	Possible plume core. Microbiological samples evenly distributed throughout well.
	MW-20	10	10	10	10	10	10	5	Closest to Spring Lake wells. Transect-B, distal plume core. Microbiological samples evenly distributed throughout well.
	MW-21	10	10	10	10	10	10		Possible downgradient plume boundary.
Primary Samples Per Event		58	58	58	58	58	58	20	Total per synoptic event.
Total Samples, Two Events		116	116	116	116	116	116	20	Total for Two (2) Synoptic Events.
For QA/QC samples, see QAPP Worksheet 20.									

1). U.S. EPA Environmental Response Team, VOC Results in Groundwater, January 2008 was used to indicate the possible extent of groundwater contamination.
2). Proposed number of samples may be modified based on results of rock matrix analytical results and existing groundwater samples.
3). Geochemical parameters samples will be collected during the first and second synoptic events. Microbiological samples will be collected during the second synoptic event.

TABLE 3-6
EXISTING AND PROPOSED PRESSURE TRANSDUCERS
CORNELL-DUBILIER ELECTRONICS SUPERFUND SITE
SOUTH PLAINFIELD, NEW JERSEY

Existing Well	Proposed Well	FLUTe Port #	Sample Interval (ft, bgs)		Pressure Transducer
			Top	Bottom	
Existing Shallow Bedrock Monitoring Wells					
MW-01A			24	49	1
MW-02A			24	49	1
MW-03			17	32	1
MW-04			29	49	1
MW-05			25	45.5	1
MW-06			29	44	1
MW-07			43	58	1
MW-08			42	57.5	1
MW-09			29	54	1
MW-10			37	52	1
MW-11			34	59	1
MW-12			35	60	1
Existing Deep Bedrock (ERT Series) Multi-Port Monitoring Well					
ERT-2		1	25	35	1
		2	40	50	1
		3	54	59	1
		4	70	75	1
		5	97	107	1
		6	113	123	1
		7	127	137	1
ERT-3		1	27	37	1
		2	55	65	1
		3	90	105	1
		4	110	120	1
		5	124	134	1
		6	138	148	1
ERT-4		1	27	37	1
		2	46	56	1
		3	61	66	1
		4	83	88	1
		5	91	106	1
		6	111	116	1
		7	128	138	1
Total for Existing Wells					20

TABLE 3-6
EXISTING AND PROPOSED PRESSURE TRANSDUCERS
CORNELL-DUBILIER ELECTRONICS SUPERFUND SITE
SOUTH PLAINFIELD, NEW JERSEY

Existing Well	Proposed Well	FLUTe Port #	Sample Interval (ft, bgs)		Pressure Transducer
			Top	Bottom	
Proposed Deep Bedrock Multi-Port Monitoring Well and Pressure Transducers					
Adjacent Existing ERT Well	Proposed Well	FLUTe Port #	Sample Interval (ft, bgs)		Pressure Transducer
			Top	Bottom	
	MW-13	Assume 10	unknown		10
	MW-14	Assume 10	unknown		10
	MW-15	Assume 10	unknown		10
	MW-16	Assume 10	unknown		
ERT-2	MW-17	Assume 5	unknown		
ERT-5	MW-18	Assume 5	unknown		
ERT-3	MW-19	Assume 5	unknown		5
	MW-20	Assume 10	unknown		10
	MW-21	Assume 10	unknown		
Existing ERT-7 to be completed.	ERT-7	Assume 7	unknown		
Total for Proposed Multi-Port Deep Bedrock Wells					45

Note:
1) Transducers will be temporarily installed in existing shallow bedrock wells during aquifer pump test only.
2) Assumes proposed deep bedrock multi-port wells will be installed with 10 ports each unless otherwise noted.
3) Assumes that ports will be located between 25 and 250 feet below ground surface.
4) Ports installed in MW-17, 18, and 19 will be installed deeper than those installed in adjacent existing ERT wells.

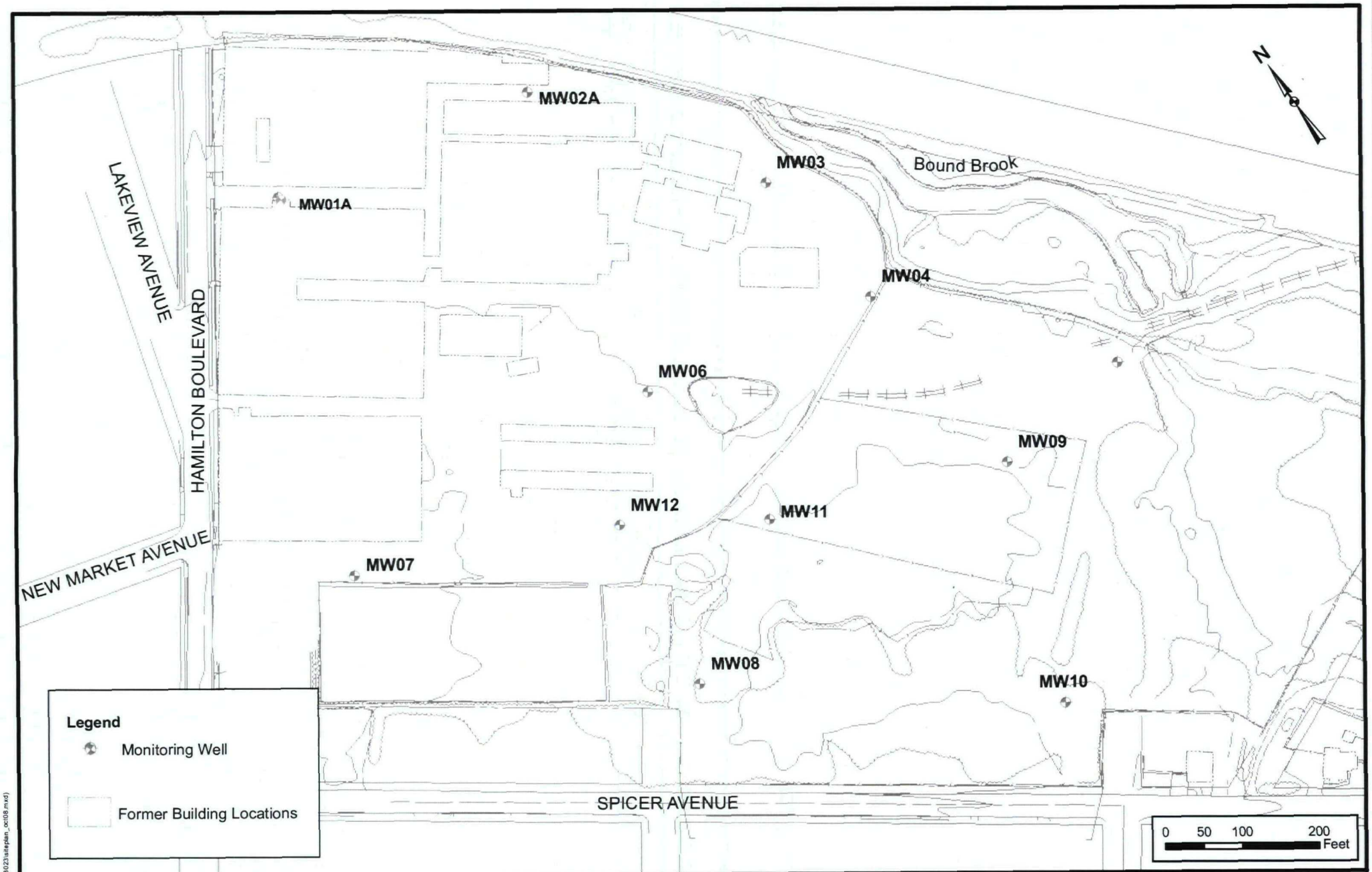
FIGURES

1000 0 1000 2000 3000 4000 5000 6000 7000 FEET

SOURCE: U.S.G.S. TOPOGRAPHIC MAP,
7.5 MINUTE SERIES, PLAINFIELD, NEW JERSEY
QUADRANGLE, 1955, PHOTOREVISED 1981

MALCOLM PIRNIE	U.S. ARMY CORPS OF ENGINEERS CORNELL–DUBILIER SUPERFUND SITE SOUTH PLAINFIELD, NEW JERSEY CONTRACT NO. W912DQ–06–D–0006	FORMER CDE FACILITY LOCATION MAP SCALE AS NOTED	MALCOLM PIRNIE, INC. OCTOBER 2008 FIGURE 2–1

User: monsanto Spec: PIRNIE STANDARD File: I:\ACAD\PROJ\455303\CLIENT\BUFFALO\04-03-07\SITE-LOC-455303-D2.DWG Scale: 1:1 Date: 10/28/2008 Time: 17:36 Layout: FIGURE 1



MALCOLM PIRNIE

CORNELL-DUBILIER ELECTRONICS
SUPERFUND SITE
SOUTH PLAINFIELD, NEW JERSEY

FORMER CDE FACILITY PLAN

MALCOLM PIRNIE, INC.

DECEMBER 2008
FIGURE 1-2

Map Document: (S:\Projects\455302\siteplan_oct08.mxd)
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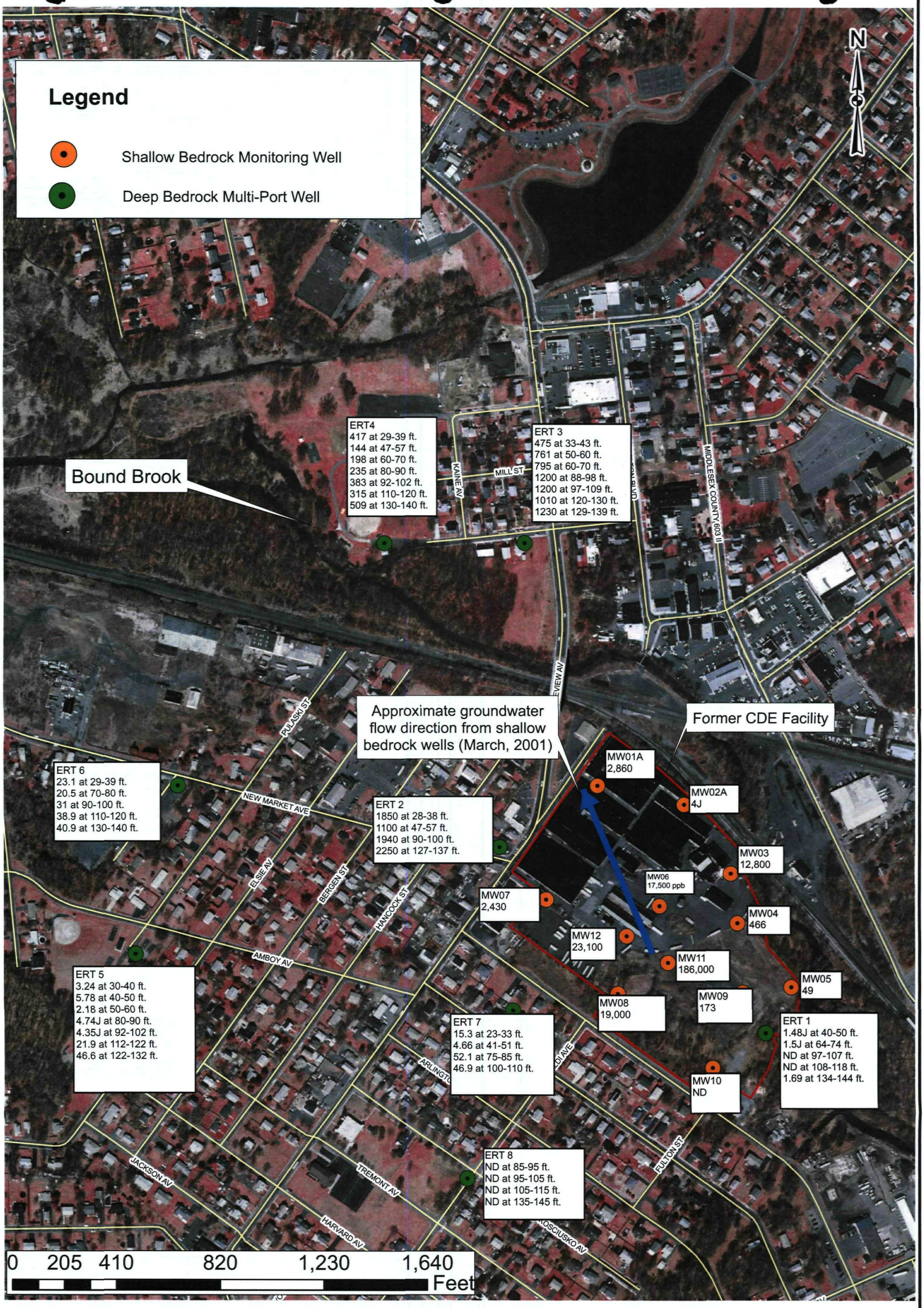

Note: Data represents packer test samples (ERT wells) and low flow sampling (MW wells).
TCE results in parts per billion (ppb) rounded to nearest significant figure.
J = Estimated Value. ND = Not Detected

MALCOLM PIRNIE	Cornell-Dubilier Electronics Superfund Site South Plainfield, New Jersey	Trichloroethene in Groundwater January 2008 Investigation	MALCOLM PIRNIE, INC. December 2008 FIGURE 1-3

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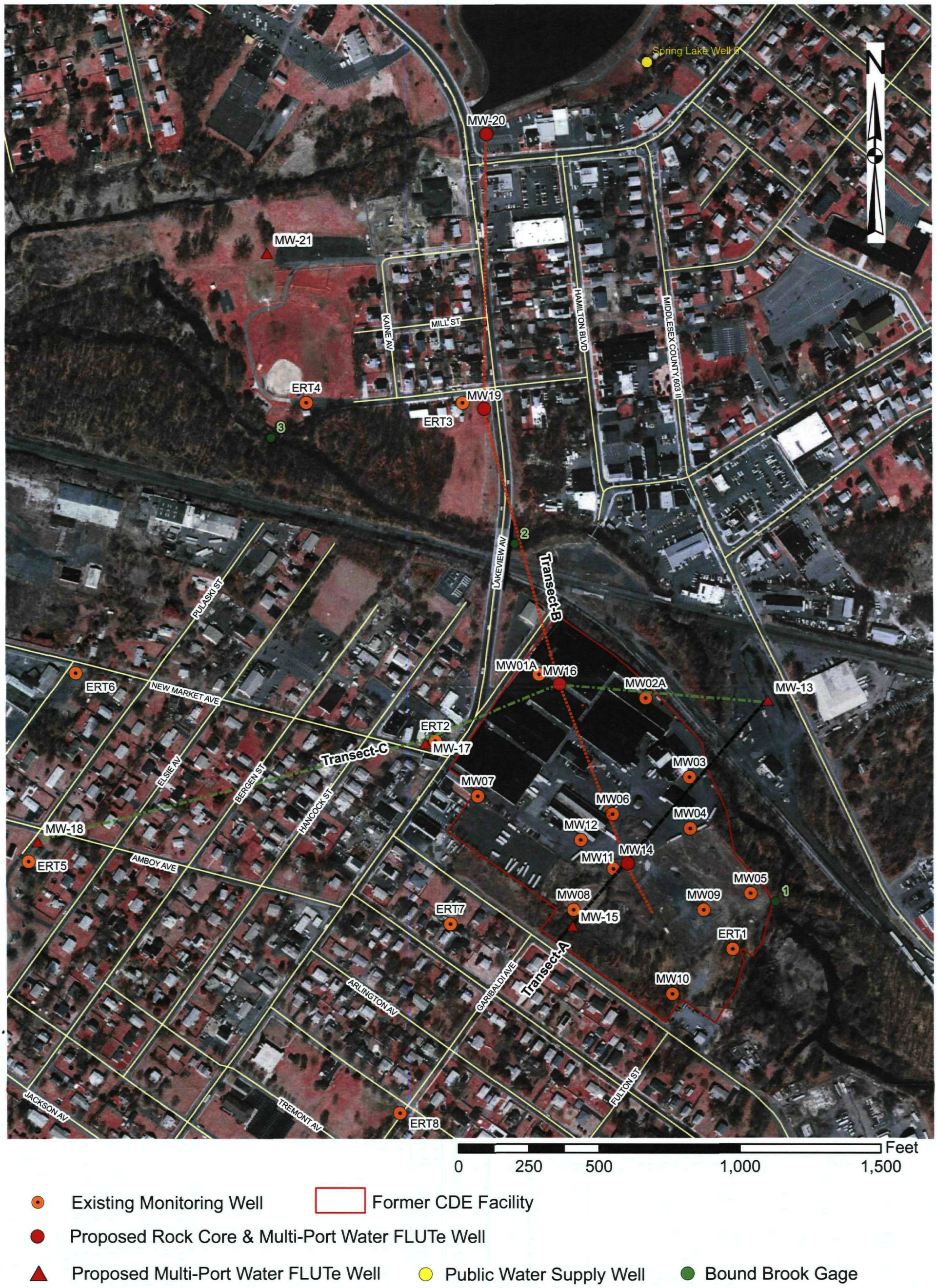

MALCOLM PIRNIE

Cornell-Dubilier Electronics Superfund Site
South Plainfield, New Jersey

Locations of Proposed Rock Cores & Monitoring Wells

MALCOLM PIRNIE, INC.

December 2008

FIGURE 3-1

Map Document: (P:\4553058\GIS\Cornell Figure mxds in 9.1\Proposed_Wells_With_Lables1.mxd)
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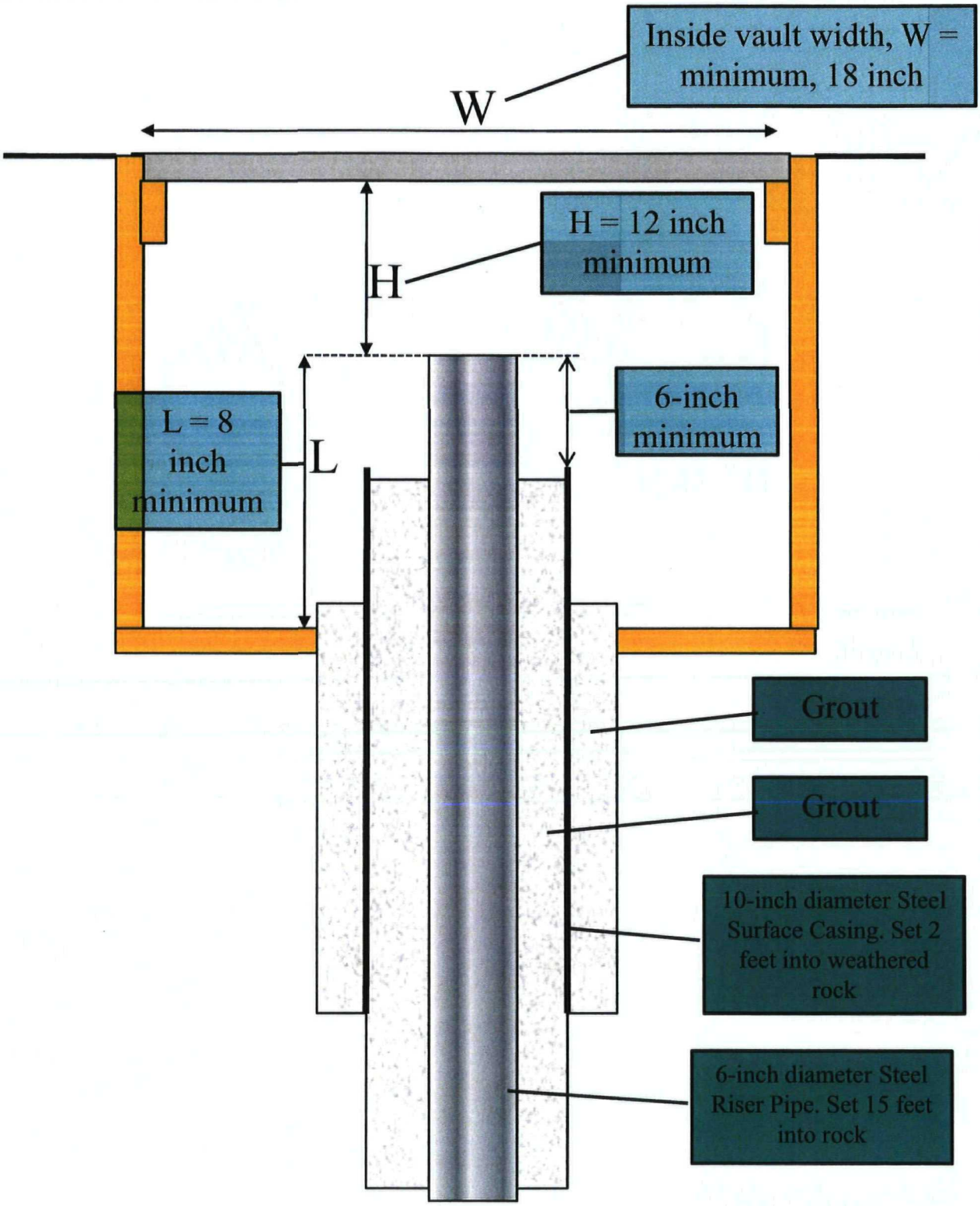
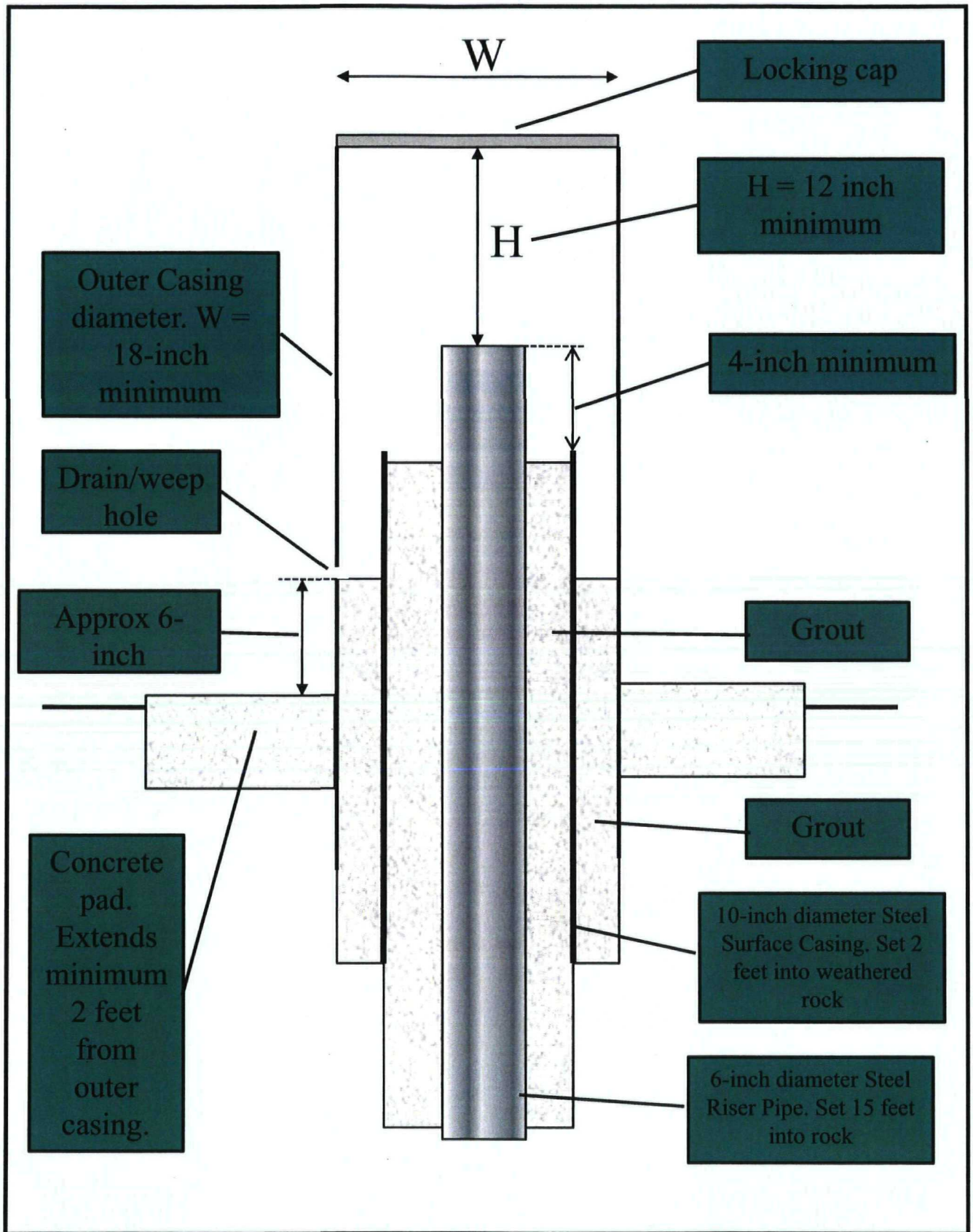


MALCOLM PIRNIE

CORNELL-DUBILIER SUPERFUND SITE
SOUTH PLAINFIELD, NEW JERSEY
Subsurface Vault for WaterFLUTe System

MALCOLM PIRNIE, INC.

FIGURE 4-1



MALCOLM PIRNIE

CORNELL-DUBILIER SUPERFUND SITE
SOUTH PLAINFIELD, NEW JERSEY
Stickup Completion for WaterFLUTe System

MALCOLM PIRNIE, INC.

FIGURE 4-2

Appendices

APPENDIX A-A
FLUTE™ GUIDANCE DOCUMENTS

FLUTe™ Flexible Liner Underground Technologies, Ltd. L.C.
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6 Easy St., Santa Fe, NM 87506
505-455-1300, www.flut.com

Guide for the use of FLUTe Hydraulic Conductivity Profiler Results

Guide for the use of FLUTe Hydraulic Conductivity Profiler results

Purpose

The FLUTe hydraulic conductivity profiling technique has evolved to a highly useful measure of the flow capacity of all significant flow paths intersected by a borehole. However, the hydrologic community is not yet widely familiar with the method nor the use of the results. This guide is to explain how the method works and more importantly how the results can be used. Unlike many geophysical methods, the FLUTe profiler results are a direct quantitative measure of the flow capacity of each significant flow path intersected by the borehole. The FLUTe results will be compared to some traditional measurements for a better understanding of the utility. (A paper on this method for journal publication is being written by Carl Keller, Profs. John Cherry and Beth Parker.)

The method

The FLUTe blank flexible liner is driven down the hole by the excess head of water inside the liner above the water table of the formation. That driving head inside the liner is approximately the driving pressure forcing the water from the hole. The driving pressure beneath the liner is essentially uniform throughout the borehole and maintained relatively constant during the measurement.

The data recorded during the liner installation are: liner depth, time, driving head, and tension on the liner. The liner tension is also controlled to be relatively constant as is the driving head inside the liner. From the data is calculated the liner velocity per unit driving head as a function of the liner depth in the hole.

As a liner is everted into the borehole, the liner descends like a perfectly fitting piston and forces the water from the borehole into the formation. When the liner starts down the hole, all the flow paths in the borehole are available for the displacement of the water. In other words, the entire transmissivity of the borehole is available for the acceptance of the water displaced by the liner.

The liner velocity down the borehole is controlled by the transmissivity of the borehole beneath the liner and the excess head in the liner. As the liner starts its descent in the borehole, it accelerates to a relatively high velocity and then quickly drops to the velocity associated with the steady state flow of the water from the hole. Thereafter, there is a relatively constant driving pressure in the borehole and a steady state radial outward flow of the water from the hole as the liner descends.

However, as the liner descends, it sequentially covers and seals the flow paths in the borehole from the top down (Fig. 1). Each time a flow path is sealed by the descending everting liner, the remaining transmissivity of the borehole is decreased by that of the flow path sealed. The drop in transmissivity causes a drop in the liner velocity as each flow path is sealed. The result is a typical velocity of the liner with depth as shown in Fig. 2. The drop in velocity provides a direct measurement of the flow that was stopped when the flow path was sealed.

Hence, the step change in velocity occurs at the location of the flow path, and the magnitude of the step change is a direct measure of the flow capacity of that flow path. As the liner passes through an extended region of flow (e.g., a permeable bed or highly fractured region) the velocity graph shows a relatively continuous decline in velocity.

From the velocity profile a transmissivity profile is calculated.

How the transmissivity profile is calculated

The change in liner velocity, dv, multiplied by the cross section of the borehole, Az, is defined as the flow, Qr, that was terminated when the interval of the borehole, dz, was sealed by the liner. Assuming a 1D radial flow into the borehole wall at a flow rate, Qr, over the interval, dz, and a hole wall area, $Ar = 2 \pi r_o dz$, we get:

$$Qr = dv\ Az = 2 \pi\ dz\ C\ dH / \ln(r/r_o)$$

Where C is the conductivity of the interval, dH is the driving head in the borehole, r_o is the hole radius, and r is the range to the ambient pressure in the formation. Since $\ln(r/r_o)$ is such a slowly varying function with r, even if r is not known, it is usually assumed to be a constant. From this equation, the transmissivity, T= dz C, is calculated or the conductivity C for the interval dz. Both are relevant to the interval dz over which the velocity change occurs. Since the velocity change is calculated for each time step, typically every 2 seconds, the length dz is the distance traversed by the liner descent in that time step.

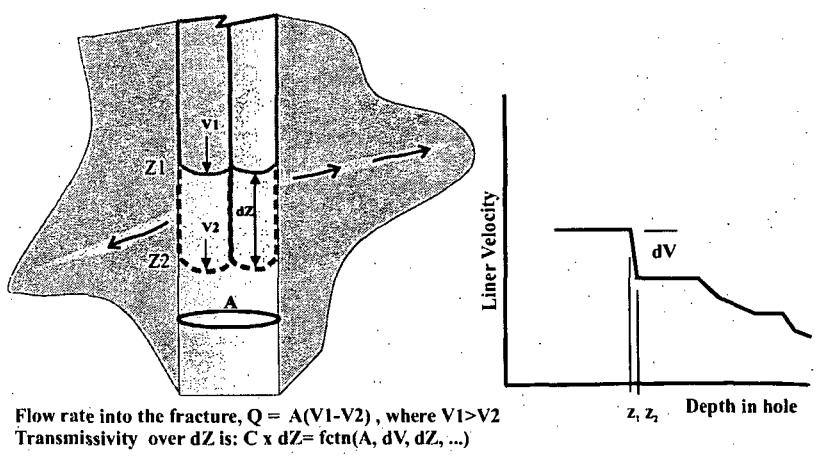


Fig. 1. The transmissivity over the interval dZ is calculated from the liner depths and velocity change between those depths.

Therefore, when the liner is traveling fast the spatial resolution is less than when the liner is traveling slowly.

How to use the results

The graph of the velocity with depth, Fig. 2, shows very clearly where the major flow paths are located. They are, for example, at 60, 140, and 245 ft. The relative change of each velocity step is the relative flow rate into that zone (i.e., the largest change in velocity corresponds to the largest flow rate into the hole wall). Therefore, the flow path at 60 ft. is much larger than the flow path at 87 ft. The smaller steps are slower flowing beds or fractures. The sloping portions of the curve are permeable zones such as highly fractured or highly porous/permeable beds. (Note, whereas we refer to "the velocity"

above, the actual plot is the velocity divided by the driving pressure or velocity per unit driving pressure. Therefore, moderate variations in the driving pressure throughout the hole are not important.)

If one wishes to use the measurement in a flow model, the transmissivity calculated from the velocity curve, Fig. 3, is the correct value for each interval of the calculation. The velocity data and the transmissivity data as a function of the depth in the hole are available from FLUTe. The actual data can be used in numerical models of the flow field. The results are normally provided in graphic form.

However, the velocity plot vs. depth is sufficient by itself for the identification of the significant flow paths in the borehole. The actual data plots are useful for plotting adjacent to other measurements in the borehole such as fracture maps, and video logs. Large changes in liner velocity are typically associated with visible fractures and bedding planes in the video log.

Fig. 2 Liner Velocity from 4" hole

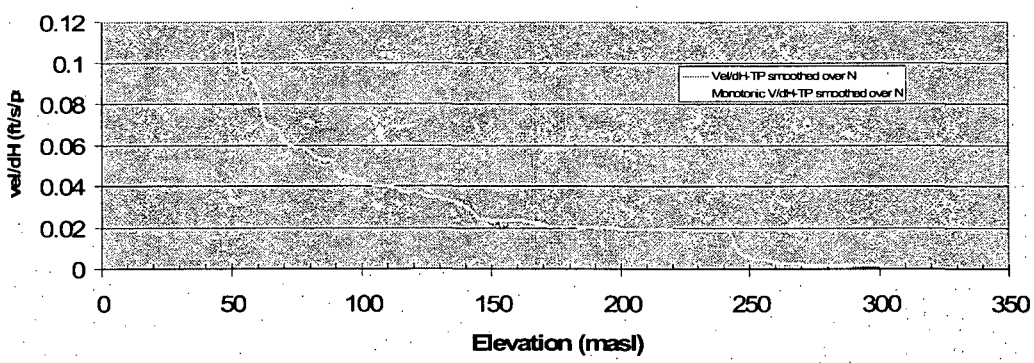
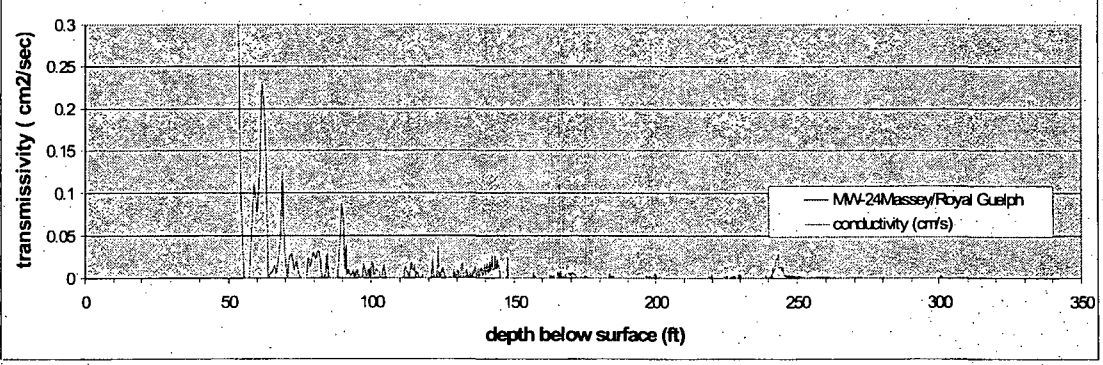


Fig. 4 Transmissivity calculated from velocity profile



Other interesting features of the velocity curve

As the liner descends and covers the flow paths, it should have a monotonically decreasing velocity. However, if the liner passes through an enlargement in the borehole, the liner will slow even though there may be no flow into the hole wall because the liner

cross section, Az, increases in the enlargement, Fig. 4. However, as the liner passes out of the enlargement, the liner will accelerate back to its original velocity, if there was no flow into the formation in the enlargement. If there was flow into the formation in the enlargement, the liner will not recover its velocity when it entered the enlargement. The net effect is to see a dip in the velocity and a subsequent rise in the velocity. The data reduction method fits a monotonic curve to the data which ignores all temporary drops of the liner velocity. The effect is to ignore an enlargement or to recognize only the total flow into the formation in the enlargement. That flow will show up in the velocity graph as a step change at the entrance into the enlargement even though the flow may have occurred anywhere within the enlargement.

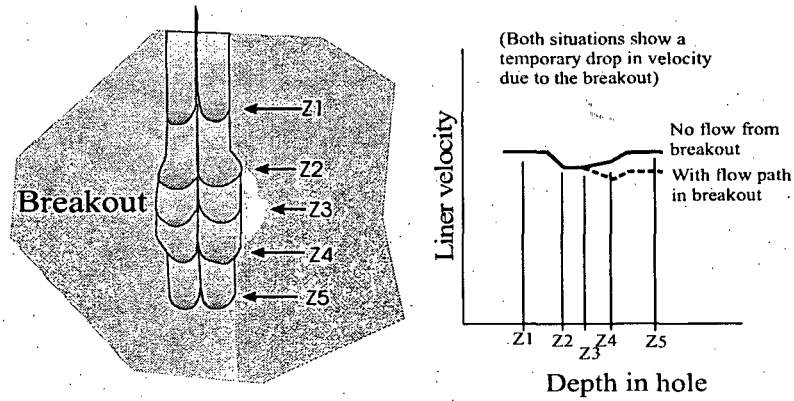


Fig. 4. Effect of liner moving thru breakout in borehole

In reality, there is some noise superimposed on the data due to the uncontrolled friction effects on the liner. The procedure and equipment have been adjusted to minimize any sources of drag, but the remaining drag tends to be primarily stick/slip kinds of drag such as that on the brake controlling the liner tension or for drag of the liner in the hole. Those stick/slip friction effects provide "noise" in the data record as oscillations about the actual velocity. This noise is most evident if the liner is weakly driven (e. g., a shallow water table) or the liner is moving very slowly. The data reduction procedure integrates/smooths the data to eliminate the noise superimposed on the velocity curve. The smoothing is done in a manner so as to not effect the location or magnitude of the significant changes in liner velocity.

If the liner is stopped for some reason, and then restarted, the liner will again accelerate and decelerate to the steady state velocity. That transient is easily recognized and eliminated from the data. However, if there is flow into the hole wall in the interval of that transient, the steady state velocity after the transient will be less than it was before the transient. That velocity change is indicative of flow path(s) in the interval masked by the transient, but the location of the flow path is only known to be in that interval, but not where in that interval. Such transients are seen when the end of the liner goes through the profiling machine and causes a "lurch" in the velocity. For slowly moving liners, the length of the transients is very short (<5 ft). Fortunately, any flow path is correctly measured for the transient interval, but may not be precisely located.

As the liner is first started down the borehole, it undergoes the same transient effect. In the data reduction, the end of the initial transient is estimated and the data through the

initial transient is cropped from the plot to prevent its being interpreted as a velocity drop due to flow into the hole wall. Unfortunately, if there are flow paths sealed during the transient, they are not measured. Ideally, the transient will have ended while the liner is still in the casing and no open hole open hole flow paths are missed. The transient probably includes useful information about the formation flow, but the analysis is currently limited to the nominal steady state flow which occurs after the transient.

Very rapidly moving liners in boreholes with very high conductivity will traverse more of the hole in the two second interval of the recording time steps. Hence the resolution is less. Fast moving liners are also likely to have more fluctuations due to liner tension variations. (Note, fast moving liners also usually have lower driving heads, and therefore the various friction sources are more influential on the velocity). For these reasons, the lower portion of the hole is often more finely resolved than the upper portion of the hole. The driving head in the borehole is corrected for the tension variations of the liner.

Brief Comparison with traditional methods

Comparison with straddle packer methods has led to several papers on the subject which are too detailed for this user's guide. However the conclusions are:

1. The FLUTe liner <u>does not</u> have any significant leakage terms in the method such as leakage past a packer either in the formation or via the packer interface with the hole wall. This is very important in highly fractured regions or rough walled holes.
2. The liner velocity is continuous in space and time and therefore does not have errors such as overlapping of straddle packer intervals or gaps between measured intervals. One hole volume is expelled, and the sum of the flow paths is the correct total for the borehole.
3. The liner velocity allows the resolution of discrete fractures and other individual flow paths. It is not limited to the straddled interval as for a packer.
4. The liner velocity measurement is typically performed in 5-15% of the normal time for discrete straddle packer measurements of a nominal 5 ft resolution over the entire hole.

Ask FLUTe for copies of those recent papers on comparison with straddle packers (some are in preparation for publication in a professional journal).

Comparisons with other geophysical methods show the direct flow measurement with the FLUTe liner to be superior to the indirect inferences of flow paths from most geophysical measurements . The nearest approach to the FLUTe technique is the pumped borehole vertical velocity log. That measurement is normally done at much lower flow rates (a few gpm) than the FLUTe measurements (5-50 gpm). The FLUTe measurements are performed with a much higher driving head than most velocity meter logs, so the effect of the local head profile on velocity is less for the FLUTe system.

A significant difference from all geophysical measurements is that once the FLUTe liner is installed in the borehole, it is usually left as a seal of the borehole.

Conclusion

The velocity versus depth for the everting liner is measured in a manner to obtain detailed information on flow path distribution in a borehole. The velocity plot allows a relatively easy location of the primary flow paths and provides the actual measurement of the flow rates in those flow paths. The technique has significant advantages over the traditional methods of location and measurement of flow paths in boreholes.

For assistance in the understanding of the FLUTe profile or if more detailed data is needed such as the digital data used for the plotted results, contact Sonny Casaus (sonny@flut.com) or Erik Storms (erik@flut.com) or FLUTe (888-333-2433)

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Installation procedure for NAPL blank liner in open borehole below the water table

This liner is a blank liner covered with a reactive covering. The liner is shipped inside-out with the cover on the inside of the liner. Care must be exercised to assure that the outer covering is not twisted with respect to the liner. This is especially true while the liner is inside-out on the reel.

1. Tag the hole depth to assure that it is known.
2. Tag the water level in the hole. Compare to the bubbler depth if a bubbler is included in the installation.
3. Smooth the top of the casing to assure that there are not very sharp edges that will threaten the liner.
4. Set the liner reel on an axle and supports to allow easy deployment of the liner. The liner is marked with an arrow. The arrow should point towards the well over the top of the reel. Unwrap the reel protective layer. Do not use any blades. A scissors without sharp edges is okay, but be careful to not cut the liner.
5. Insert the steel rod in the end of the vent tube. Tape the last 2 inches of the rod so that it is wedged firmly in the vent tube and will not fall out of the tube. Install the vent tube to the bottom of the hole. Tie the top end of the vent tube so that it is suspended about 4 inchs off the bottom of the borehole.
6. Pull off about 10 ft. of liner and evert the liner back upon itself about 5 ft. as in the drawing. Evert the covering over the everted liner if the liner is not stapled to the cover. Be careful that the covering is not twisted relative to the liner. The cover may have been stapled to the liner to assure that it is not twisted.
7. Slide the everted liner down into the casing as shown.
8. Slit the open end of the liner/cover about 6 inches and fold about 3 inches of the liner and cover over the top edge of the casing. If the casing is large, make two slits on opposite sides. Tape the cover to the top of the casing. Put two clamps over the tape to hold the liner and cover securely to the casing. The vent tube can pass through the slit.
9. Set the wellhead roller over the casing. Drape the inverted liner over the wellhead roller to guide the liner into the hole.

10. Water can now be added to the interior of the liner. Lower the liner until about 10 ft deep in the casing. Fill the liner to the top of the casing, if the wt. is not too great.
11. Stop the water addition, and lower the liner to the water table. See the footage marks for that distance (if footage marks are on the liner). The hanging wt. of the liner should decrease dramatically as the liner descends into the water.
12. Keeping about 10 lb. of tension on the liner (i.e., hold it back from descending), continue to add water to the liner until it reaches the bottom of the hole. At about the half distance into the hole (i.e., the eversion point is at the half depth.), the liner will stop until the air has bled out of the end of the liner via the press. relief valve. Add another 10 gallons of water to submerge the air bubble to hasten the air vent. When the liner starts to move again, continue adding water until the liner reaches the bottom of the hole. *This venting of the air is only needed at the end of the liner*. If the liner is longer than the hole, the trapped air will be in the liner between the casing and the reel.
13. Be very careful to not let the tether go down the hole. Secure it to the wellhead roller frame when the liner is all the way down the hole. If there is no tether because of the liner length, tape and tie the liner so that it does not continue down the hole. The liner should be sitting on the bottom of the hole.
14. Leave about 10 ft. of excess water head in the hole. More is not a concern. This will press the liner/cover firmly against the hole wall.
15. Let the liner sit in the hole until the retrieval is to be done. This can be done after ~1/2-1 hr. if desired. -or longer to allow a good absorption from fractures.

The retrieval

1. Untie the tether(or liner) and extend it from the wellhead over the roller. Locate the winch plate so that the tether/liner can be pulled from the well over the wellhead roller.
2. Lower a Grundfos or similar pump into the liner to remove the water. Do not lower the pump below a point 5 ft or more above the original water level in the hole. Pump the water from the liner until the pump runs dry. Immediately stop the pump. Never allow the water level inside the liner to be lower than 5 ft above the water table. A somewhat higher level is

better insurance that the liner will not buckle in the hole and become jammed.

3. Winch the tether from the hole. This will go slowly for a time, but then it will be easier. This will take about 30 lb. of tension on the tether at the least. (note at about 150 lb. of tension on the winch, the crank is very hard to operate. Any additional tension is that needed to pull the water into the well.) The crank should be relatively easy to turn. Note, if the liner, and not the tether, is being pulled from the well, it may be easier to pull it out by hand without the winch, using several people to pull on the liner.
4. Wind the tether, and later the liner, on the reel as it is pulled from the hole.
5. Adjust the pump speed to keep the water level a short distance above the pump. This will reduce the winching effort.
6. When the liner knot and valve appears at the surface (if the tether was being used to pull the liner), the liner may be able to be pulled from the well by several people over the roller.
7. When the liner reaches the depth of the water table, the pump can be removed from the liner. Do not pump out the last 5 ft of water in the liner.
8. Now the liner can be pulled from the hole without further pumping until the water begins to spill out the top of the hole. Lower the pump into the liner and remove the remaining water.
9. Pull the liner out of the hole.

Examination of the liner.

1. Remove the clamps from the well head, and wrap the liner on the reel.
2. Pull the liner out on a relatively clean surface. If the liner will not be reused, it does not need abrasion protection. Measure the distance from the open end of the liner to the hole depth plus 3 ft on the liner. Cut the liner off at this point, including the cover inside the inverted liner. (Use a stapler to staple the cover perimeter, inside the unused liner, to the liner. This will prevent the cover from becoming twisted inside the unused liner.) The end of the liner cut off is the section of the liner that was exposed to the fluid in the hole. Slide the liner piece off of the cover to expose the white backside of the cover. If the liner is very long, the liner may need to removed by bunching the liner, pulling the cover out,

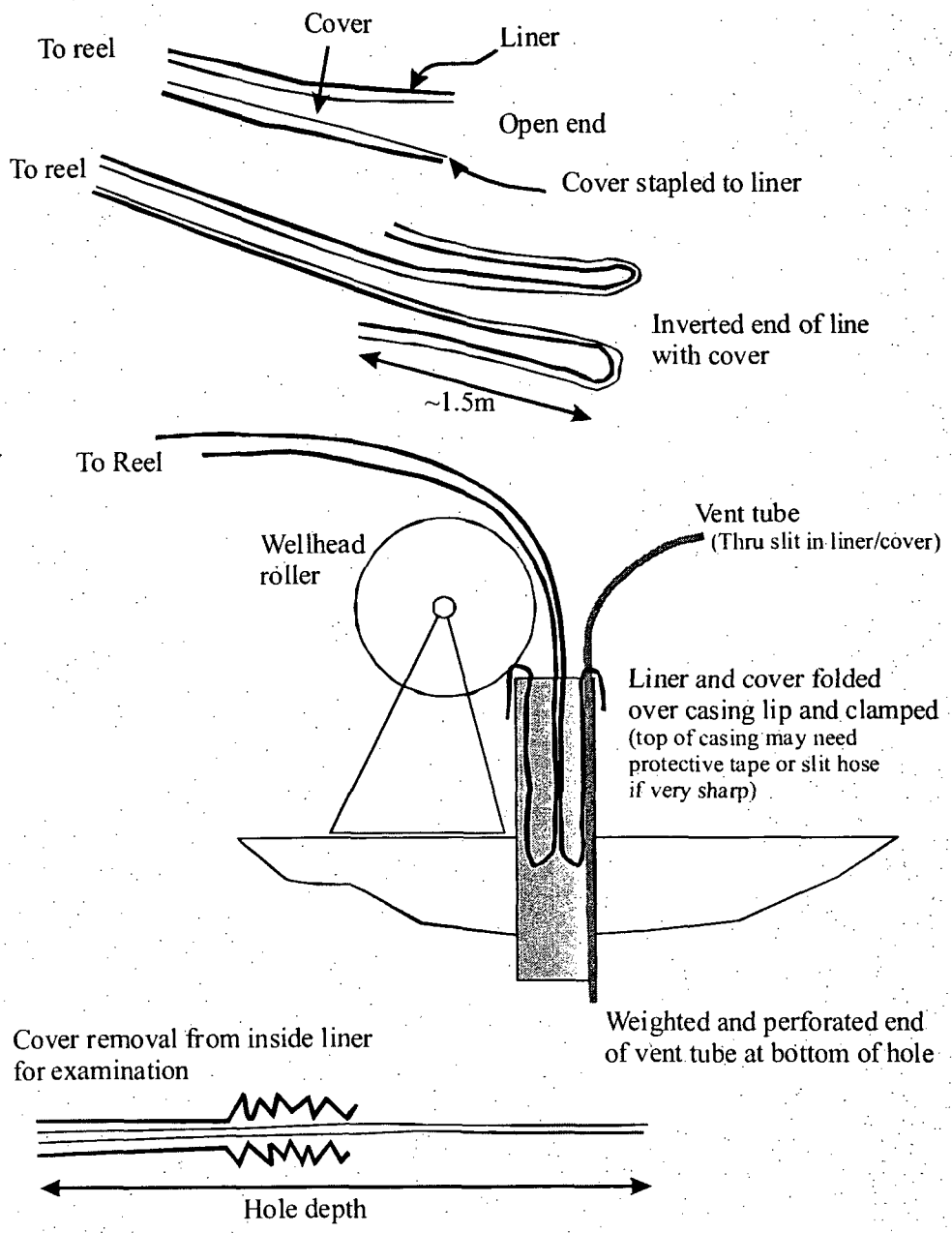
bunching the liner, etc… until the cover is removed. (the wet cover adheres a great deal to the wet liner, so sliding a portion of the liner off the cover is easier.) Spread the cover flat to allow easy examination of the white (unstriped) surface. Turn it over to examine the other side.

3. Lay a tape measure the entire length of the cover with the top at the ground surface position.
4. Record and photograph the location of any stains on the cover caused by the wicking of the dye through to the white side of the covering. The stains will be obvious. They will not be the faint variations of the dye lines that will be seen through the cover. If in doubt, expose a small piece of the cover to the pure product under water. For oily substances (non-solvents), the stains will be clear and translucent, like an oil drop on paper. If the NAPL is dark, the stains on the cover will also be dark.

If you have any questions, call FLUTe at 888-333-2433. Or call Carl Keller on his mobile phone 505-930-1154.

Note: If the liner is being used piecemeal (i.e., the liner is longer than twice the hole depth) the tether will not go into the hole until the last portion of the liner is being used. In that case, the liner will be pulled from the hole by the excess liner rather than by the tether. Air trapped in the liner is normally vented by a check valve at the end of the liner. If the end of the liner is not in the hole, the check valve will not vent the trapped air. The air should not be allowed to dilate the liner in the hole so as to prevent the liner descent. If the reel is moved further from the hole, the air will reside in the liner between the wellhead and the reel. Each time the liner is rolled back onto the reel, try to force the air to the open end of the liner, so as to reduce the trapped air the next time the liner is installed.

NAPL FLUTe liner installation in open borehole



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Use of Pressure Transducers with Water FLUTe System

Use of Pressure Transducers with Water FLUTe System

Purpose
This guide explains where the pressure transducer is located in the Water FLUTe system and how the formation head is measured at each port both manually and with the pressure transducer. The manual measurement is required to determine the precise depth of the pressure transducer in the borehole. Given the depth of the transducer, all future head measurements can be determined from the transducer measurements.

Geometry
The Water FLUTe multi level sampling system is shown in Fig. 1. The formation water flows from the formation into the spacer interstitial space and via the annular spacer into the port. The water is conducted from the port to the bottom of the hole and upward into the pump tube volume through the first check valve. The water from the port fills the pump tube to the level of the natural head in the formation.

When pressure transducers are part of the design, the pressure transducer is located just below the first check valve on the port to pump tube (Fig 2.). The pressure transducer measures the water pressure in the tube at the elevation of the transducer diaphragm shown in Fig. 2.

The water from the port also fills the sample tube though the second check valve. If the system has been pumped, the sample tube may also be filled to the surface.

Calibration of the pressure transducer
Since the pressure transducer measures the head in the port-to-pump tube which is open to the formation, the pressure measured is the head in the formation at the depth of the transducer diaphragm (Fig 2). If one knows the depth of the transducer below the ground surface, the water table depth below the surface can be calculated directly for that port (WT bgs = depth transducer - head measured by transducer).

Another simple means of measuring the water table depth is to tag the depth of the water in the pump tube (Fig. 2) if the water table is less than 200 ft. (For deeper water tables, contact FLUTe for a special tag line.) If one has measured the depth to the water in the pump tube, he can calculate the depth to the transducer (depth to transducer = WT Depth bgs+ head measured by transducer). Since the actual depth of the transducer is not precisely known when the liner is emplaced due to a variety of factors including the actual depth of the hole, the depth of the transducer is best determined from this manual measurement of the water table in the pump tube. The first check valve has a Teflon ball so as to minimize any perturbation to the equilibration of the water in the pump tube at the actual water table for that port.

However, because of the fact that the check valve will prevent the water flow out of the pump tube to the port, the water level in the pump tube can not follow a dropping water table in time. In order to obtain the correct current water table, the entire contents of the pump tube and sampling tube must be expelled to allow the pump tube to fill to the

current water table level. Therefore, a gas pressure source should be used with the normal water sampling valves to expel all of the water from the system just as described for the purge procedure in the water sampling procedure (Fig. 3). The water sampling procedure is available from FLUTe.

It is important to remember that the pressure transducer is connected directly to the formation and is not measuring through any check valves. Therefore, the transducer follows the head changes in the formation. As the pump refills, the transducer will follow the last portion of the rising head in the pump when the flow velocity of the water in the pump is very low.

Procedure for calibration of the pressure transducer

To obtain the depth of the transducer in the hole, purge the pump of all water and let it equilibrate to a new water level equal to the water table in the formation. Measure the depth of the water table in the pump tube. This is the depth of the water table in the formation for that port (it may not be the water table measured in the open hole). Add the head measured in the pump tube to the head measured by the transducer. The sum is the depth of the transducer below the surface. For all future head measurements in the formation (water table depths below the surface), subtract the transducer head measurement from the transducer depth. This same procedure is used for each port. This will provide the depth to the water table for each port elevation in the formation. For absolute elevations, convert the depths below the surface to elevation values.

Once the depth of each transducer has been determined, it does not need to be remeasured. However, with time the transducer calibration may drift. The manual measurement of the water table allows the transducer effective depth to be determined as often as desired.

FLUTe provides pressure transducers for specified ports as a service to our customers for the retail price of the transducer and cable with appropriate fittings. The transducer calibration information is also provided by FLUTe as received from the manufacturer. The warranty of the transducer and associated hardware is the responsibility of the manufacturer. For information on the transducers, please contact the manufacturer.

For any questions about this procedure, call 888-33-32433.

Fig. 1 Water FLUTe Port and Pump System
(Single port system shown for clarity)

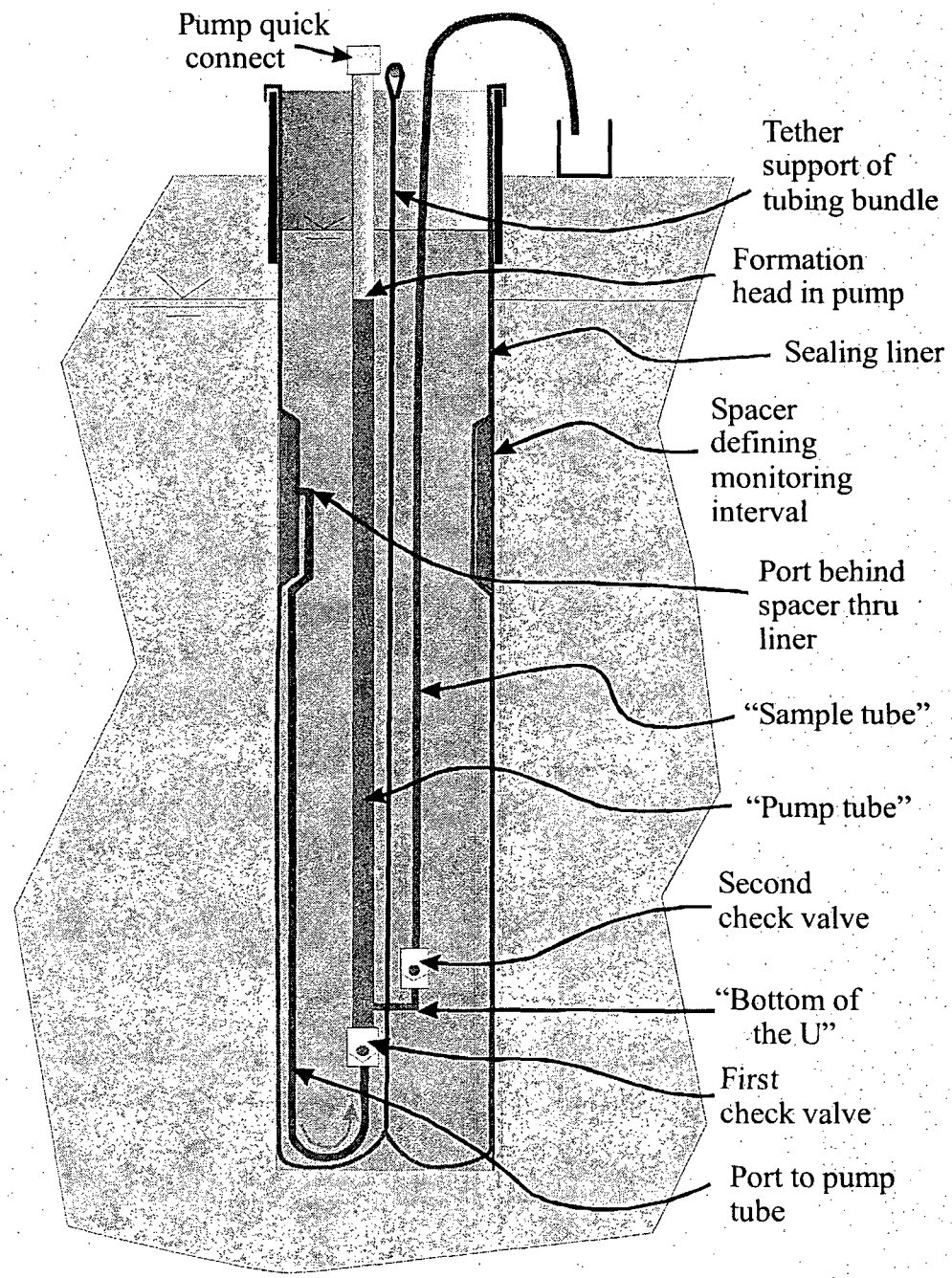


Fig. 2 Water FLUTe port/pump with Transducer

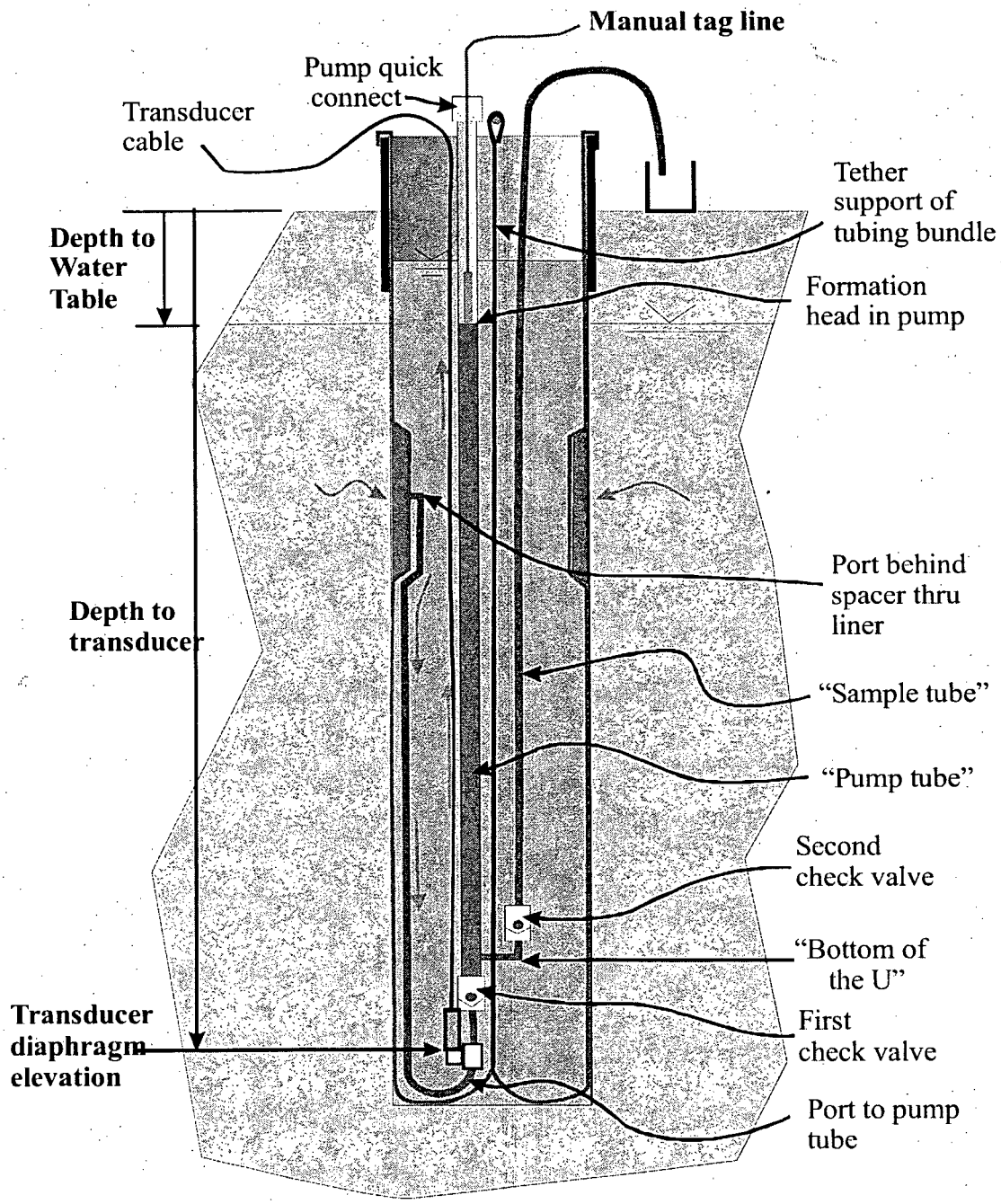
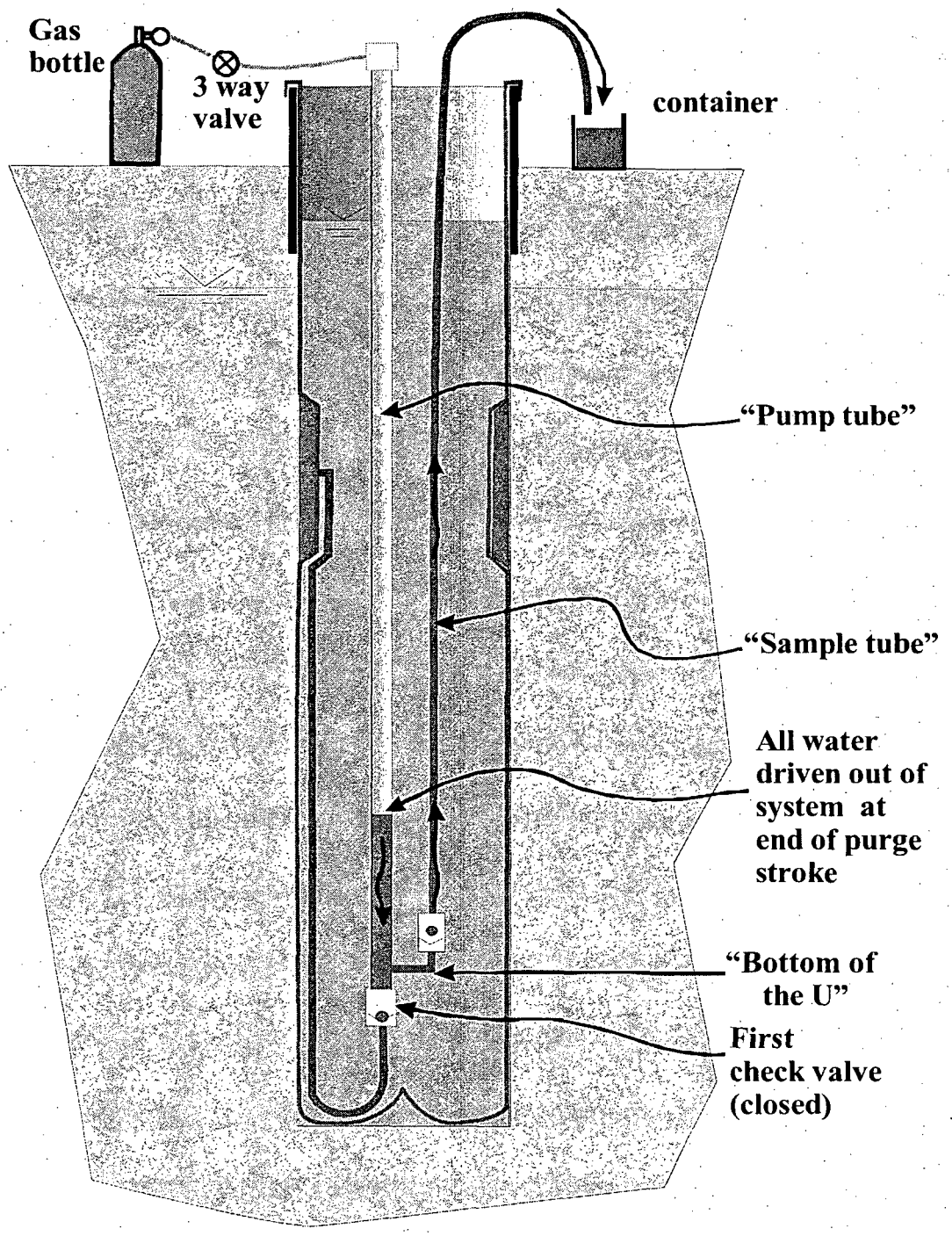


Fig. 3. Purge Pumping Procedure



FLUTe™ Flexible Liner Underground Technologies, Ltd. L.C.
For a medley of innovative designs
6 Easy St., Santa Fe, NM 87504
505-455-1300, www.flut.com

Sampling guidelines for *Water FLUTe* systems

Rev. April, 2007

Water level in the liner.

The liner water level should be 10 ft above the highest formation water level to provide a good seal of the liner in the hole (5 ft minimum excess head). The formation water level can be measured via the "pump tube" for each port. The water level inside the liner should be tagged in the ½" id tube labeled "TAG" adjacent to the sampling tubes. If the water level inside the liner is measured in the liner, outside the Tag Tube, lower the weighted tag line very slowly to avoid damage to the liner. Water can be added to the liner by simply pouring water into the liner or through the TAG tube, whichever is easier. Do not fill the liner more than 10 ft above the highest formation water level. The water level in the liner should be checked prior to each sampling episode. (Beware that filling the liner with de-ionized water can give a false water level reading.)

Water flow

The water flow into the pumping system is shown in Fig. 1. Water flows from the formation through the spacer pore space, through the port tube, through the first check valve, and fills the "pump tube". The "sample tube" is also filled at the same time. The water level rises in the pump tube to the water table for that port.

Setting up the gas pressure source

The water is pumped with gas pressure. The FLUTe pump design is such that there is very low risk of aeration of the sample. The gas source is usually a nitrogen bottle with a regulator for setting the prescribed driving pressure. The arrangement of the FLUTe gas drive system is shown in Fig. 2. The regulator is set to the proper gas pressure defined later by closing the three way valve to prevent gas flow out of the quick connect fitting. The pressure gauge on the FLUTe pump driver is much more sensitive than the regulator for setting the regulator pressure. The FLUTe pump driver must be securely connected to the regulator at the normal ¼" NPT connection on the regulator outlet.

The regulator is attached to the top fitting on the gas bottle (a special nitrogen regulator fitting connects to a nitrogen bottle). Turn the pressure regulator handle counter-clockwise until is moves freely (the no pressure position). Rotate the main valve on the regulator (nearer the bottle) clockwise to fully closed. Open the valve on the bottle (counter clockwise). The main bottle pressure gauge on the regulator will rise to the bottle pressure. Close the regulator valve (clockwise) until the pressure starts to rise on the pressure gauge on the FLUTe pump driver (three way valve closed with no flow out of the quick connect). Adjust the regulator to the desired pressure for purging, provided by FLUTe. Connect the quick connect to the top fitting of the pump tube (see Fig. 2). Open the three way valve to drive the water out of the pump.

Purging

Water is pumped from the tubing by applying the gas pressure to the interface at the static water level in the pump tube (Fig. 1 and 2). The water is driven down in the pump tube and up through the second check valve to the surface via the sample tube. By driving the water with a sufficient gas pressure (the "recommended purge pressure") to drive all of the water in the pump tube and the sample tube to the surface, the water in the pump tubing is nearly all expelled. The purge stroke is complete when gas is expelled from the sample tube following the water flow. The pressure in the system must then be vented (i.e., dropped to atmospheric by turning the three way valve to the vent position), to allow the pump tube to refill by flow via the port tube. The recharge flow from the port tube consists of the port tube water, the water in the pore space of the spacer, and water from the medium. Because of the relatively large volume in the pump tube, most of the recharge is from the medium. The recharge will take about as long as the first purge stroke. However, a low conductivity medium will require more time.

Purging the pump tube a second time will remove any of the water that has resided in the spacer and port tube volume. That is highly recommended, since the water resident in the tubing and spacer is probably not typical of the formation water. If the refill has been prompt, the second purge water volume will be similar to the first stroke. If in doubt, or if in a sedimentary formation or screened well, a third purge stroke is recommended to remove water that may have been in long contact with the liner or spacer.

Sampling

The sampling flow is best driven on the third (or fourth) cycle by a "recommended sampling pressure" which is less than that needed to drive gas through the bottom of the pump tube. The pressure recommended is that which will drive the water to near, but not out of, the bottom of the large tube. That recommended pressure, "the sampling pressure," is calculated in the spreadsheet provided with each system. The pressure regulator is set to the sample pressure, which is lower than the purge pressure. Opening the three way valve will now apply the sample pressure to the system causing flow from the sample tube.

The first flow of the sampling cycle sweeps along droplets of water left in the tubing from the purge cycle. That residual water is depleted of volatile components. Tests have shown that the first tube volume of the sample flow should be discarded as depleted in volatiles (the "discard volume" is also calculated in the spreadsheet). Thereafter, the samples can be collected from the sample tube outflow. The volume to be discarded is shown in the spreadsheet as "discard volume". The sample tube water flow rate will start fast, then slow, and finally stop. That occurs as the water column being driven approaches the applied pressure/head. The typical sampling pressure drives to within 25 ft. of the bottom of the pump tube (the U). The large buffer zone remaining in the pump tube assures against aeration of the sample.

This procedure should provide an ample sample of good quality drawn directly from the formation.

Caution: If the pumping system refills very slowly, there may not be sufficient water in the pump to fill the "sample tube" to the surface when the stroke is performed. In that case, there will be spitting of gas from the sample water and it will be followed by a flow of gas only. The sample water should never show "spitting" and the stroke should never end with gas flow from the sample tube. The proper sample flow will slow until it stops flowing. Should this evidence of insufficient recharge be observed, allow the pump to refill for a longer time and repeat the sample stroke. One can

tag the water level in the large tube, as described in the head measurement procedure, to assure that the pumping system has been sufficient refilled.

Measuring the head in the system
The water level in the large tubes may not be the current water level. After sampling, if there is any leakage of the second check valve (sand in the tube, etc...) the water in the sample tube can backflow into the larger tube, adding to the water that fills the large tube during the recharge. Also, if the water level in the formation is dropping between head measurements, the water level in the pump tube will not follow the descent if the first check valve is a good seal. For these two reasons, and for the freezing concern below, it is best to finish the sampling stroke by raising the pressure to the "purge pressure" value to purge the pumping system of all water. Then upon refilling, the level is the current head for each port. If head measurements are made between sampling events, each port's pumping system should be first be purged to allow the tubing to refill to the current head value.

If the water might freeze in the sampling tubing near the surface, purge the entire volume of water from each sampling line, after sampling, before leaving it. Use the recommended purge pressure to remove all water, not the sampling pressure. **Each line should be blowing gas when the purge is complete.** If the lines were purged after sampling for head measurements, that is sufficient.

If the Water FLUTe uses PVDF tubing, the purge of the entire system after sampling should not be neglected, even if head measurements are not to be made. This removes the water column in the sampling tube. For deep water tables, the long term pressure of the standing water in the sampling tube might lead to excessive creep of the tubing which is susceptible to "cold flow", a characteristic of Teflon like materials. (This is not a concern except for very deep water tables (>300 ft).

In most cases, the performance of a final purge of the system after sampling is useful, even if not essential.

Simultaneous purge and sampling of all tubes
The FLUTe pumping system for each port is essentially identical in length, pump volume and elevation in the hole. This allows all ports to be purged and sampled simultaneously for a great saving in sampling time. The only

difference for simultaneous sampling is that the pressure source must include a tube to each port fitting at the wellhead. FLUTe offers a manifold pump driver system at extra cost (the single port driver is provided with the Water FLUTe). The recommended purge and sample pressures are the same as used for single port sampling.

In some cases, the buoyancy of the sampling system is so great when emptied of water during the simultaneous purge that the tubing bundle can cause the liner to invert. The sampling volume spreadsheet provided with the liner notes whether the system can be purged simultaneously. This is only a problem for smaller hole diameters, many ports, and a small excess head in the liner. However, increasing the excess head in the liner to overcome the buoyancy of the tubing can be a hazard to the liner.

A short summary is provided as the following checklist:

Check List

1. Check/restore the water level in the liner.
2. Connect the gas driver source to the gas drive tube for the port.
3. Set the regulator to the recommended purge pressure.
4. Expel the tube water at the suggested purge pressure. Collect the purged water volume for verification of a good purge. Note the water flow time of the purge stroke.
5. Allow the tubing to refill. Repeat the purge. Collect the purge volume to assure the amount removed is at least the "port tube volume". Was the refill long enough?
6. Purge a third time, if desired.
7. Allow the tubing to refill for the sample stroke.
8. Reduce the driving pressure to the "sampling pressure". Apply the pressure and collect the first flow to measure the discard volume. Discard that water.
9. Reduce the pressure, if needed, to slow the flow and collect the samples.
10. Perform a final purge of the water out of the sampling lines by raising the driving pressure to the purge pressure value.

11. When the sampling system has refilled, tag the water level, if desired, for the current water table. If a port system is refilling very slowly, tag it at a later time.

See the spreadsheet provided with each *Water FLUTe* for the recommended purge and sampling pressures. Those are the pressures that can be used for a simultaneous purge of the several ports, but be sure that the buoyancy of the tubing will not lift the tubing, and the wellhead. The spreadsheet flags the condition where all ports should not be purged simultaneously. In most cases, several, to all, of the ports can be purged simultaneously.

Optimum sampling procedure:
Since it is often desirable to minimize the amount of time that the sample water resides in the pumping tubing, it is useful to note the actual time that is required for the recharge of the system. Since the fill rate slows dramatically for the last portion of the recharge, it is not necessary to wait for a complete refill. For most formations, the recharge is dominated by the tubing pressure drop. In that case, the time required for the purge stroke to be completed is about the same time required for the refill. (The exception is for a tight formation that recharges the tubing very slowly.) Hence the second purge can be started after waiting the same length of time as the first purge endured. If the second purge is of a similar volume (usually somewhat less) than the first purge volume, the refill time was long enough. After the same delay, the sampling stroke can be initiated. This timing of the strokes allows one to reduce the retention time in the pumping system. For very large sample volumes produced, the refill time can be shortened even more, as long as the sample volume is adequate after the discard of the first flow.

In some situations, the retention time is still too long. FLUTe can often increase the sample tube and port tube diameters for greater flow rates. However, the standard design is well matched for to a wide range of hole diameters, depths, and water table elevations. For very deep wells, the tubing may need to be of higher pressure capacity for the required driving pressures. For water table depths below 700 ft., this may be a concern. FLUTe initiated a design change from Nylon 11 to PVDF tubing in the Water FLUTe systems in 2002 to avoid any concern about tubing interaction with the sample water. However, the prescribed purge is sufficient for the use of Nylon tubing systems.

Questions: Call 888-333-2433 and ask for Carl Keller, or a field engineer.

Figure 1. Water FLUTe pump system

(Single port system shown for clarity)

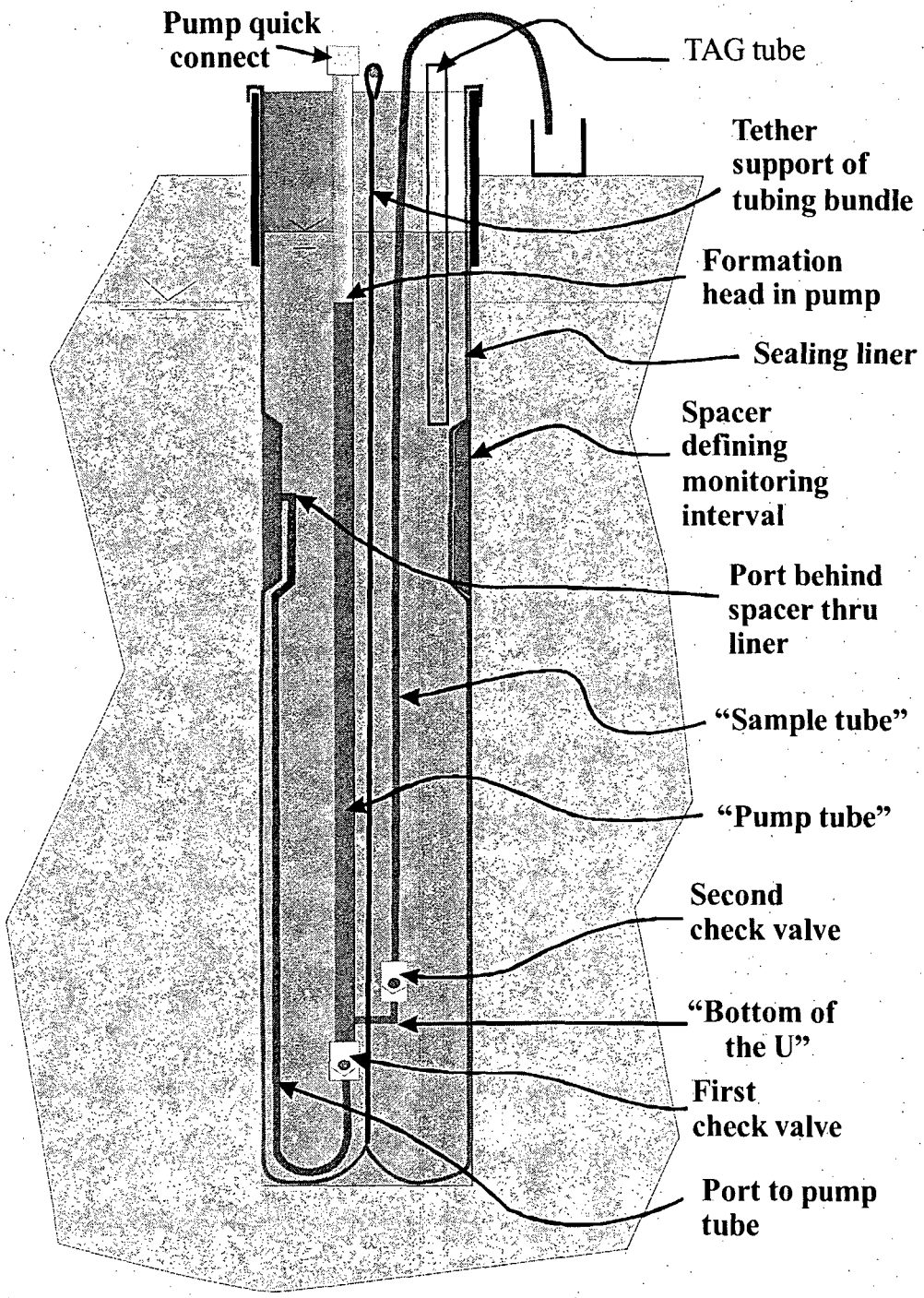
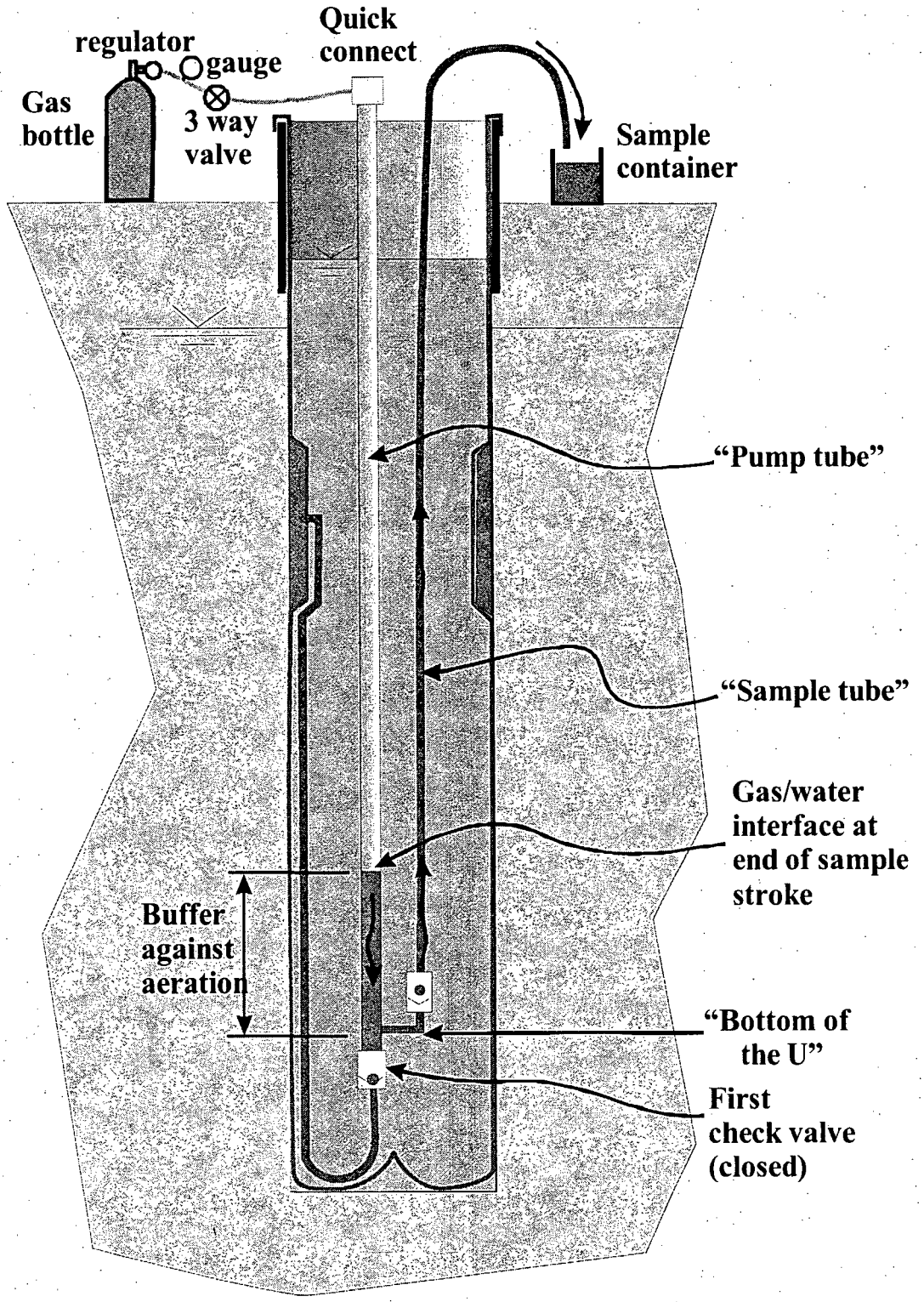


Fig. 2. Pumping Procedure



APPENDIX A-B
WELL ASSESSMENT CHECKLIST
ROCK CORE AND CONVENTIONAL DRILLING LOGS
STANDARD OPERATING PROCEDURES

EPA Region 2 Superfund Well Assessment Checklist

Facility Information

Site Name: _____
Site Address: _____
Site County: _____
Site State: _____
EPA Site ID Number: _____
Site Owner: _____
EPA Project Manager: _____

Well Locational Information

State Well ID: _____
Well Tag ID: _____
Well Installation date: _____
Latitude (by GPS): _____
Longitude (by GPS): _____
Ground surface elevation (by GPS): _____
GPS Instrument used: _____
Cross streets (if applicable): _____

Well Construction Details

Type of well (Circle one) Flush Mount Stick up Multilevel Well
Well casing material: _____
Surface casing material: _____
Measuring point\elevation: _____
Elevation (top of inner casing): _____
Well lock\security type: _____
Well Depth (as installed): _____ *ftbgs*
Well Depth (as measured): _____ *ftbgs*
Well Diameter: _____ *inches*
Surface Casing diameter: _____ *inches*
Screened interval: _____ *ft*
Open hole interval: _____ *ft*
Depth to water: _____ *ftbtoc*

EPA Region 2 Superfund Well Assessment Checklist

Well Headspace Readings

PID/FID Reading taken inside top of casing: _____ *ppm*

Do site conditions necessitate other headspace measurements?	Yes	No

If yes, list other measurements taken: _____

Do readings indicate unsafe conditions exist?	Yes	No

Well Condition

Is the concrete pad in good condition?	Yes	No
Is the well surface casing in good condition?	Yes	No
Is the surface casing vertical?	Yes	No
Is there an internal well seal?	Yes	No
Has there been physical damage to the well?	Yes	No
Does sounding depth match completed depth?	Yes	No
Is measuring point marked?	Yes	No
Is the well clearly labeled?	Yes	No
Flush mount - Is it secure from runoff?	Yes	No

Other Comments _____

_____

_____

Recommendations

Well needs to be redeveloped	Yes	No
Well needs to be re-surveyed.	Yes	No
Well needs to be repaired.	Yes	No
Well needs to be replaced.	Yes	No
Well needs to be properly abandoned.	Yes	No
No action necessary.	Yes	No

Inspected by: _____

Date of Inspection: _____

MALCOLM PIRNIE, INC.

17-17 Route 208 North, Fair Lawn, NJ 07410

BORING:
MW-

PROJECT NAME: Cornell-Dubilier Electronics Superfund Site. DATE:
JOB NUMBER: 4553058 LOCATION:
DRILLING FIRM: WEATHER:
DRILLING METHOD: HQ (2.5-inch, 6.35 cm) Triple barrel core. Vertical. ELEVATION:
DRILLER: DATUM: Ground Surface
HELPER: HYDROGEOLOGIST:

SAMPLES		Quality		ROCK CORE						Coring Start at ft bgs.
Sample Number	Parameters	Total Recovery, %	RQD, %	Fracture Sketch	Core Condition	Discontinuity Spacing	Depth (ft)	Discontinuity Description. Specifically state which discontinuities are oriented relative to dip direction of bedding.	Run #, Depth Range,	Rock Mass Description

Total Recovery = (Core length/core run)*100. RQD = (sum lengths >= 4-inch)/core run)*100. Discontinuity spacing = (No. fracts/ft). Discontinuity description = type, depth & dip angle.
NI = not intact/broken zone = core pieces <2.5-inch.

MALCOLM PIRNIE, INC.

17-17 Route 208 North, Fair Lawn, NJ 07410

BORING:

MW-

PROJECT NAME: Cornell-Dubilier Electronics Superfund Site.	DATE:
JOB NUMBER: 4553058	LOCATION:
DRILLING FIRM:	WEATHER:
DRILLING METHOD:	ELEVATION:
DRILLER:	DATUM: Ground Surface
HELPER:	HYDROGEOLOGIST:

SAMPLE INFORMATION										
No.	Depth	Rec	Blows per 6"				Depth	SOIL DESCRIPTION	SUMMARY	REMARKS
	0						0 - 2			
	2						2 - 4			
	4						4 - 6			
	6						6 - 8			
	8						8 - 10			
	10						10 - 12			
	12						12 - 14			
	14						14 - 16			
	16						16 - 18			
	18						18 - 20			
	20						20 - 22			
	22						22 - 24			
	24						24 - 26			
	26						26 - 28			
	28									

Procedure for Calibration and Operation of a Photoionization Detector

I. Introduction

This procedure describes the equipment and methods to be used to calibrate and operate a Photoionization Detector (PID).

II. Equipment and Supplies

The following equipment will be needed:

1. PID (HNU or similar model)
2. Operating manual
3. Probes: 9.5 eV, 10.2 eV, or 11.7 eV
4. Battery charger for PID
5. Spare batteries
6. Tygon tubing
7. Traceable calibration gas
8. Isobutylene standards for calibration
9. Benzene reference standard
10. "T" valve for calibration
11. Field Data Sheets/Site Logbook
12. Intake assembly extension
13. Strap for carrying PID
14. Teflon tubing for downhole measurements
15. Plastic bags for protecting the PID from moisture and dirt

III. Guidelines

Start-up/Calibration:

1. Allow the temperature of the unit to equilibrate to its surrounding. This should take about five minutes.
2. Attach the probe to the read-out unit. Match the alignment key, then twist the connector clockwise until a distinct locking is felt.
3. Make sure the microswitch (red button) is depressed by the locking ring.
4. Turn the FUNCTION switch to the battery check position. Check to ensure that the indicator reads within or beyond the green battery arc on the scale plate. If the indicator is below the green arc, or if the red LED comes on, the battery must be charged prior to use.
5. To zero the instrument, turn the FUNCTION switch to the STANDBY position and rotate the ZERO POTENTIOMETER until the meter reads zero. Wait 15-20 seconds to ensure that the zero adjustment is stable; if not, then readjust.
6. Check to see that the SPAN POTENTIOMETER is set at the appropriate setting for the probe being used (i.e., 9.8 for the 10.2 eV probe, 5.0 for the 11.7 eV probe, 1 for the 9.5 eV probe. Note: The setting may vary based on the intensity of the light source).
7. Set the FUNCTION switch to the desired range (i.e., 0-20, 0-200, 0-2000).
8. Listen for the fan operation to verify fan function.
9. Look for ultraviolet light source in the probe to verify function. Do not look at light.
10. Record the following information in the site logbook: the instrument ID number (U.S. EPA decal or serial umber if the instrument is a rental), the initial and final span settings, the date and time, the source and lot number of the calibration gas, concentration and type of calibration gas used, and the name of the person who field calibrated the instrument.
11. Check instrument with an organic point such as a magic marker, prior to survey to verify instrument function.
12. Under no circumstances is work to be done without a proper functioning instrument.

13. Set the FUNCTION switch to the range setting which includes the concentration of the calibration gas.
14. Attach a regulator to a disposable cylinder of calibration gas. Connect the regulator to the probe of the HNU with a piece of clean tygon tubing. Open the valve on the regulator.
15. After 15 seconds, the meter reading should equal the response value as indicated on the calibration gas cylinder used. If the reading is within ±15% of the response value, then the instrument can be field calibrated to the response value using the external SPAN ADJUSTMENT control. The SPAN ADJUSTMENT control should be adjusted to a lower setting until the correct reading has been obtained. The lower the number on the SPAN ADJUSTMENT control, the greater the instrument sensitivity. If the SPAN ADJUSTMENT control has to be adjusted below a setting of 4.00, the unit should be red-tagged and returned for repairs. 5. If the meter reading is greater than ±15% of the response value of the calibration gas used, then the instrument should be red-tagged and returned for re-calibration.
16. If the PID does not start up, check out, or calibrate properly, the instrument should not be used. Under no circumstances is work requiring air monitoring with a PID to be done without a proper functioning instrument.
17. In some field applications, with the exception of the probe's inlet and exhaust, the PID should be wrapped in clear plastic to prevent it from becoming contaminated and to prevent water from getting inside in the event of precipitation.

Operation:

1. All readings are to be recorded in the site logbook. Readings should be recorded, following background readings, as ppm.
2. As with any field instrument, accurate results depend on the operator being completely familiar with the operator's manual. The instructions in the operating manual should be followed explicitly in order to obtain accurate results.
3. Position the probe assembly close to the area to be monitored because the low sampling rate allows for only very localized readings. Under no circumstances should the probe tip assembly be immersed in fluid. While taking care to prevent the PID from being exposed to excessive moisture, dirt, or contamination, monitor the work activity as specified in the site Health and Safety Plan.
4. The PID survey should be conducted at a slow to moderate rate of speed and the intake assembly (the probe) slowly swept from side to side. There is a three to five second delay in read-out depending upon the instruments sensitivity to the contaminant.
5. During drilling activities, PID monitoring is performed at regular intervals downhole, at the headspace, and in the breathing zone. In addition, where elevated organic vapor levels are encountered, monitoring may be performed in the breathing zone during actual drilling. When the activity being monitored is other than drilling, readings should emphasize breathing zone conditions.
6. When the activity is completed or at the end of the day, carefully clean the outside of the PID with a damp disposable towel to remove any visible dirt.

IV. References

Title: Decontamination of Sampling Equipment

I. Introduction

This procedure describes the methods used to decontaminate rock and groundwater sampling equipment and sample processing tools for the Cornell-Dubilier Project. The procedures specifically address equipment used to collect rock and groundwater samples.

II. Definitions

PPE - Personal Protective Equipment

III. Equipment and Supplies

A. Groundwater Sampling

The following equipment will be used to decontaminate equipment and tools used to collect water samples:

1. Tap water for initial cleaning and rinsing of equipment.
2. Analyte free water for final rinsing of equipment after tap water or solvent rinse.
3. Non-phosphate detergent (*e.g.*, Alconox™) for cleaning equipment.
4. Dishwashing detergent (*e.g.*, Joy™ which provides suds in seawater) to remove oily or organic residue.
5. Nitric acid as a 1% solution for removing metal contaminants from equipment
6. Isopropyl alcohol
7. Organic solvent for final cleaning of equipment (*e.g.*, acetone or equivalent)
8. Personnel protective equipment (PPE) - including disposable gloves (Nitrile preferred), disposable wipes, eye wash system, first aid kit, and waterproof outerwear (if necessary).
9. Re-sealable buckets approved for waste collection and transportation.
10. Squirt bottles for water, alcohol, and solvents.
11. Brushes for cleaning equipment.
12. Field notebooks, pens, pencils, and digital camera to document decontamination procedures.

B. Rock Sampling

The following equipment will be used to decontaminate equipment and tools used to collect rock samples:

1. High pressure steam-cleaner.
2. Tap water for initial cleaning and rinsing of equipment.
3. Analyte free water for final rinsing of equipment after tap water or solvent rinse.
4. Non-phosphate detergent (*e.g.* Alconox™) for cleaning equipment.
5. Dishwashing detergent (*e.g.* Joy™ which provides suds in seawater) to remove oily or organic residue.
6. Nitric acid as a 10% solution for removing metal contaminants from equipment
7. Organic solvent for final cleaning of equipment (*e.g.* Acetone)
8. Personnel protective equipment (PPE) - including disposable gloves (nitrile preferred), disposable wipes, eye wash system, first aid kit, and waterproof outerwear (if necessary).
9. Re-sealable buckets approved for waste collection and transportation.
10. Squirt bottles for water, alcohol, and solvents.
11. Brushes for cleaning equipment.
12. Field notebooks, pens, pencils, and digital camera to document decontamination procedures.

IV. Guidelines

A. Groundwater

The following equipment will be used to collect water samples and require decontamination:

1. QED Bladder pumps
2. Plastic tubing
3. Water Interface probe
4. Other equipment that comes into contact with the sample (*e.g.*, buckets, etc.).

Collection of water for laboratory analysis requires that the equipment be cleaned between sample locations to avoid sample contamination. Generally, the cleaning procedures to be followed between sample locations are as follows:

Decontamination: all sample collection tools that contact the sample as well as all bowls and mixing/distribution implements in accordance with the following procedures.

1. Disassemble item
2. Rinse each item with tap water.
3. Scrub the item with a brush and soapy water, using non-phosphate detergent such as Alconox™ for non-oily residue, or a detergent (*e.g.*, Joy™) for items with oily or other sticky organic residue. Be sure to scrub the inside of bladder pumps, bottles, etc. (inside and out), cover bucket, etc.
4. During the scrubbing process, be sure to bleed Alconox™ solution or equivalent through small passageways/nozzles/vents, etc.
5. Rinse the item with tap water to remove all residual soap. Be sure to bleed tap water through small passageways/nozzles/vents, etc.
6. Rinse the item with 10% nitric acid to remove residual metals. Be sure to bleed 10% nitric acid through small passageways/nozzles/vents, etc.
7. Rinse the item with de-ionized water. Be sure to bleed de-ionized water through small passageways/nozzles/vents, etc.
8. Rinse the item with organic solvent (*e.g.*, acetone or equivalent). Be sure to bleed organic solvent through small passageways/nozzles/vents, etc.
9. Rinse the item with de-ionized or analyte-free water and allow to air dry. Be sure to bleed analyte-free water through small passageways, nozzles, vents, etc.
10. Re-assemble item(s).
11. Wrap the item(s) in aluminum foil or plastic bag to protect it until it is used.

B. Soil and Rock

The following equipment will be used to collect rock cores, ream and drill boreholes, and require decontamination:

1. Rotary and rock core drilling rig (truck-mounted or skid type) sampling equipment (e.g., rock core barrels). Large drilling equipment (*e.g.,* tri-cone bits, casing, augers, rods, core barrels, core bits, etc.) will be pressure steam-cleaned only.
2. Only those rock crusher parts, including the crushing cell and funnel that come into contact with rock samples, will be decontaminated by Stone Environmental according to SEI-6.37.0 (Appendix A-D).
3. Rock core cutting, splitting and preparation equipment that come into contact with rock samples will be decontaminated by Stone Environmental according to SEI-6.37.0 (Appendix A-D).

Collection of rock samples for chemical analysis requires that the equipment be cleaned between boring locations to avoid cross-contamination. Generally, the cleaning procedures to be followed between boring locations are as follows:

Decontaminate all rock sample collection tools that contact the sample as well as in accordance with the following procedures.

1. High pressure steam-wash each item with tap water to remove mud, dirt, or other visually present material.
2. Scrub the item with a brush and soapy water, using non-phosphate detergent such as Alconox™ for non-oily residue, or a detergent (*e.g.* Joy™) for items with oily or other sticky organic residue.
3. Rinse the item with tap water to remove all residual soap
4. Rinse the item with organic solvent (*e.g.* acetone)
5. Rinse the item with analyte-free water and allow to air dry.
6. Wrap the item(s) in aluminum foil or plastic bag to protect it until it is used.

All solvents must be captured and disposed of in appropriate, labeled, aqueous waste containers. All instruments that come into contact with the sample water must be cleaned in the same manner as the sampling device. Liquids collected into the chemical waste container must be discarded in an appropriate waste stream. Staff performing decontamination procedures shall wear appropriate PPE, gloves (*e.g.*, Nitrile) and eye protection. Care must be taken in cleaning not to allow contact of cleaning solutions with clothing as much as possible. If circumstances dictate contact will occur (*e.g.*, splashing, high wind), waterproof outer clothing must be worn (*e.g.*, foul weather gear or rain gear).

Decontamination procedures may vary depending on specific Field Sampling Plan specifications, and unique contaminants of concern at specific locations. The project workplan may designate collection of equipment rinse samples to document effectiveness of cleaning.

V. Reference

American Society for Testing and Materials (ASTM), 1994. Standard Practice for Decontamination of Field Equipment Used at Nonradioactive Waste Sites. Designation: D 5088 – 90.

Quality Assurance Manual

Prepared by:

Malcolm Pirnie Inc.

July 2008

Revision 0

Review and Approval Sign Off Sheet
Quality Assurance Manual

Malcolm Pirnie, Inc., Vice President:

Ray Lees **Signature:** _____

Malcolm Pirnie, Inc., Environmental Laboratory Manager (ELM):

Max Bateman **Signature:** _____

Malcolm Pirnie, Inc., Quality Assurance (QA) Officer:

James McCann **Signature:** _____

Audit Trail Form

Version	Date Changed	Description	Reason	By Whom

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Acronyms

ATC	Automatic Temperature Compensation
BNC	Bayonet Neill Concelman
DI	Deionized
DNAPL	Dense Non-Aqueous Phase Liquid
DO	Dissolved Oxygen
ELM	Environmental Laboratory Manager
Eh	A measure of the oxidation/reduction state of a solution (h: hydrogen)
FSP	Field Sampling Plan
HASP	Health and Safety Plan
ID	Inner Diameter
LNAPL	Light Non-Aqueous Phase Liquid
mg/L	milligrams per liter
Min	Minute
mL	milliliter
mS/cm	milliSiemen per centimeter
mV	millivolt
LCD	Liquid Crystal Display
N.J.A.C.	New Jersey Administrative Code
NJDEP	New Jersey Department of Environmental Protection
NTU	Nephelometric Turbidity Unit
ORP	Oxidation Reduction Potential
PDR	Personal DataRam
PID	Photoionization Detector
PPE	Personnel Protective Equipment
ppm	Parts per Million
PT	Performance Testing
redox	Oxidation-Reduction Potential
QA	Quality Assurance
QAM	Quality Assurance Manual
SAP	Sampling and Analysis Plan
SOP	Standard Operating Procedure

1.0 Introduction

New Jersey Administrative Code (N.J.A.C.) 7:18, *Regulations Governing the Certification of Laboratories and Environmental Measurements*, October 20, 2003, established a certification program for laboratories performing environmental analyses under NJ State regulatory programs. As part of the certification program, the laboratory is required to develop a Quality Assurance Manual (QAM) as well as standard operating procedures (SOP) for the analyses of concern. This QAM and the attached SOPs were developed for Categories WPP02 (Inorganic Parameters, Nutrients and Demands) and WPP03 (Analyze-Immediately Inorganic Parameters). These categories cover analytical results used under the Water Pollution Program and include specific conductance, turbidity, oxygen (dissolved), pH, and temperature. Hereinafter, these are referred to as listed parameters.

The purpose of this QAM is to establish and document the policies and procedures relating to laboratory quality assurance for Malcolm Pirnie for the collection and analysis of the above listed parameters for groundwater samples. These procedures are established to ensure the quality of the data produced. All personnel collecting or analyzing analyze-immediately parameters under N.J.A.C. 7:18 will be required to read and understand the elements of this QAM.

In addition to the QAM, site-specific planning documents will also be developed. If there is a conflict between this QAM and the site-specific document, the more stringent procedure will apply.

2.0 Program Organization and Responsibility

The purpose of this section is to identify the individuals responsible for administering the N.J. Department of Environmental Protection (NJDEP) laboratory certification program and detail their responsibilities. All personnel identified in this section meet the personnel requirements outlined in N.J.A.C. 7:18-2.10, *Environmental Laboratory Personnel Requirements.* Personnel information including college degree(s) received, training courses attended, health and safety program documentation, and resumes are kept on file at Malcolm Pirnie's corporate offices in White Plains, New York.

Max Bateman, Environmental Laboratory Manager (ELM)

The ELM, Max Bateman, or his designee, has the overall responsibility of overseeing the program including the applicable duties listed in 7:18-2.11(a)1. Other responsibilities of the ELM include, but are not limited, to the following. Note that any designee of the ELM will also meet the requirements in N.J.A.C. 7:18-2.10.

- Supervising the development and revision of this QAM and the associated SOPs
- Ensuring field sampling and measurement equipment is properly maintained and calibrated
- Ensuring that all laboratory personnel meet the requirements in N.J.A.C. 7:18
- Conducting personnel training regarding operation of the water quality equipment and ensuring the technical support staff are properly prepared for equipment operation
- Conducting audits of field sampling and equipment use procedures, if applicable
- Developing and maintaining program files
- Performing performance testing (PT) sample analysis and reporting the results to the NJDEP

Max Bateman, Supervisor

The Supervisor, Max Bateman, or his designee, is responsible for the day to day operations of the program including the applicable duties listed in 7:18-2.11(a)2. Other responsibilities of the Supervisor include, but are not limited to the following. Note that any designee of the Supervisor will meet the requirements in N.J.A.C. 7:18-2.10.

- Maintaining up-to-date SOPs for the field equipment
- Performing regular maintenance and calibration of the equipment utilized in performance of the program
- Housing the equipment at the Monsey Equipment Facility Lab when it is not utilized at a project site
- Maintaining equipment maintenance records

James McCann, Quality Assurance (QA) Officer

The QA Officer, Jim McCann, or his designee, is responsible for the applicable duties listed in 7:18-2.11 (a)3 and is responsible for ensuring that laboratory personnel follow the quality control procedures as discussed in 7:18 including implementing the procedures presented in this QAM. Note that any designee of the QA Officer will meet the requirements in N.J.A.C. 7:18-2.10 (b).

Technical Support Staff

Technical support staff are responsible for the field collection and analysis of analyze-immediately parameters. Technical support staff will have the necessary experience and knowledge to conduct sample collection and water quality testing as outlined in this QAM and the associated SOPs. Responsibilities of the technical support staff include, but are not limited to:

- Maintaining all equipment in their possession; this includes, as required, keeping the equipment clean and in working order, performing daily calibrations, and performing any other minor maintenance required (*e.g.*, checking/changing the batteries)
- Familiarizing themselves with the requirements contained in this QAM and the associated SOPs
- Keeping all required records for the collection and analysis of analyze-immediately parameters
- Signing this manual as evidence of their review and understanding of this program

3.0 Health and Safety

The purpose of this section is to describe the minimum health and safety procedures associated with determination of listed parameters in groundwater samples. Since these samples do not require sample preservation, nor does decontamination of the sample equipment require any chemicals beside phosphate-free soap and water, the only personnel protective equipment (PPE) required are disposable powder-free nitrile gloves. The gloves are used to prevent cross-contamination during sample collection and analysis. PPE requirements could be modified based on site-specific conditions.

It should be noted that chemical samples may be collected along with samples for the listed parameters. Therefore, more stringent site-specific PPE requirements may be necessary. Site-specific health and safety requirements will be included in each site's Health and Safety Plan (HASP).

4.0 Sample Acquisition

The objective of this QAM is to describe the procedures used to produce data that are legally defensible and meet the quality requirements detailed in N.J.A.C. 7:18. Specific procedures for collecting and analyzing the samples, including sample container requirements, are contained in the specific SOPs attached to this QAM. These procedures detail the minimum sampling requirements and are meant to compliment any requirements contained in an approved site-specific Field Sampling Plan (FSP) or Sampling and Analysis Plan (SAP).

5.0 Equipment Maintenance and Calibration

All equipment must be properly maintained to ensure their proper function so they can produce technically valid data. All equipment must also be calibrated, as required by the manufacturer, as well as calibrated daily in the field, to ensure the accuracy and reliability of the measurements. Equipment specific calibration and maintenance procedures are described in the SOPs attached to this QAM. However, in general, the following procedures apply to all equipment.

- All applicable SOPs and equipment manuals will be readily available to personnel using the equipment.
- Only personnel trained in the use of the field equipment will operate them.
- All equipment will be calibrated as required by the equipment manufacturer, or as needed if the daily calibration indicates the instrument is out of calibration. Records of each calibration will be maintained by the Laboratory Supervisor.
- During use, each piece of equipment will, at a minimum, be calibrated at the beginning and end of each day. The calibration information will be recorded in the site's field logbook. Any problems with the equipment will be reported to the Laboratory Supervisor. If the equipment does not pass the calibration, it will not be used in the field.
- All calibration standards will be traceable to a reputable source
- The field personnel in possession of the equipment will be responsible for the day-to-day maintenance of the equipment (*e.g.*, battery checks) and all equipment will be inspected prior to use. Normal upkeep of equipment includes, but is not limited to, the following. Note that all equipment will be maintained as described in the equipment manual.
    - Cleaning
    - Lubrication of moving parts,
    - Check, charge, or change battery
    - Inspect for damage
    - Inspect all hoses and lines

6.0 Record Keeping

Generally, all calibration information will be recorded on the Instrument Calibration Record. Standards used to calibrate equipment will be documented by trade name, lot number and expiration date. Any unusual readings and routine maintenance procedures will also be documented. Additionally, each instrument will be accompanied by an equipment calibration record indicating historical and current calibration information. Additional record keeping requirements are given in the attached SOPs.

ATTACHMENTS

Standard Operating Procedures

SOP LCP001 - Listed Parameter Sample Equipment Decontamination Procedures

SOP LCP002 - Listed Parameter Sample Collection Procedure

SOP LCP003 - MiniRae Photoionization Detector Calibration and Maintenance

SOP LCP004 - Personal Dataram Model PDM-3 Calibration and Maintenance

SOP LCP005 - On-Site Turbidity Measurement of an Aqueous Sample Using a Portable Turbidity Meter, Method 180.1

SOP LCP006 - On-Site pH Measurement of an Aqueous Sample Using a Portable pH Meter, Method 150.2

SOP LCP007 - On-Site mV Measurement of an Aqueous Sample Using a Portable pH/mV Meter, Method 2580

SOP LCP008 - On-Site Conductivity Measurement of an Aqueous Sample Using a Portable Conductivity Meter, Method 120.1

SOP LCP009 - On Site Dissolved Oxygen Measurement of an Aqueous Sample Using a Portable DO Meter, Method 360.1

SOP LCP010 - Field Measurements of an Aqueous Sample Using a Portable Multiple Parameter Meter

SOP LCP011 - On-Site Temperature Measurement of an Aqueous Sample Using a Portable Field pH Meter.

SOP LCP012 - Heron Dipper - T Water Level Indicator Operation and Maintenance

SOP LCP013 - Heron Model H Oil Interface Operation and Maintenance

SOP LCP014 - Record Keeping

Procedure for Decontamination of Sample Equipment Used for Analyze-Immediately Sample Analyses

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1.0 PURPOSE AND APPLICATION

This procedure describes how the technical support staff will decontaminate equipment used to collect groundwater samples for listed parameter analysis. These procedures are not meant to supersede any requirements contained in an approved site-specific Field Sampling Plan (FSP) or Sampling and Analysis Plan (SAP). In addition, all health and safety requirements contained in a site's Health and Safety Plan (HASP) must also be followed.

These procedures are used to prevent contamination and cross-contamination of equipment used to evacuate and sample groundwater. Removing or neutralizing contaminants ensures protection from contaminants, mixing of incompatible substances, and minimizes error in the collection of samples.

2.0 SUMMARY

All non one-time use equipment used to collect groundwater samples will be decontaminated prior to use. All acids/reagents used in the decontamination will be either ultra-pure, or pesticide-grade, as applicable, and the soap will be phosphate-free. All non one-time use sampling equipment that contacts the groundwater will be cleaned with a phosphate-free soap and water solution. The equipment will then be rinsed with potable water, followed by a rinse with deionized (DI) water. Additional decontamination procedures that will be followed for specific sampling equipment are detailed below. It should be noted that additional procedures may be required in site-specific planning documents.

All tubing that comes in contact with the samples will be either dedicated to a well or one-time use. Tubing used to collect the sample will be Teflon®-lined; other tubing will be PVC, polyethylene or polypropylene.

3.0 PROCEDURE

3.1 Grundfos Pump with Manifold and Flow Through Cell and QED Bladder Pump with Manifold and Flow Through Cell

1. Clean the pump and electric line with phosphate-free soap and water solution
2. Dismantle the pump and clean it internally with phosphate-free soap and water solution
3. If collecting inorganic samples in addition to analyze-immediately samples, rinse the pump, pump parts, and stainless steel manifold with a 10% nitric acid solution, followed by a DI water rinse.

4. If collecting organic samples in addition to analyze-immediately samples, rinse the pump, pump parts, and manifold with solvent, following by a DI water rinse.
5. Rinse the equipment with either DI or analyte-free water, air dry, and re-assemble.
6. Wrap the pump and manifold in aluminum foil

3.2 Honda Centrifugal Pump

1. Clean the check/foot valve with phosphate-free soap and water; rinse with DI water
2. Drain the pump and rinse with potable water
3. Rinse the pump with DI water

3.3 Dedicated Disposable Bailer

All dedicated disposable bailers will be acclimated with ground water prior to sample collection and disposed of after use.

3.4 *Heron/Solinst Water Level Indicator*

The measuring tape and probe will be cleaned with phosphate-free soap and water and then rinsed with potable water. The probe and at least the first 6 feet of tape will be rinsed with deionized water and air dried.

3.5 Heron Oil/Water Interface Meter

1. Wipe down the measuring tape and probe with an oil-absorbing cloth; clean the tape and probe with phosphate-free soap and water; then wipe down the tape and probe with an oil-absorbing cloth again
2. Rinse the probe and any heavily stained tape with hexane or isopropyl alcohol to remove any accumulated product. Collect the rinseate on an oil-absorbing cloth.
3. Rinse the probe with DI water and air dry

4.0 CORRECTIVE ACTION

Any deviation from this procedure must be documented.

5.0 WASTE MANAGEMENT

All decontamination fluids will be disposed of in accordance with the procedures outlined in the site-specific sampling and analysis plan.

Procedure for Collection of Listed Parameter Samples

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1.0 PURPOSE AND APPLICATION

This procedure describes how the technical support staff will collect groundwater samples for listed parameter analyses. These procedures are not meant to supersede any requirements contained in an approved site-specific Field Sampling Plan (FSP) or Sampling and Analysis Plan (SAP). In addition, all health and safety requirements contained in a site's Health and Safety Plan (HASP) must also be followed.

These procedures are used to collect samples that are legally defensible and meet the requirements of N.J.A.C. 7:18.

2.0 PROCEDURE

In general, the following factors will be considered during selection of monitoring well sampling equipment and collection procedures:

- Regional groundwater flow direction
- Depth of the aquifer of concern
- Well location and accessibility
- Topography of the site and well elevations
- Physical condition of well and well construction
- Analytical problems caused by contaminant interaction with well construction materials
- Presence of fixed samplers or pumps
- Well depth and water levels

Once monitoring wells have been chosen for sample collection, proper sampling procedures must be employed to obtain a sample that is representative of the groundwater at the location and to ensure that the sample is neither altered nor contaminated by the sampling and handling procedures. If necessary, water levels should be determined prior to sampling.

To obtain a representative groundwater sample, the volume of stagnant water within the monitoring well must first be removed. After the initial static water level within the monitoring well has been determined, the acceptable procedure to purge the well is to evacuate three to five volumes of standing water from the well prior to sampling, or to pump the well dry. Another method that may be used to verify the collection of a representative groundwater sample is to evacuate the well until measurements of pH and conductivity have stabilized over three to five well volumes.

The following considerations should be taken into account during groundwater sampling activities:

1. All equipment entering the well should be cleaned with appropriate decontamination solutions and should be kept clean by staging the equipment on a minimum of 0.3-ml polyethylene plastic
2. Plastic sheeting should be placed around the monitoring well to prevent sampling equipment from coming into contact with the ground surface
3. All down-hole equipment must be constructed of Teflon®, stainless steel, or polypropylene
4. Bailers and pumps should have either polypropylene monofilament, stainless steel, or Teflon® coated wire for retrieval lines, or at a minimum, leaders of these materials
5. Bailers or pumps should be lowered slowly into wells and should not be allowed to drop freely
6. The bailer should not be allowed to rest against the outside well casing or scrape along the interior of the well
7. Outer gloves should be decontaminated or changed before sampling or handling clean equipment and should be changed in between sample locations
8. Every attempt should be made to sample the mid-screen area of the well
9. The sampling team needs to be cognizant of the water level in the well. Draw down in the well may slow the pump discharge rate and over-pumping the well will aerate the groundwater.
10. Make sure the well cap is closed after sampling

2.1 Sample Containers

This SOP covers analyze-immediately parameters, which have to be analyzed in the field within 15 minutes of sample collection. Therefore, none of these samples will be shipped to an off-site laboratory for analysis. As such, the samples will typically be temporarily collected into a sample container, analyzed, and then the sample aliquot is disposed of. The containers used for these samples will either be one-time use containers made of glass or plastic, or the containers will be decontaminated between samples using lab grade soap and water followed by a triple rinse of deionized water. All sample containers will be of approved materials as described in N.J.A.C. 7:18 Tables 9-1 and 9-2. None of these samples will be preserved prior to analysis.

2.2 Preparing the Monitoring Well for Sample Collection

When collecting groundwater samples from a monitoring well using low-flow sampling procedures, the well is typically purged until the indicator parameters have stabilized. When low-flow sampling procedures are not used for groundwater collection, three to five volumes of standing water are typically removed from the well prior to sample collection, unless the well is pumped dry. This is done to ensure that the samples collected are representative of the groundwater in the aquifer of concern.

2.3 Determining Depth to Groundwater

Procedures for use of an electronic water level indicator to determine the water level within a monitoring well are as follows. Note that all health and safety procedures included in the site-specific HASP must be followed prior to opening the monitoring well.

1. Open the well cap and measure the height of the stick-up.
2. Carefully lower the contact electrode until the electrode touches the groundwater surface. At that point, the electronic circuit of the water level indicator will be complete and a positive beeping sound will emit from the meter. Record the depth from the top of the stick-up to the groundwater surface.
3. To determine depth to groundwater, subtract the height of the stick-up from the depth to groundwater from the top of the stick-up.

2.4 Determining Monitoring Well Excavation Volumes

Once the depth to groundwater has been determined, the volume of water that should be evacuating from the monitoring well prior to sampling must be calculated. This is calculated using the following steps.

1. Clean all down-hole equipment according to all established decontamination procedures.
2. Measure the depth to groundwater from the top of the stick-up using a steel tape with weight attached.
3. Measure the diameter of the well and determine the radius.
4. Determine the depth of the water column in the well by subtracting the depth to the bottom of the well from the depth to groundwater.
5. Determine the volume of standing water in the well using the following equation:

$$V = \pi r^2 L_w (7.48 \text{ gallons/ft}^3)$$

Where:

V	=	volume of the well
π	=	3.14
r^2	=	radius of the well in feet, squared
L_w	=	depth of the water column in feet
7.48	=	gallons of liquid per cubic foot

2.5 Evacuating Monitoring Wells

Methods used to evacuate monitoring wells are dependent upon the depth of the well. While all wells may be hand bailed, the volume of water that needs to be removed will

determine whether this method is practical. In addition, wells under 28 feet in depth from the top of the stick-up to the bottom of the well may be evacuated using surface-positioned centrifugal pumps while wells with a depth greater than 28 feet from the top of the stick-up to the bottom of the well may be evacuated using submersible pumps.

When at least three volumes of groundwater have been purged from a monitoring well, the evacuation procedure may be confirmed with water quality monitoring readings (temperature, pH, conductivity, turbidity, dissolved oxygen, and oxidation-reduction potential). The following procedure may be employed to confirm proper evacuation of a monitoring well.

Note: If the well is pumped dry, and evacuation is obviously confirmed, pH and conductivity readings for this purpose are not necessary and the following procedure will not apply.

1. Using a 1,000 ml beaker, flow through cell, or other acceptable receptacle, collect a sample of the well volume being evacuated from the monitoring well.
2. Record the water quality readings of the sample.
3. Collect samples from the second and third consecutive aliquots purged from the monitoring well. Measure and record the readings.
4. If the three consecutive well sample aliquots are representative of the groundwater in the aquifer, all values will be within pH ± 0.5 units and all other parameter readings will be within ± 3 percent of each other.
5. If the three water quality measurements are stable, sufficient quantity of standing water has been removed from the monitoring well to ensure collection of a representative groundwater sample.

2.6 Groundwater Sample Collection

All information associated with monitoring well sampling (e.g., depth to water in the well, parameter readings, analyses, sample ID, sample date) will be recorded in a field logbook and/or the Monitoring Well Sample Log and store in the project specific files.

2.6.1 Groundwater Sampling Using a Bailer

Procedures for the collection of groundwater samples using a bailer are as follows.

1. Attach a 3 ft stainless steel wire/Teflon® leader to the top of the sampler using stainless steel bolts.
2. Attach a sufficient length of solid braided polyethylene rope to the stainless steel Teflon coated leader.
3. Securely fasten the guy line to a stable immovable object.

4. Carefully lower the bailer into the well to mid-screen depth to collect the sample. Note: Do not allow the bailer to freely drop into the well or scrape the sides of the well casing when collecting the sample.
5. Carefully raise the bailer from the well, coiling the bailer guy line as the sample is retrieved.
6. Do not allow the bailer guy line or other sampling equipment to come in contact with the ground surface. A clean bucket is a good resource for using to keep the sampling equipment from contacting the ground. In the event that a bailer is lost down a monitoring well, the bailer can be easily retrieved using a 1 1/2 oz. metal hook (obtained from a Sporting Goods store) attached to either a length of the bailer guy line or to a length of monofilament fishing line.

2.6.2 Groundwater Sampling via Low-Flow Sampling With a Variable Rate Submersible Pump

The preferred collection of groundwater samples will be conducted using the low rate (less than 500 milliliters (ml)/minute (min)) purging and sampling procedures. This type of sampling is conducted to minimize physical or chemical alteration of the sample during withdrawal.

With this sampling procedure, it is important to have a good connection between the 0.25-inch inside diameter reduced diameter Teflon® tubing and the pump outlet order to avoid air bubbles in the tube. Therefore, a hose clamp or Teflon® tape should be used to seal the connection between the two tubes.

When adjusting the flow rate on the submersible pump for the various parameters, the rate must be adjusted extremely slowly in order to avoid raising the turbidity of the water. If a well has insufficient yield, it may dewater during purging. It is important to avoid loss of pressure in the tubing line as a result of dewatering of the well below the level of the pump's intake. Therefore, purging of the well should be interrupted before the water level in the well drops below the top of the pump, and the well should never be pumped dry. In cases where the well becomes dewatered, samples should be collected as soon as the volume of water in the well has recovered enough to allow sample collection.

In some instances, the key indicator parameters may not stabilize. When one or more of the key indicator parameters do not stabilize after four hours, one of the following procedures should be followed. Note that procedure 4 is the preferred procedure.

1. Continue purging in order to achieve stabilization;
2. Stop purging and do not collect any samples. The steps followed should be documented in the logbook;
3. Stop purging and collect the samples. The steps followed should be documented in the logbook; or

4. Stop purging and secure the well. Purging and sampling should continue the next day.

In order to prevent cascading and/or formation of air bubbles in the tubing, it is important to ensure that the flow rate is sufficient to maintain pump suction. This can be accomplished by minimizing the length and diameter of the tubing (*i.e.*, 1/4 or 3/8 inch inner diameter (ID)) in order to keep the tubing filled with groundwater.

To prevent cross-contamination between wells, dedicated pumps should be used whenever possible. If this is not feasible, the pumps should be decontaminated using the procedure outlined in SOP LCP 001.

The procedure for the low rate purging and sampling is made up of the following steps:

1. Decontaminate the pump as well as the other non-disposable equipment using the decontamination procedure outlined in SOP LCP001.
2. Measure the depth to water and the depth to the bottom of the well (described previously in this SOP).
3. Lower the pump, as well as associated equipment, into the well. The pump must be lowered in a manner that will minimize disturbance to the water column. At a minimum, the pump must be kept at least two feet above the bottom of the well in order to prevent disturbance and resuspension of any sediment or other constituents present at the bottom of the well. It should be verified that at least one foot of water will be over the pump intake at all times in order to avoid the risk of pump suction being broken. Whenever possible, the variable rate stainless steel submersible pumps will be dedicated to each monitoring well. At no time will the Teflon®-lined polyethylene tubing be reused between wells.
4. After placing the pump in the well, re-measure the water level. Leave the water level measuring device in the well.
5. Begin purging the well. The purging rate will be between 200 and 500 ml/min. The water level should be monitoring approximately every five minutes. The purging rate should be set so that there is < 0.3 feet of drawdown. Care must be taken to maintain pump suction and to avoid the formation of air bubbles in the tubing. If any adjustments need to be made to the pumping rate, the adjustment as well as the water level immediately after the adjustment should be recorded.
6. Monitor and record measurements of field parameters during purging (approximately every five minutes) to provide an indication of whether the water in the screened interval is representative of the formation. Field parameters to be monitored include: turbidity, temperature, specific conductance, pH, oxygen-reduction potential (ORP: Eh or mV), and dissolved oxygen (DO).
7. After each of the field parameters have stabilized to within the following ranges of acceptable deviation for three consecutive readings; the total volume of water removed from the well will be recorded and purging will be completed.

pH - ±0.1
specific conductance - ±3%
ORP - ±10%
DO - ±10%
Turbidity - ±10%

8. After purging, the sampling discharge rate will be decreased to between 100 and 250 ml/min. The drawdown in the well still may not exceed 0.3 ft. Sample collection will be conducted by filling the sample containers directly from the discharge line. Sampling will follow purging without stopping the flow of the submersible pump.

3.0 CORRECTIVE ACTION

Any deviation from these procedures must be documented.

4.0 WASTE MANAGEMENT

All fluids will be disposed of in accordance with the procedures outlined in the site-specific SAP.

Procedure for MiniRae Equipment Calibration and Maintenance

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MINIRAE 2000 PHOTOINIZATION METER

1.0 ACCURACY

The MiniRae 2000 Photoionization Detector (PID) is temperature compensated so that a 20 degree Celsius change in temperature corresponds to a change in reading of less than two percent full-scale at maximum sensitivity. The useful range of the instrument is from 0.2 to 2000 parts per million (ppm). Response time is less than three seconds to 90 percent of full-scale.

2.0 CALIBRATION

- Press and hold the **N/-** key and the **ON/MODE** key at the same time for about five to 10 seconds.
- Meter will display calibrate/select gas.
- Press the **Y/+** key for fresh air calibration.
- Meter will display "fresh air cal?".
- Press the **Y/+** key again.
- The meter will automatically calibrate for fresh air.
- When it is finished, the meter will display the fresh air calibration reading.
- Press the **Y/+** key to accept fresh air calibration.
- Press the **N/-** key to begin span calibration. Make sure the calibration kit with 100ppm isobutylene is ready.
- Once span cal? appears on the screen, press **Y/+** for calibration with isobutylene.
- The meter will calibrate automatically.
- Press and hold the mode key to return to the main display screen.

3.0 PROCEDURE

- Press the **ON/MODE** key for one second to start the instrument.
- Meter will go through a diagnostics check out.
- When the meter displays ready, press the **Y/+** key to begin sampling.

4.0 MAINTENANCE

1. If any of the following conditions occur, consult the troubleshooting guide provided in the Instruction Manual:

a. No meter response
b. Pump malfunction indication.
c. Erratic meter reading display
d. Instrument response slow or irreproducible
e. Low battery indicator

If the troubleshooting techniques fail to resolve the problem, contact the equipment facility manager who will coordinate the return of the instrument to the manufacturer for repair and maintenance.

2. The particle filter inside the probe tip will be checked prior to each day of service. Visual dirt on the filter will require replacement of the filter. During periods of high humidity, the unit will be operated using the water trap filter provided in the instrument kit.
3. The meter battery life will be noted in the usage logs for loss of life indication. If the meter fails to operate for an eight-hour period, the user will record this in the specific instrument log. The battery will receive a deep discharge service upon arrival at the equipment facility as recommended by the manufacturer.

5.0 RECORD KEEPING

All calibration information will be recorded on the Instrument Calibration Record. Standards used in calibration of equipment will be documented by trade name, lot number and expiration date. Any unusual readings and routine maintenance procedures will also be documented. Additionally, each PID will be accompanied by an equipment calibration record indicating historical and current calibration information.

Procedure for Personal Dataram Model PDM-3 Respirable Dust Meter Equipment Calibration and Maintenance

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PERSONAL DATARAM MODEL PDM-3 RESPIRABLE DUST METER

1.0 CALIBRATION

The Thermo Personal DataRam (PDR) Model PDM-3 is factory-calibrated using a representative dust. The meter should be calibrated annually by either the manufacturer or by a calibration facility. The current calibration record will accompany the unit.

1. Place PDR in the Zero bag provided with the instrument. Use the aspirator to fill the bag with clean air.
2. Press the ON/OFF Key to turn the instrument on.
3. Select the START ZERO function from the instrument display. The unit will automatically begin zeroing itself.
4. Once completed, the instrument will display that calibration is ok. Press the On/Off to accept and return unit to initial setup.

2.0 PROCEDURE

Press the ON/OFF key to start the unit. From the meter display, select the GO TO RUN: NEXT Key. Press the ENTER key to start measurements.

3.0 MAINTENANCE

Instrument maintenance consists of keeping the instrument clean and periodically checking the zero setting using the zero bag. At three month intervals, the 9-volt battery should be removed and the battery contacts cleaned of any corrosion. If the instrument has been exposed to a very dusty or corrosive atmosphere, more frequent battery servicing should be used.

On-Site Turbidity Measurement of an Aqueous Sample Using a Portable Turbidity Meter

Method 180.1

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11.0 WASTE MANAGEMENT 4

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1.0 PURPOSE AND APPLICATION

1.1 This procedure determines the turbidity of an aqueous sample using a tungsten filament lamp, a 90 degree detector to monitor scattered light and a transmitted light detector.

1.2 This method is applicable to surface, ground, and saline waters, domestic and industrial wastewaters, and storm waters.

2.0 METHOD SUMMARY

The turbidity of a well-mixed aliquot is determined electronically utilizing the ratio of the transmitted light and the scattered light from a sample.

3.0 FIELD EQUIPMENT DESRIPTION

The Hach 2100P measures turbidity.

Turbidity specifications are:

Principle:	Scattered/Transmitted Light
Range:	0-1,000 NTU
Resolution:	.01 NTU
Accuracy:	±2% Full Scale
Readout:	LCD
Calibration:	4-point Auto

4.0 COLLECTION/STORAGE

4.1 Turbidity samples are collected in unpreserved polyethylene or glass containers.

4.2 All samples are to be analyzed within 15 minutes of collection and should be analyzed immediately.

5.0 CALIBRATION

1. With the instrument off, place a cleaned sample cell filled with prepared diluted standard into the cell compartment. Align the orientation mark of the sample cell with the mark on the front of the cell compartment. Close the lid and press I/O
2. Press CAL. The instrument will display the value of the zero standard.

3. Press READ. The instrument will count down from 60 to 0. When finished, use the displayed value for the correction factor of the 20 NTU standard. Remove the sample from the compartment.
4. The unit will automatically display S1 and 20 NTU. Place a well-mixed sample of the 20 NTU standard into the compartment and press READ. The unit will countdown from 60 to 0, measure the turbidity, and store the value. The display will automatically move to the next standard value. Remove the sample from the compartment.
5. The display will show S2 and 100 NTU. Place the well-mixed 100 NTU standard into the compartment and press READ. The unit will count down from 60 to 0, measure the turbidity, and store the value. The display will automatically move to the next standard value. Remove the sample from the compartment.
6. The display will show S3 and 800 NTU. Place the well-mixed 800 NTU standard into the compartment and press READ. The unit will count down from 60 to 0, measure the turbidity, and store the value. Remove the sample from the compartment.
7. Press CAL to accept the calibration. The unit will automatically advance to measurement mode.

6.0 MEASUREMENT PROCEDURE

6.1 Place the sample in a clean sample cell dedicated for the 2100P meter.
6.2 Cap the sample cell and wipe with a soft, lint free cloth.
6.2 Gently tap on the side of the sample cell to remove air bubbles.
6.3 Press I/O to turn the instrument on. Place the 2100P on a flat surface. Do not hold the unit while taking measurements
6.4 Place the sample cell into the meter cell compartment with the orientation marks on the cell lined with the mark on the cell compartment.
6.5 Press the Range – the unit will show "AUTO RNG".
6.6 Press READ. The unit will show the reading in NTU. Record the reading.

7.0 MAINTENANCE

7.1 Follow the maintenance procedures in the equipment manual
7.2 Replace broken or highly scratched sample cells. Small, slight scratches may be covered with a light coat of silicon oil. Cells with a buildup of matter which cannot be removed will be discarded.
7.3 Lamp and focusing adjustments are not considered routine maintenance and will be performed only when the instrument readings are suspect

8.0 QUALITY CONTROL

All samples are to be analyzed immediately (within 15 minutes of collection).

9.0 CORRECTIVE ACTION

Any deviation from this procedure must be documented.

10.0 RECORD MAINTENANCE

10.1 A calibration and maintenance logbook will be maintained at the MPI equipment facility for each turbidity meter. Standards used in calibration of the equipment will be documented by trade name, lot number, and expiration date. The calibration logbook will be archived as necessary. Calibration and maintenance data will be retained for 10 years.

10.2 Field data records and field calibrations will be recorded on instrument-specific field logbooks. The logbooks will be archived at the equipment facility as necessary. The field logbooks records will be retained at the MPI equipment facility for 10 years.

11.0 WASTE MANAGEMENT

11.1 The sample bottles are of appropriate size to obtain a sample, but limiting the amount of sample for disposal.

11.2 Wastewater samples will be returned to the source, placed into on-site waste containers, or disposed into the sanitary sewer using large amounts of dilution water. The method of disposal will depend on the site-specific planning documents.

12.0 METHOD REFERENCES

12.1 Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020, Revised March 1983, Method 150.2

12.2 Standard Methods for the Examination of Water and Wastewater, 21st Edition, American Water Works Association, Method 4500H.

12.3 Hach Model 2100P Instruction Manual, 2/95, Revision 2.

On-Site pH Measurement of an Aqueous Sample Using a Portable pH Meter

Method 150.2

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11.0 WASTE MANAGEMENT 4

12.0 METHOD REFERENCES 4

1.0 PURPOSE AND APPLICATION

1.1 This procedure determines the pH of an aqueous sample using a glass electrode.
1.2 This method is applicable to surface, ground, and saline waters, domestic and industrial wastewaters, and storm waters.

2.0 METHOD SUMMARY

The pH of a well-mixed aliquot is determined electronically with a pH electrode. The pH electrode is an integral part of the pH/temperature measuring meter.

3.0 FIELD EQUIPMENT DESRIPTION

The Oakton Model 11 measures pH, mV, and temperature.

pH specifications are:

Principle:	Glass Electrode
Range:	pH -2.00 – 16.00 pH
Resolution:	0.01 pH
Accuracy:	±0.01 pH
Readout:	LCD
Calibration:	2-5 Point Auto

4.0 COLLECTION/STORAGE

4.1 pH samples are collected in unpreserved polyethylene or glass containers.
4.2 All samples are to be analyzed within 15 minutes of collection and to the extent practical should be analyzed immediately.

5.0 CALIBRATION

Calibration of the Model 11 meter will be performed using three standard buffer solutions of pH = 7.00, pH = 4.00, and pH = 10.00. Buffer solutions are standardized at 25 degrees Celsius against National Bureau of Standards. The following procedure will be used for calibration:

1. Ensure that the meter is in the pH mode. If not, press the MODE key until pH is indicated in the right hand corner of the meter display.

2. Rinse the pH and temperature electrodes with DI water. Place the electrodes into the pH 7.00 buffer.
3. Press CAL/MEAS to enter calibration mode. The CAL indicator will appear. Wait for the reading to stabilize then press the HOLD/Enter key to confirm the setting.
4. Rinse the electrodes in DI water and place them in the next buffer. Repeat steps 2 and 3.
5. Rinse the electrodes in DI water and place them in the next buffer. Repeat steps 2 and 3 again.
6. After the meter is calibrated with the third buffer, the meter will return automatically to the measurement mode.

6.0 MEASUREMENT PROCEDURE

6.1 Place the sample in a clean, straight sided polyethylene or glass container using a sufficient amount of sample to cover the tip of the pH electrode.
6.2 Rinse the probe with DI water to make sure it is clean.
6.3 Place the probe into the sample and gently agitate/stir the sample.
6.4 Allow the reading to stabilize, then record the pH reading on the display.

7.0 MAINTENANCE

7.1 Follow the maintenance procedures in the equipment manual
7.2 When not in use, or between measurements, the pH probe will be kept immersed in, or moistened with, the pH = 4.00 buffer solution
7.3 The batteries will be replaced when the "LO BAT" indicator remains on during the instrument check out
7.4 The pH electrode will be replaced whenever the probe is cracked or irremovable deposits build up on the junction
7.5 If response time or stability problems develop and cannot be corrected, the meter will be sent to the manufacturer for maintenance

8.0 QUALITY CONTROL

All samples are to be analyzed immediately (within 15 minutes of collection).

9.0 CORRECTIVE ACTION

Any deviation from this procedure must be documented.

10.0 RECORD MAINTENANCE

10.1 A calibration and maintenance logbook will be maintained at the MPI equipment facility for each pH meter. Standards used in calibration of equipment will be documented by trade name, lot number, and expiration date. The calibration logbook will be archived as necessary. Calibration and maintenance data will be retained for 10 years.

10.2 Field data records and field calibrations will be recorded on instrument-specific field logbooks. The logbooks will be archived at the equipment facility as necessary. The field logbooks records will be retained at the MPI equipment facility for 10 years.

11.0 WASTE MANAGEMENT

11.1 The sample bottles are of appropriate size to obtain a sample, but limiting the amount of sample for disposal.

11.2 Wastewater samples will be returned to the source, placed into on-site waste containers, or disposed into the sanitary sewer using large amounts of dilution water. The method of disposal will depend on the site-specific planning documents.

12.0 METHOD REFERENCES

12.1 Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020, Revised March 1983, Method 150.2

12.2 Oakton pH 11 Instruction Manual, 11/03, Revision 2.

On-Site Oxidation-Reduction Potential Measurement of an Aqueous Sample Using a Portable pH/mV Meter

Method 2580

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11.0 WASTE MANAGEMENT 4

12.0 METHOD REFERENCES 4

1.0 PURPOSE AND APPLICATION

1.1 This procedure determines the redox potential (ORP) of an aqueous sample using a platinum electrode.

1.2 This method is applicable to surface, ground, and saline waters, domestic and industrial wastewaters, and storm waters.

2.0 METHOD SUMMARY

2.1 The ORP of a well-mixed aliquot is determined electronically with a dedicated millivolt (mV) Meter. The ORP electrode is an integral part of a pH/mV/Temperature measuring meter.

3.0 FIELD EQUIPMENT DESRIPTION

The Oakton Model 11 measures pH, mV, and temperature.

3.1 ORP specifications are:

Principle:	Platinum Electrode
Range:	-1999 to 1999 mV
Resolution:	0.1 mV
Accuracy:	1 mV
Readout:	LCD
Calibration:	Single Point Manual

4.0 COLLECTION/STORAGE

4.1 ORP samples are collected in unpreserved polyethylene or glass containers.

4.2 All samples are to be analyzed within 15 minutes of collection and should be analyzed immediately.

5.0 CALIBRATION

1. Connect the ORP Electrode to the Bayonet Neill Concelman (BNC) connector of the Model 11 pH/mV/Temperature meter.
2. Use the mode key to set the display in the upper right hand corner of the meter to mV.
3. Rinse the ORP electrode with DI water, then place it into the redox solution. The meter reading should be between 200 and 275 mV. (Use Hanna Model HI 7020 Redox Solution for

Platinum and Gold Electrodes. The solution will check the operation of the meter and electrode in the range from 200-275 mV.)

4. If the meter display is below 200 mV, use the small green scrubber pad to clean the metal tip of the ORP electrode. After performing this action, rinse the electrode in DI water and recheck the mV reading.

6.0 MEASUREMENT PROCEDURE

6.1 Place the sample in a clean straight sided polyethylene or glass container using a sufficient amount of sample to cover the tip of the ORP electrode.

6.2 Rinse the probe with DI water to make sure it is clean.

6.3 Place the probe into the sample and gently agitate/stir the sample.

6.4 Allow the reading to stabilize, and then record the ORP reading on the display.

7.0 MAINTENANCE

The maintenance procedures in the equipment manual will be followed. The ORP electrode should be cleaned with warm soap and water. When not in use, the electrode should be stored with a moist cotton ball and placed in the plastic cap provided.

8.0 QUALITY CONTROL

All samples are to be analyzed immediately (within 15 minutes of collection).

9.0 CORRECTIVE ACTION

Any deviation from this procedure must be documented.

10.0 RECORD MAINTENANCE

10.1 A calibration and maintenance logbook will be maintained at the MPI equipment facility for each ORP meter. Standards used in calibration of equipment will be documented by trade name, lot number, and expiration date. The calibration logbook will be archived as necessary. Calibration and maintenance data will be retained for 10 years.

10.2 Field data records and field calibrations will be recorded on instrument-specific field logbooks. The logbooks will be archived at the equipment facility as

necessary. The field logbooks records will be retained at the MPI equipment facility for 10 years.

11.0 WASTE MANAGEMENT

11.1 The sample bottles are of appropriate size to obtain a sample, but limiting the amount of sample for disposal.

11.2 Wastewater samples will be returned to the source, placed into on-site waste containers, or disposed into the sanitary sewer using large amounts of dilution water. The method of disposal will depend on the site-specific planning documents.

12.0 METHOD REFENCES

Standard Methods for the Examination of Water and Wastewater, 21st Edition, American Water Works Association, Method 2580.

Oakton pH 11 Instruction Manual, 11/03, Revision 2.

On-Site Conductivity Measurement of an Aqueous Sample Using a Portable Conductivity Meter

Method 120.1

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1.0 PURPOSE AND APPLICATION

1.1 This procedure determines the specific conductance of an aqueous sample using four voltage electrodes.

1.2 This method is applicable to surface, ground, and saline waters, domestic and industrial wastewaters, and acid rain.

2.0 METHOD SUMMARY

2.1 The specific conductance of a well-mixed aliquot is determined electronically utilizing four electrodes (two voltage detecting electrodes and two voltage applying electrodes). The electrodes are an integral part of a multiple parameter measuring meter.

3.0 FIELD EQUIPMENT DESRIPTION

The YSI Model 30 Portable Conductivity Meter measures temperature, salinity, and conductivity.

Conductivity specifications are:

Principle:	4- Electrodes
Range:	0-200 mS/cm
Resolution:	0.01 mS/cm
Accuracy:	±0.5% Full Scale
Readout:	LCD
Calibration:	2-point Manual

4.0 COLLECTION / STORAGE

4.1 Conductivity samples are collected in unpreserved polyethylene or glass containers.

4.2 All samples are to be analyzed within 15 minutes of collection and should be analyzed immediately.

5.0 CALIBRATION

5.1 Turn the instrument on and allow it to complete its self test.

5.2 Select a standard that most closely matches the samples to be measured

5.2.1 Fresh water - around 1 milliSiemen per centimeter (mS/cm)

5.2.2 Brackish water - around 10 mS/cm

5.2.3 Salt water - around 50 mS/cm

5.3 Place at least 3 inches of calibration solution in a clean sample container.

5.4 Insert the probe into the solution, suspended at least ¼ inch above the bottom of the container.

5.5 Press the MODE key until the unit reads conductivity.

5.6 Allow at least one minute for the reading to become stable. Move the probe from side to side to remove air bubbles.

5.7 Press and release both the UP ARROW and DOWN ARROW keys at the same time. The unit is now in calibration mode.

5.8 Use the UP ARROW or DOWN ARROW key to adjust the meter to the calibration solution value.

5.9 Press the ENTER key. "SAVE" will flash across the display indicating the calibration has been accepted. The instrument will automatically return to measurement mode.

6.0 MEASUREMENT PROCEDURE

6.1 Use the ON/OFF key to turn the instrument on. The instrument will perform a self test for a few seconds. When completed, the temperature will be displayed in the lower right corner indicating the meter is ready to take measurements.

6.2 Place a sample in a clean straight sided polyethylene or glass container using a sufficient amount of sample to completely cover the conductivity probe.

6.3 Rinse the probe with DI water to make sure it is clean.

6.4 Place the probe into the sample and move it from side to side to remove air bubbles. Do not rest the probe on the bottom of the sample container.

6.5 Allow the reading to stabilize, and then record the conductivity reading that is on the display.

7.0 MAINTENANCE

7.1 Follow the maintenance procedures in the equipment manual

7.2 Clean the cell in a warm, phosphate-free soap and water solution. Use a nylon brush to clean the inside of the electrode chamber

7.3 Store the probe inside the meter storage chamber

8.0 QUALITY CONTROL

All samples are to be analyzed immediately (within 15 minutes of collection).

9.0 CORRECTIVE ACTION

Any deviation from this procedure must be documented.

10.0 RECORD MAINTENANCE

10.1 A calibration and maintenance logbook will be maintained at the MPI equipment facility for each conductivity meter. Standards used in the calibration of equipment will be documented by trade name, lot number, and expiration date. The calibration logbook will be archived as necessary. Calibration and maintenance data will be retained for 10 years.

10.2 Field data records and field calibrations will be recorded on instrument-specific field logbooks. The logbooks will be archived at the equipment facility as necessary. The field logbooks records will be retained at the MPI equipment facility for 10 years.

11.0 WASTE MANAGEMENT

11.1 The sample bottles are of appropriate size to obtain a sample, but limiting the amount of sample for disposal.

11.2 Wastewater samples will be returned to the source, placed into on-site waste containers, or disposed into the sanitary sewer using large amounts of dilution water. The method of sample disposal will depend on the site-specific planning documents.

12.0 METHOD REFERENCES

12.1 Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020, Revised March 1983, Method 120.1

12.2 Standard Methods for the Examination of Water and Wastewater, 21st Edition, American Water Works Association, Method 2510.

12.3 YSI 30/30M Instruction Manual.

On-Site Dissolved Oxygen Measurement of an Aqueous Sample Using a Portable DO Meter

Method 360.1

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11.0 METHOD REFERENCES 4

1.0 PURPOSE AND APPLICATION

1.1 This procedure determines the dissolved oxygen (DO) of an aqueous sample using a membrane electrode.

1.2 This method is applicable to surface, ground, and saline waters, domestic and industrial wastewaters, and storm waters.

2.0 METHOD SUMMARY

A YSI Model 55 DO meter and a Horiba U-10 Water Quality Checker (see SOP LCP014) will be used to collect DO readings.

The accuracy of the YSI Model 55 is ±2% of air saturation or ±0.3 milligrams per liter (mg/L).

The range of the instrument is 0-200% air saturation or 0-20 mg/L.

Resolution is 0.1% saturated air or 0.01mg/L.

Temperature accuracy, which can also be measured with this instrument, is ±0.2° C.

3.0 FIELD EQUIPMENT DESRIPTION

The YSI Model 55 Portable DO Meter measures temperature and DO. It consists of a micro-processor with an attached DO probe. It is designed for field use and has a stainless steel body within the probe and a rugged plastic housing for the micro-processor.

4.0 CALIBRATION

1. Ensure the sponge inside the calibration chamber is wet. Insert the probe into the calibration chamber
2. Press the ON/OFF button. Wait 15 minutes for the readings to stabilize.
3. Press and release the UP ARROW and DOWN ARROW keys at the same time.
4. Enter the local altitude in hundreds of feet. Press ENTER
5. The unit should now indicate CAL in the lower left of the display.
6. Make sure that the large DO reading is stable, and then press ENTER. The LCD will prompt you to enter the salinity of the water. Enter any number from 0 to 40 (Zero for fresh water – 40 for salt water). Press ENTER.
7. Remove the probe from the calibration chamber and place it into a solution of Zero Oxygen Standard. Do not mix or agitate the solution. Ensure the probe is

completely submerged within the solution. Cover the top of the sample vessel as much as possible during zero calibration check. Allow reading to stabilize and record the lowest observed reading. Remove the probe and rinse it with DI water.

8. Each time the Model 55 is turned off, re-calibrate before taking measurements. Calibrate at a temperature within 10 degrees C of the sample temperature.

5.0 MEASUREMENT PROCEDURE

After the instrument calibration has been performed, the Model 55 will automatically move to normal operation and is ready for measurements. The MODE Key can be used to move the DO reading from mg/L or air saturation.

6.0 MAINTENANCE

1. When not in use, or between measurements, keep the DO probe either immersed in DI water or inside the calibration chamber and moistened with DI water.
2. Clean the probe and cable in warm phosphate-free soapy water.
3. Membranes will last indefinitely depending on use, but should be replaced if readings become unstable or the membrane solution is beyond its shelf life.
4. The unit should be calibrated each time the unit is turned on and before taking measurements.

7.0 QUALITY CONTROL

All samples are to be analyzed immediately (within 15 minutes of collection).

8.0 CORRECTIVE ACTION

Any deviation from this procedure must be documented.

9.0 RECORD MAINTENANCE

9.1 A calibration and maintenance logbook will be maintained at the MPI equipment facility for each DO meter. The calibration logbook will be archived as necessary. Calibration and maintenance data will be retained for 10 years.

9.2 Field data records and field calibrations will be recorded on instrument-specific field logbooks. The logbooks will be archived at the equipment facility as necessary. The field logbooks records will be retained at the MPI equipment facility for 10 years.

10.0 WASTE MANAGEMENT

10.1 The sample bottles are of appropriate size to obtain a sample, but limiting the amount of sample for disposal.

10.2 Wastewater samples will be returned to the source, placed into on-site waste containers, or disposed into the sanitary sewer using large amounts of dilution water. The method of disposal will depend on the site-specific planning documents.

11.0 METHOD REFERENCES

11.1 Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020, Revised March 1983, Method 360.1

11.2 Standard Methods for the Examination of Water and Wastewater, 14th Edition, Method 422F.

11.3 YSI MODEL 55 Dissolved Oxygen and Temperature System Operation Manual.

On-Site Measurements of an Aqueous Sample Using a Portable Multiple-Parameter Meter

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1.0 PURPOSE AND APPLICATION

1.1 The U-10 determines the turbidity of an aqueous sample utilizing a pulse lighting infrared emission diode and a light scattering receptor. Conductivity is measured through four voltage electrodes. pH is determined electronically with a dedicated pH electrode. Temperature is determined through a thermister that measures the change in resistance that accompanies temperature changes.

1.2 These methods are applicable to surface, ground, and saline waters, domestic and industrial wastewaters, and storm waters.

2.0 METHOD SUMMARY

The Horiba U-10 Water Quality Checker will be used to conduct pH, temperature, conductivity, DO, and turbidity readings. The accuracy for the meter, for each parameter, is:

a.	pH	0.01 pH
b.	Temperature	0.1° Celsius (C)
c.	Conductivity in the 0-1 mS/cm range (mS/cm)	0.01 milliSiemen per centimeter
d.	Conductivity in the 1-10 mS/cm range	0.1 mS/cm
e.	Conductivity in the 10-100 mS/cm range	1 mS/cm
f.	DO	0.1 milligram per liter (mg/L)
g.	Turbidity	1 nephelometric turbidity unit (NTU)

3.0 EQUIPMENT DESCRIPTION

The Horiba U-10 Water Quality Checker simultaneously measures multiple parameters of water quality. It is hand-held and has a large LCD readout. Measurements are taken by immersing the probe directly into a water sample, a small sample container, or a flow through cell. It is a versatile instrument utilized for on-site water monitoring operations.

4.0 COLLECTION/STORAGE

Samples are measured through a variety of methods including direct immersion of the probe, collection in unpreserved polyethylene or glass containers, or in a flow through cell.

All samples are to be analyzed within 15 minutes of collection and should be analyzed immediately.

5.0 CALIBRATION

The Horiba U-10 can either be calibrated for all parameters at once or for each individual parameter. To calibrate the meter for all parameters, a lot certified automatic calibration solution is used for pH, conductivity, and turbidity while DO is calibrated using moist ambient air. The following procedures are used to calibrate the Horiba U-10.

5.1 All Parameters (Auto Calibration)

a. Fill the U-10 Calibration Cup to the Fill line with Auto Calibration Solution.
b. Place the clean and DI rinsed probe into the calibration cup.
c. With the power on, Press the MODE key to move the cursor on the lower display to AUTO.
d. Press the ENTER key. The readout will show CAL. The cursor will automatically move to each parameter. When the calibration is completed, the display will show End and will automatically switch to the MEAS mode. The meter is now ready to take measurements.

5.2 pH

1. With the power on, and pH highlighted, press the MODE key to put the unit into MAINT mode.
2. Press the MODE key to move the cursor to ZERO.
3. Fill the calibration cup with pH 7 buffer and place the probe into the cup.
4. When the reading has stabilized, use the UP/DOWN keys to set the value of the pH buffer (based on buffer value at the temperature of the buffer).
5. Press ENTER to set the zero calibration.
6. Remove the probe from the calibration cup and rinse it with DI water.
7. Press the MODE key to move the cursor to SPAN.
8. Fill the calibration cup with pH 4 or 10 buffer and place the probe into the cup.
9. When the reading has stabilized, use the UP/DOWN keys to set the value of the pH buffer (based on buffer value at the temperature of the buffer).
10. Press ENTER to set the span calibration.

5.3 Conductivity

1. With the power on and COND highlighted press the MODE key to put the unit into MAINT mode.
2. Press the MODE key to move the cursor to ZERO.
3. Use the UP/DOWN keys to set the readout to zero.
4. Press ENTER to set the zero calibration (for a thoroughly cleaned and DI water rinsed probe).
5. Use the MODE key to move the cursor to SPAN.

6. Fill the calibration cup with conductivity standard in the 0-1 range and place the probe into the calibration cup.
7. When the reading has stabilized, use the UP/DOWN keys to set the value of the conductivity standard value.
8. Press ENTER to set the span calibration for this conductivity range.
9. Repeat steps 6 through 8 to calibrate each of the two remaining ranges (1-10 and 10-100).

5.4 Dissolved Oxygen

1. With the power on and DO highlighted press the MODE key to put the unit into MAINT mode.
2. Press the MODE key to move the cursor to ZERO.
3. Submerge the probe into a zero oxygen standard solution (do not use the calibration cup for DO calibration).
4. When the reading has stabilized, press the UP/DOWN keys to set the value to 0.0.
5. Remove the probe from the zero standard and rinse it with DI water.
6. Place the probe in a container of air-saturated water (use an air pump to bubble air through the water).
7. Move the probe slowly up and down through the water and allow the reading to stabilize.
8. Refer to the following table for DO values at various temperatures for the DO setting:

Temp (in °C)	DO (in mg/L)	Temp (in °C)	DO (in mg/L)
0	14.16	21	8.68
1	13.77	22	8.53
2	13.40	23	8.39
3	13.04	24	8.25
4	12.70	25	8.11
5	12.37	26	7.99
6	12.06	27	7.87
7	11.75	28	7.75
8	11.47	29	7.64
9	11.19	30	7.53
10	10.92	31	7.42
11	10.67	32	7.32
12	10.43	33	7.22
13	10.20	34	7.13
14	9.97	35	7.04
15	9.76	36	6.94
16	9.56	37	6.86
17	9.37	38	6.76
18	9.18	39	6.68

Temp (in °C)	DO (in mg/L)	Temp (in °C)	DO (in mg/L)
19	9.01	40	6.59
20	8.84		

9. Press the ENT key to set the span to the correct dissolved oxygen value.

5.5 Turbidity

1. With the power on and TURB highlighted press the MODE key to move the cursor to zero.
2. Fill the calibration cup with <1.0 NTU standard and place the cleaned probe into the calibration cup.
3. After the reading is stabilized, use the UP/DOWN to set the reading to 0.0.
4. Press the ENT key to set the zero calibration.
5. Remove the probe from the calibration cup and rinse it with DI water.
6. Use the MODE key to move the cursor to SPAN.
7. Fill the calibration cup with a NTU standard from 100-800 NTU.
8. After the reading has stabilized, use the UP/DOWN keys to set the reading to the value of the calibration standard.
9. Press the ENT to complete the turbidity span calibration.

5.6 Temperature

With the power on and TEMP highlighted, check the temperature reading of the U-10 with the reading of a certified thermometer. Any deviation of the reading will be recorded and used as a temperature correction factor for measurements taken with the water quality checker.

6.0 MEASUREMENT PROCEDURE

Using a Sample Container

Place the sample in a clean straight sided polyethylene or glass container using a sufficient amount of sample to submerge the probe.

1. Rinse the probe with DI water to make sure it is clean.
2. Place the probe into the sample and gently agitate/stir the sample.
3. Use the SELECT Key to move the upper cursor to view each parameter
4. Allow the readings to stabilize, and then record each parameter reading indicated on the instrument display.

Using a Flow Through Cell

1. Sample is analyzed in a flow through cell during low flow purging and sampling activity.
2. Rinse the probe with DI water to make sure it is clean.
3. Sample is mixed inside the flow through cell through continuous movement of source water through the flow through cell by means of a sampling pump.

4. Use the SELECT Key to move the upper cursor to view each parameter
5. Allow the readings to stabilize, and then record each parameter reading indicated on the instrument display.

7.0 QUALITY CONTROL

All samples are to be analyzed immediately (within 15 minutes of collection).

8.0 CORRECTIVE ACTION

Any deviation from this procedure must be documented.

9.0 RECORD MAINTENANCE

9.1 A calibration and maintenance logbook will be maintained at the MPI equipment facility for each multi-parameter meter. Standards used in calibration of equipment will be documented by trade name, lot number, and expiration date. The calibration logbook will be archived as necessary. Calibration and maintenance data will be retained for 10 years.

9.2 Field data records and field calibrations will be recorded on instrument-specific field logbooks. The logbooks will be archived at the equipment facility as necessary. The field logbooks records will be retained at the MPI equipment facility for 10 years.

10.0 WASTE MANAGEMENT

10.1 The sample bottles are of appropriate size to obtain a sample, but limiting the amount of sample for disposal.

10.2 Wastewater samples will be returned to the source, placed into on-site waste containers, or disposed into the sanitary sewer using large amounts of dilution water. The method of disposal will depend on the site-specific planning documents.

11.0 METHODS REFERENCE

11.1 Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020, Revised March 1983.

11.2 Standard Methods for the Examination of Water and Wastewater, 21st Edition, American Water Works Association.

11.3 Horiba U-10 Instruction Manual, 11/91, Revision 2.

On-Site Temperature Measurement of an Aqueous Sample Using a Portable Field pH Meter

Method 170.1

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1.0 PURPOSE AND APPLICATION

1.1 This procedure determines the temperature of an aqueous sample thermometrically using either a dedicated temperature electrode or combination electrode.

1.2 This method is applicable to surface, ground, and saline waters, domestic and industrial wastewaters, and storm waters.

2.0 METHOD SUMMARY

2.1 The temperature of a well-mixed aliquot is determined thermometrically with a dedicated temperature electrode. The temperature electrode is an integral part of a pH/temperature meter.

3.0 FIELD EQUIPMENT DESRIPTION

The Oakton Model 11 pH Meter measures pH, temperature, and ORP.

Temperature specifications are:

Principle:	Thermister Electrode
Range:	-10° to 110° Celsius (C)
Resolution:	0.1° C
Accuracy:	±0.5° C
Readout:	LCD

4.0 COLLECTION/STORAGE

4.1 Temperature samples are collected in unpreserved polyethylene or glass containers.

4.2 All samples are to be analyzed within 15 minutes of collection and should be analyzed immediately.

5.0 CALIBRATION

5.1 Make sure the temperature probe is connected to the phono jack of the pH meter.

5.2 Switch the meter on. Press the MODE key to select pH mode.

5.3 Press the CAL/MEAS key.

5.4 While in pH calibration mode, press the MODE key to enter the temperature calibration mode.

5.5 Place the ATC probe and a certified thermometer into small container of DI water. Allow time for the readings to stabilize.

5.6 Use the MI/^ or MI/˘ to set the temperature value to the certified thermometer reading.

5.7 Press the HOLD/ENTER key to set the temperature. The meter automatically returns to the measurement mode.

The temperature probe calibration will be checked on a monthly basis with a certified thermometer. Both the temperature probe and the certified thermometer will be placed in a liquid solution and their temperature readings will be recorded. Note: The Model 11 temperature probe is factory calibrated and the manufacturer only suggests re-calibration if temperature errors are suspected.

6.0 PROEDURE

6.1 Place the sample in a clean polyethylene or glass container using a sufficient amount of sample to cover the tip of the temperature electrode.

6.2 Rinse the electrode with DI water to make sure it is clean.

6.3 Place the electrode into the sample and gently agitate/stir the sample.

6.4 Allow the reading to stabilize, and then record the temperature reading on the display.

7.0 QUALITY CONTROL

7.1 All samples are to be analyzed immediately (within 15 minutes of collection).

8.0 CORRECTIVE ACTION

8.1 Any deviation from this procedure must be documented.

9.0 RECORD MAINTENANCE

9.1 A calibration and maintenance logbook will be maintained at the MPI equipment facility for each temperature meter. The calibration logbook will be archived as necessary. Calibration and maintenance data will be retained for 10 years.

9.2 Field data records and field calibrations will be recorded on instrument-specific field logbooks. The logbooks will be archived at the equipment facility as necessary. The field logbooks records will be retained at the MPI equipment facility for 10 years.

10.0 WASTE MANAGEMENT

10.1 The sample bottles are of appropriate size to obtain a sample, but limiting the amount of sample for disposal.

10.2 Wastewater samples will be returned to the source, placed into on-site waste containers, or disposed into the sanitary sewer using large amounts of dilution water. The method of disposal will depend on the site-specific planning documents.

11.0 METHOD REFERENCES

11.1 Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020, Revised March 1983, Method 170.1

11.2 Standard Methods for the Examination of Water and Wastewater, 21st Edition, American Water Works Association, Method 2550.

11.3 Oakton pH 11 Instruction Manual, 11/03, Revision 2.

Procedure for Heron Dipper - T Water Level Indicator Operation and Maintenance

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2.0 MAINTENANCE 2

HERON DIPPER –T WATER LEVEL INDICATOR

1.0 Operation

The Heron Model Dipper-T Water Level Indicator works using an electric circuit encased within a measuring tape. When the probe tip contacts water, the unit will emit a loud beep to indicate the depth to water in a monitoring well or other point source. There is no need to calibrate the water level indicator. Prior to use, ensure that the probe reacts to water when exposed to the conductive source. The beeping tone will indicate that the instrument is working normally. To operate use the following steps:

1. Turn the instrument on by pressing and holding the ON/OFF button.
2. Sensitivity can be adjusted as needed using the + or – buttons.
3. Lower the tape into the well or other point source.
4. The meter will emit a loud pulsating beep when the tip contacts water.
5. Raise the tape back into the reel, cleaning the tape as it is raised back into the instrument. The tape should be cleaned using a non-phosphate soap and water solution.
6. Turn the instrument off holding down the ON/OFF button OR the unit will automatically turn off after one minute.

2.0 Maintenance

1. Clean the probe and tape with non-phosphate soap and water.
2. Rinse the probe and tape with potable water; followed by DI water.
3. Wipe the excess water using a clean cloth.

Procedure for Heron Model H Oil Interface Operation and Maintenance

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HERON MODEL H.0IL INTERFACE METER

1.0 Operation

The Heron Model H Oil Interface Meter works with an infra-red circuit that detects the presence of a liquid. A conductivity circuit differentiates between conductive liquid (water) and non-conductive liquid (*e.g.*, light non-aqueous phase liquid (LNAPL) or dense non-aqueous phase liquid (DNAPL)). The meter is supplied with a length of measuring tape to determine the amount of product layer on the top and/or bottom of a groundwater monitoring well. There is no need to calibrate the interface meter.

However, it should be ensured that the probe reacts to water when exposed to the conductive source. To check the interface portion of the instrument, place the probe in DI water. The solid tone will indicate that the infrared sensor is working normally.

To take measurements, from a monitoring well, lower the tape into the well casing or other point-source. A steady tone will be activated when the probe is exposed to product and a beeping tone when the probe is exposed to water. The difference in the two measurements will provide the depth of the layer/s of product/s.

2.0 Maintenance

1. Wipe down the measuring tape and probe with an oil-absorbing cloth
2. Clean the tape and probe with a solution of non-phosphate soap and water
3. Wipe the tape and probe again with an oil-absorbing cloth
4. Rinse the probe and any portion of the tape heavily stained with Hexane or Isopropyl Alcohol to remove any accumulated product. The rinsate will be collected onto an oil-absorbing cloth.
5. Rinse the probe with DI water and air dry.

Procedure for Record Keeping

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1.0 Purpose

The purpose of this SOP is to document the proper record keeping procedures for the calibration records for all equipment used to analyze analyze-immediately parameters under N.J.A.C. 7:18. This SOP also discusses the proper record keeping for the QAM.

2.0 Records

Calibrations will be performed in the laboratory and in the field.

- Full calibrations (*i.e.*, zero plus the span of the instrument) will be performed in the laboratory prior to each mobilization (*i.e.*, use by the field team). Additional periodic calibrations will be performed, as required to meet N.J.A.C. 7:18 requirements.
- Field calibrations will be performed as described in the specific SOP for each instrument.

During these calibrations, the following records will be kept.

2.1 Laboratory Records

The laboratory calibration data will be recorded in bound laboratory notebooks that are kept in the laboratory. Information to be recorded during laboratory calibration includes:

- Instrument Name, Model, and Serial Number
- Date and time of the calibration
- Instrument Settings
- Standard Information including Standard, Manufacturer, Lot Number, and Expiration Date
- Comments (*e.g.*, was any maintenance required)
- Name of Person Performing the Calibration

2.2 Field Records

Once instruments are issued to a field crew, they will be calibrated as required in the instrument SOPs included in this QAM, as well as in accordance with any site-specific requirements, whichever is more stringent. Information to be recorded during field calibration includes:

- Instrument Name, Model, and Serial Number
- Date and time of the calibration
- Instrument Settings
- Comments (*e.g.*, was any maintenance required)

- Name of Person Performing the Calibration

Field calibration information will be logged on an equipment calibration record card (example shown below), which is issued with every instrument. These cards will be pasted into bound logbooks, which are dedicated to each instrument, once the instrument is returned to the laboratory. These logbooks will be maintained at the laboratory.

Malcolm Pirnie Equipment Calibration Record

Item/Model #/Serial # _____

Date	Settings	Standard	Comments	Name

2.3 Document Records

All personnel collecting or analyzing analyze-immediately parameters under N.J.A.C. 7:18 will be required to read and understand the elements of this QAM. To document that they have read the QAM, they will be required to fill out and sign the form shown below.

Employee Sign-Off Sheet

Organization: Malcolm Pirnie, Inc.

This sign-off sheet signifies that all personnel that have signed and dated this sheet have fully read and understand the requirements in this Quality Assurance Manual, which is associated with the sampling and analysis program performed under N.J.A.C. 7:18.

Printed Name	Signature	Title	Phone Number	Date

Low-Flow Purging and Sampling

A. Method Summary and Application

The purpose of Low-Flow Purging and Sampling (LFPS) is to collect groundwater samples from monitor wells that are representative of ambient groundwater conditions in the aquifer. This is accomplished by setting the intake velocity of the sampling pump to a flow rate that limits drawdown inside the well. LFPS has three primary benefits. First, it minimizes disturbance of sediment in the bottom of the well, thereby producing a sample with low turbidity. Second, LFPS minimizes aeration of the groundwater during sample collection. Third, the amount of groundwater purged from a well is usually reduced as compared to conventional groundwater purging and sampling methods.

Because the method allows collection of groundwater samples with low turbidity, it was originally used for collecting samples for inorganics analysis. The method typically allows the collection of samples for total metals analysis and eliminates the need to filter the samples for dissolved metals analysis. In addition, since the method minimizes aeration of the samples, it can be used to collect samples for analysis of volatile and semi-volatile organic compounds (VOCs and SVOCs), provided that appropriate pumps are used in sample collection, as discussed below.

Advantages of LFPS are:

- Groundwater samples tend to be more representative of actual aquifer conditions with respect to mobile contaminants and turbidity
- It causes minimal disturbance of the formation adjacent to the screened interval
- It is generally less prone to sampling variability compared to other groundwater sampling techniques (e.g., bailers)
- Smaller purge volumes and associated disposal expense
- Increased sample consistency from dedicated systems and reproducibility of data due to reduced operator variability

Disadvantages of LFPS are:

- Misconceptions regarding reduced purging and sampling time
- Sampling from non-dedicated systems requires greater set-up time
- Sampling from dedicated systems requires higher initial capital expenses
- Increased technical complexity
- Increased training needs for sampling personnel
- Attractiveness of advantages may lead to improper and inconsistent application
- Typically not a "first round" sampling option
- Not recommended for wells with long screen intervals unless multiple samples are collected

1. Introduction

The following procedures are specific to LFPS of monitor wells in New Jersey. These procedures were developed in consideration of the USEPA-Region I guidance document dated July 30, 1996

(http://www.epa.gov/region01/measure/well/lowflow8.pdf) and the USEPA-Region II guidance document dated March 16, 1998 (http://www.epa.gov/Region2/desa/hsw/lowflow.txt). In addition, the U.S. Geological Survey's (USGS) Techniques of Water-Resources Investigations, Book 9, National Field Manual for the Collection of Water-Quality Data was consulted (http://water.usgs.gov/owq/FieldManual/). The reader is encouraged to review these guidance documents prior to performing LFPS. The procedures provided in the USEPA and USGS guidance must be followed except where they differ from the information provided below.

2. Low Flow Policy

In the event that a responsible party is conducting a Remedial Investigation without Departmental oversight, submittal of a sampling plan is not required. However, it is highly recommended that the responsible party seek approval for any deviations from this guidance prior to conducting LFPS. In the event that a responsible party decides to use LFPS without submitting a sampling plan and receiving approval, it must be recognized that any deviations from this guidance may result in rejection of the data. In addition, when submitting the results of the LFPS event, the responsible party must include specific details of the LFPS techniques used which demonstrate that they were consistent with the guidance specified below. The responsible party shall also provide adequate rationale justifying any deviations from this guidance whether or not they were previously approved by the Department.

It is also Departmental policy that LFPS is not an acceptable method for any wells with screened or open borehole intervals greater than 5 feet in length **unless:** 1) multiple locations at five-foot intervals along the screen/borehole are sampled, or 2) the data quality objectives (DQOs) warrant sampling a specific zone (e.g., the shallow water table to investigate the potential for vapor intrusion inside a building) or specific zones where sufficient geophysical (e.g., heat-pulse flowmeter, caliper and temperature logs, etc.) and hydrogeological information (e.g., tracer tests) or other evidence (e.g., stained soils or fractures noted on boring logs) that **clearly** identifies the depth(s) at which contaminants are entering the well screen or open borehole.

Once the collection of multiple samples (vertical profiling) in a well has been completed, long-term sampling of the well may require LFPS at fewer depth intervals, or even just one depth interval, depending on the data quality objectives of the sampling and the types of contamination present in the groundwater (e.g., LNAPL, DNAPL, etc).

3. Laboratory Certification (N.J.A.C. 7:18)

N N.J.A.C. 7:18 requires that any environmental laboratory* submitting analytical data to the Department, regardless of quality level, must be certified by the Office of Quality Assurance. This applies to those firms using LFPS instruments associated with the "analyze immediately" category of water quality indicator parameters (WQIPs) including pH, temperature, and dissolved oxygen. Regardless of whether or not the equipment in question is rented or privately owned the requirement for certification can not be ignored. All certification documentation must accompany the instrument into the field and accompany all WQIP data submitted to the Department. (*Environmental laboratory is defined as any laboratory, facility, consulting firm, government or private agency, business entity or other person that the Department has authorized, pursuant to N.J.A.C. 7:18, to perform analysis in

accordance with the procedures of a given analytical method using a particular technique as set forth in a certain methods reference document and to report the results from the analysis of environmental samples in compliance with a Departmental regulatory program).

B. Specific LFPS Considerations

1. Pump Intake Location

When LFPS is performed correctly, the data being collected should be a snapshot of a narrow zone along a length of well screen or fracture in an open borehole. For these reasons, it is important to place the pump intake in the zone of highest contaminant concentration or contaminant flux along the screened/open-hole interval. This is particularly important in wells constructed with more than 5 feet of well screen.

Information to be considered when selecting the pump intake depth should include: 1) evidence of soil/sediment contamination from boring logs; 2) soil/sediment sampling analytical results; 3) vertical profiles of groundwater and soil contamination developed from direct-push sampling and field-screening techniques; and; 4) lithology/stratigraphy, particularly the permeability of the aquifer materials.

Typically, the most permeable zones are selected for the pump intake location since the majority of contaminant mass will be transported through them, particularly as the plume migrates downgradient of the source area. Identification of these zones may be made from borehole geophysical data, (e.g., resistivity, fluid conductance, or natural gamma logging, etc.) and hydraulic conductivity data or grain-size analyses. The use of a series of passive-diffusion-bag samplers in a well may also help to identify the zone of highest VOC contamination. The physical/chemical behavior of the contaminants of concern should be considered when determining the pump intake depth . For example, gasoline-related contaminants may be present near the water table while chlorinated VOCs may be present deeper in the aquifer. If a well is contaminated by both types of contaminants, both may need to be sampled, each from a discrete sampling interval.

As discussed above, LFPS is not an option in wells with screened intervals that exceed 5 feet in length, **unless** multiple sample locations at five-foot intervals along the screen/borehole are investigated. Monitor wells screened across zones of significant geologic heterogeneity or open boreholes in fractured rock may be subject to significant vertical flow. Under those conditions, use of packers to isolate specific zones should be considered.

2. Water Quality Indicator Parameters (WQIPs)

For groundwater investigations in New Jersey utilizing LFPS, the following parameters must be measured in order to determine when well stability has been achieved prior to sampling. Their respective measurements must fall within the stated range for three consecutive readings. If the anticipated "third" reading of any individual parameter does not fall within the stated range, then the process to achieve three consecutive readings for that parameter must be restarted. If, after four

hours, stability has not been achieved for the parameters listed below, follow the recommendations above.

Water Level Drawdown	< 0.3 ft*
pH	± 0.1 unit
Specific Conductance	± 3%
Temperature	± 3%
Dissolved Oxygen	± 10%
Turbidity	± 10% for values greater than 1 NTU
ORP/Eh	± 10 millivolts

* During pump start-up, drawdown may exceed the 0.3-ft target and then recover as flow-rate adjustments are made. In wells with short screens (i.e., 5 to 10 ft long) or when sampling for gasoline constituents at the water table, it is much more important to limit the drawdown to less than 0.3 ft, for example, than a well with 15 ft of screen being sampled for metals only with the pump intake set in a permeable zone 5 ft or more below the water table. When sampling groundwater for VOCs and SVOCs, aerating the water by allowing it to cascade down the inside of the well should be avoided. Therefore, drawdown should not expose the screen more than 0.3 ft below the static water level in the well.

Measurements should be taken once every 5 to 6 minutes. This interval is based upon the time it takes for purge water to replace one flow-through-cell volume (generally 250 ml) and the time it takes to measure and record the data. If the purge rate decreases or if the flow cell volume is increased, the time required for purge water replacement will increase. Forms at the end of this document should be used to record drawdown and the WQIPs.

WQIP measurements must be collected in a manner that will insure integrity of the data being collected. To insure consistency of the data, consideration of the following must be made: 1) tubing diameter, length, and material of construction; 2) flow-through cell design, capacity, decontamination, and "purge-train" set-up; 3) pump selection and plumbing fittings; 4) calibration of flow-through cell probes; 5) purge rate; and, 6) water-level-measurement technique.

3. Purge Volume vs. Stabilization Time

In some cases, it may take considerable time to achieve stabilization of the WQIPs. In other cases, they may never stabilize. However, as provided in USEPA guidance, the following options are available if stability has not been achieved after **FOUR** hours of purging: 1) continue purging until stabilization occurs, no matter how long it takes; 2) discontinue purging, do not collect a sample and document the attempts to reach stabilization; or 3) discontinue purging, collect a sample and document the attempts to reach stabilization. In situations where WQIPs do not stabilize, the sampler must document that LFPS could not be performed and document in the report how the samples were collected.

While every effort should be taken to assure that all of the WQIPs stabilize prior to sample collection, one should keep in mind that the stabilization of some WQIPs may be more difficult to achieve than others. Also, achieving stabilization of some WQIPs may be more important with respect to some contaminant types (e.g., metals versus VOCs, etc.) than others. For example, total metals concentrations tend to increase with increasing turbidity of a water sample due to sorption of metals on solids in the water. Similarly, VOC concentrations may be affected by dissolved oxygen (DO) concentrations (i.e., whether the groundwater is aerobic or anaerobic). In addition to providing information on the effectiveness of LFPS, collection of accurate DO data also aids in the evaluation of monitored natural attenuation (MNA) of VOC plumes. Similarly, temperature data can provide useful information regarding the sampling method. For example, temperature increases resulting from dissipation of heat generated by the submersible pump or from exposure of the tubing to excessive heat at the ground surface can have a significant impact on VOC concentrations in water samples.

If, for whatever reason, a WQIP is not accurately measured during the monitoring process or a certain WQIP does not stabilize, and that particular WQIP **is not** significant with respect to the type of contaminant of concern, sample collection may still proceed. For example, if DO data do not stabilize but all of the other WQIPs including drawdown and turbidity stabilize and samples will be collected for metals only, then the samples may be collected. However, any WQIPs that are affected by field conditions or instrument malfunction, must be discussed in the text of the report in order to alert the end-user of potential data bias. If questions arise regarding when stabilization occurs, the sampler should contact the Department's assigned case manager for the site, if any, either prior to (preferably) or when performing LFPS.

4. Tubing

The inside diameter (ID) of tubing should be no greater than three-eighths of an inch (3/8-in). Quarter-inch (1/4-in) tubing is preferred. Larger tubing diameters reduce flow velocity resulting in a corresponding increase of pump speeds to maintain flow. Increased pump speed will, in turn, elevate the potential for turbulent flow across the screened interval and this may affect the quality of the water being sampled. Conversely, any reduction in flow velocity may allow air to become trapped in the tubing, which may ultimately affect air-sensitive parameters or allow particulates to settle, which may affect turbidity values.

The length of tubing, from the top of the well casing to the flow-through chamber, should be the shortest length manageable. Attention to this detail will help ensure that: 1) exposure to ambient temperature, direct sunlight, and bubble formation are kept to a minimum, and 2) deposited solids or air bubbles will less likely be trapped in tubing bends and re-mobilized after accidental movement. Occurrence of any one or combination of these factors can cause variations in WQIP measurements, which could increase stabilization time. Therefore, tubing must be completely full of water at all times.

If the sampling plan calls for multiple sample locations within the well screen, sampling should proceed from the top location to the bottom location. This will require that additional tubing be coiled at the surface to allow for pump relocation to the next deeper sampling location. In these instances,

the coiled tubing must be protected from ambient conditions and the ground surface, in order to avoid impact to the WQIPs and sample data.

The tubing's material of construction must be either Teflon® or Teflon®-lined polyethylene up to the flow-through cell. This is consistent with collection of any groundwater sample. Tubing downstream of the flow cell may be constructed of a lower-quality, more flexible material. However, when sampling for metals analysis only, the tubing may be constructed of flexible polypropylene or polyethylene.

Tubing "reuse" is not recommended when sampling well-to-well since decontamination of tubing is difficult and time consuming. If tubing is to be reused, it must undergo a rigorous decontamination procedure, which must include a hot water wash/hot air drying process. In addition to the hot water wash/hot air drying, separate decontamination solutions of acetone and nitric acid may have to be pumped through the tubing for 15 minutes, followed by copious amounts of distilled, deionized water rinses. The cost of labor associated with decontamination, including the special handling of cleaning solvents and acid, often exceeds the cost of simply discarding the old tubing and using new tubing for each well. If a decision is made to reuse tubing, then one of the following requirements in the USGS "Water-Quality National Field Manual" must be considered: 1) Collect additional field blanks if VOC concentrations in the last sample collected through the tubing are greater than 500 μg/L, or 2) The tubing should be replaced, rather than cleaned, if VOC concentrations in the last sample exceed 700 μg/L.

5. Flow-Through Cell

Typical flow-through cell design is not complicated and almost all on the market today have common shared features. Cells should be transparent in order to "see" the physical condition of the purge water or air bubbles passing through the system. Highly turbid or iron bacteria-laden water can be visually monitored for change as the purge progresses. The cell must be sealed against unwanted exposure to the atmosphere, thus insuring accurate measurement of air-sensitive parameters (dissolved oxygen, pH, etc.). The total capacity of the cell must be small (<300 ml) in order to maintain a desirable turnover rate of water coming into the cell to ensure real-time data integrity. The in-line design must allow for purge water to enter the flow cell from a bottom port and exit at the top. The discharge may be fitted with a check valve.

Upon initial pump startup, it is good practice to not connect the pump discharge line to the flow-through cell. This will allow the sampler time to monitor drawdown, stabilize the flow rate and prevent fowling of probes by bacteria, sediment, or NAPL. Once drawdown measurements indicate that the flow rate has been controlled and a few minutes (<10) have been allowed to clear any unwanted material, the pump discharge line can then be connected to the flow cell.

Flow cell decontamination is important, not only to reduce the potential for cross contamination, but also to ensure data integrity and consistent instrument performance. The cell and probes should be rinsed with distilled/deionized water between each monitor well as accumulation of suspended material may impact probe performance. If they are exposed to contaminants, use a mild detergent or laboratory glassware cleaning solution. Flow cell exposure to high levels of contamination may

damage probes and require their repair by the manufacturer. Since LFPS is NOT normally a first-round sampling option, knowledge of contaminant levels will generally be known prior to the cell's exposure to purge water.

The location of the flow cell or cells in relation to the sample port is critical. Samples for turbidity measurement, general chemistry and laboratory analysis must be collected ahead of the flow cell. When two cells are used in series, the dissolved oxygen probe must be located in the first cell.

Set up the flow-through cell in a location which will cause minimal fluctuation of the flow rate due to elevation changes in the sample tubing as the tubing is disconnected from the cell prior to sample collection. It is also important to locate the flow-through cell as close as possible to the well head in order to minimize the length of tubing needed between the well head and flow-through cell. The flow-through cell must be protected from ambient conditions and the ground surface.

6. Pump Selection

Pumps used for monitoring WQIPs must be submersible, positive-displacement pumps. Examples of acceptable positive-displacement pumps include bladder, variable-speed submersible-centrifugal, reciprocating-piston, progressive-cavity, and gear pumps. The pump discharge must be fitted appropriately to receive either ¼- or 3/8-inch inside-diameter (ID) Teflon® or Teflon®-lined polyethylene tubing.

Peristaltic pumps are suction-lift pumps which can create a negative pressure gradient. Therefore, their use is not appropriate when collecting groundwater samples for analysis of organic compounds. However, peristaltic pumps may be used for the collection of groundwater samples for analysis of inorganic compounds. It should be kept in mind, however, that sampling with peristaltic pumps may affect the stabilization of some WQIPs including dissolved oxygen, pH and redox potential. Since these WQIPs can be affected by the peristaltic pump, this pump should not be used when these data are to be used to evaluate the effectiveness of Monitored Natural Attenuation of groundwater.

Two basic collection scenarios have a bearing on pump selection. These include: 1) a permanently installed pump system, or 2) a portable (well-to-well) pump installation. Bladder pumps can be used for either scenario, however, only those with disposable bladders and easily cleaned parts are suitable when sampling on a well-to-well basis. Variable-speed submersible-centrifugal pumps, gear or progressive-cavity pumps can be used for either scenario as long as they are constructed of easy to clean stainless steel/Teflon® parts. Pumps constructed with impellers, helicoils, or gears, which are difficult to clean or are constructed of unacceptable plastic parts, are not suitable for sampling. In addition, when conducting LFPS on a portable basis, the power or gas supply line should be isolated from the sample tubing. Power supply and sample tubing lines that form a single unit do not allow for easy decontamination and are not recommended.

7. Plumbing Fittings

A check valve should be incorporated into the tubing train or flow cell discharge to eliminate accidental drainage and subsequent aeration of the flow cell. More importantly, a check valve will

prevent a back-surge of purged water being reintroduced at the screen interval of the well should the power source or pump experience mechanical failure. A back-surge of purge water into the screened interval of the well may result in variability of the WQIPs and create analytical bias. In order to avoid the need to decontaminate the check valve, it may be placed on the discharge side of the flow cell or installed immediately above the pump discharge. Some flow-through cells have check valves built into the unit. By design, bladder pumps also have a check valve built into their construction.

A ¼- or 3/8-inch ID barbed "T" or "Y" fitting, placed ahead of the flow cell, may be used to establish the line which will receive a needle valve for turbidity, general chemistry and analytical sample collection. The "T" or "Y" fitting used should be constructed of Teflon® or stainless steel and decontaminated between each use, if used for analytical samples. The fitting may be constructed of polyethylene and decontaminated between each use if it is only used to sample for turbidity and general chemistry parameters. If analytical samples are collected through the "T" or "Y" fitting and needle valve, then those parts must be incorporated into the field blank collection technique.

When collecting a sample at the port ahead of the flow cell, a flow control valve (stainless-steel needle valve [preferred] or stainless steel/Teflon ball valve [optional]) must be used to prevent backpressure and air bubbles from forming in the tubing (see http://water.usgs.gov/owq/FieldManual/chap4_rpt.pdf, page 84). The "needle valve" offers versatility as it can be used for collection of turbidity, general chemistry **and** analytical samples. It can be used with Teflon® tubing and can be used to control sample flow rate because the design significantly reduces any backpressure gradient. Like all other sampling equipment, the "needle valve" must be decontaminated before use at any well.

8. Calibration of Probes

CALIBRATION OF THE PROBES USED TO MONITOR WATER QUALITY INDICATOR PARAMETERS MUST TAKE PLACE **IN THE FIELD PRIOR** TO THE DAY'S EVENTS. THE OFFICE OF QUALITY ASSURANCE MUST CERTIFY THE PROBES USED FOR pH, DISSOLVED OXYGEN AND TEMPERATURE MEASUREMENT.

There are no exceptions to these rules. Probe calibration is critical to the accurate and precise measurement of WQIPs.

For warranty purposes, **all** manufacturers' instructions for proper care and calibration must be followed. Solutions for probe calibration must be held to the temperature of the liquid (groundwater) being measured as temperature correlation is critical in calculating conductivity, dissolved oxygen and pH. Tables and equations to compensate for the difference between ambient groundwater and calibration solution temperature are sometimes provided in the operating manuals or with the calibration solutions. Some instruments are designed with internal features to compensate for this difference in temperature. The respective difference between calibration of conductivity and specific conductivity requires compensation for groundwater temperature at the time of calibration vs. solution temperature adjusted to 25°C at the time of calibration. For dissolved oxygen, the flow cell itself must

be maintained at the temperature of groundwater during calibration. All efforts made to account for proper temperature control of solutions during calibration must be reported to the end user. All steps must be recorded in the field notes. No sampling shall commence until all instruments are calibrated and operating properly. See the "Tips" section below for further discussion on Temperature of Calibration Solutions.

9. Water Level Measurements

The depth to the top of the water column must be recorded prior to pump installation and/or prior to purging. If the **total** depth of the well needs to be determined (e.g., to verify the correct well designation and/or to determine if silt has accumulated in the bottom of a well), it should be measured at least 48 hours prior to sample collection or after the sample has been collected and the pump removed. Total depth measurements must never be taken immediately before purging as this may cause the re-suspension of solids in the well and prolong the purge time.

Once the initial water-level measurement has been recorded and the pump installed, suspend the water-level probe in the well at the point at which drawdown is equivalent to a 0.3-foot drop. Record water levels simultaneously with WQIP measurements once every five minutes.

Water-level-measurement devices, which may impart some disturbance to the water column (i.e., stainless steel "popper" or coated tape), are not acceptable.

10. Pump Installation

LFPS pump installation can be divided into two general collection scenarios: permanent and portable (well-to-well). Permanent pump installation is the most desirable. Among other advantages are improved consistency in data acquisition and reduced long-term labor, preparation and material costs. However, permanent installation is more typically associated with long-term monitoring due to the high initial capital investment required.

The more common practice is to use a pump on a portable or well-to-well basis. While initial capital investment is comparatively less than that of a permanent installation, this practice requires close attention to quality control aspects of pump selection, preparation and decontamination.

Once pumps have been properly decontaminated and fitted with appropriate tubing, installation of the pump can begin. Ideally, pumps should be installed 24 to 48 hours prior to initiation of purging. However, this is not always practical, especially when site security can not be guaranteed. In addition, wells constructed with flush-mount casing are difficult to protect from storm water or infiltration of other contaminants during the extended period monitor wells are open.

Pumps must be installed in such a manner as to insure any disturbance in the well is kept to an absolute minimum. Once pumps reach the top of the water column, their descent should proceed very slowly through the water column. The actual level where the pump intake is to be suspended must be predetermined. Under no circumstance should the pump make contact with, or be "bounced" off, the bottom of the well.

One helpful method to insure proper intake location is to accurately measure and pre-cut the tubing for each individual well prior to site activity. A mark can be made on the tubing, which coincides with the top of the well. Cutting the tubing off-site in a controlled setting is most desirable. Tubing can be wiped down with paper towels, moistened with distilled/ deionized water, labeled and then sealed into plastic bags until needed. If this practice is used, be sure to allow enough tubing to account for the distance from the top of the well casing to the flow cell.

11. Purge Rates

Control over the purge rate is one of the most critical aspects of this technique. Once the pump is set within the screened interval at the desired location, a clean electronic water-level-monitoring device is lowered approximately 0.3 ft into the water column. Start the pump at a speed that results in a flow rate in the range of 100 to 500 ml/min. Pump the initial purge water to waste in order to prevent any fouling of the flow-through cell. *With the pump running,* connect the tubing to the cell. Make sure that all air is purged from the tubing and flow cell as the system fills with purge water. For LFPS, the pump speed must remain constant such that flow rates never exceed 500 ml/min and, once stabilized, the flow rate must not be varied, even during sample collection. If drawdown continues to exceed 0.3 ft., reduce the pump speed until the drawdown has stabilized but do not adjust pump speed to a flow rate below 100 ml/min. Flow rates below this level may induce pump stalling and undo the effort to reach stabilization. If drawdown does not come under control at 100 ml/min, then a field decision should be rendered as to how far to allow drawdown to continue until sample collection. At no time should evacuation allow any portion of the well screen to be exposed (for wells screened below the water table) or bring the well to dryness.

Adjustments to pump speed are best made during the first 15 minutes. Once a "feel" for the purge rate is obtained, begin recording well stabilization indicators. Any significant change to purge rates after this time may negatively impact well stabilization measurements.

Purge rates are best monitored by measuring the flow from the discharge side of the flow cell with a graduated cylinder. Record all of the required WQIPs once every 5 minutes. Once stability has been attained and recorded, begin sample collection

12. Sampling

Once WQIPs have stabilized, or a 4-hour time decision has been rendered, sampling can proceed. Do not adjust the flow rate; maintain the same pumping rate during sampling that was used to purge the well. Collect the sample directly from the needle valve at the sample port. The needle valve allows for sample collection with significantly reduced backpressure and turbulence and offers the best means for sample collection without affecting water quality. It also allows for monitoring using the flow-through cell during sample collection, thereby allowing a final WQIP measurement to be recorded immediately after sample collection. This is the preferred method, especially if volatile organic compounds are the parameters of concern. Any exceptions to this technique must first be approved in writing from the NJDEP on a case-by-case basis before commencing sampling operations.

If higher than expected water temperatures are being observed, evaluate whether the submersible pump is overheating. If the pump motor is not suspected, check the system for any exposure to direct sunlight, especially during warmer periods of the year.

13. Pump Decontamination

The pump forms one of the two key elements of sampling equipment (tubing is the other). The importance of proper pump decontamination is especially true when pumps are rented and utilized on a well-to-well basis. Never assume that rented pumps have been thoroughly cleaned. **Pumps constructed with plastic parts, or sealed inner workings that are inaccessible to direct handling are not an option for LFPS well-to-well consideration because of their limited ability to be decontaminated thoroughly**.

Most bladder pumps can not be easily decontaminated in the field due to their unique construction. For that reason, bladder pumps are not employed on a well-to-well basis **unless** they are constructed with easy to clean parts and *disposable* bladders. Bladder pumps are best suited for dedicated (permanently installed) scenarios. Another popular pump, the variable-speed, 2-inch diameter submersible, is more adaptable for well-to-well sampling; however, close attention to decontamination is warranted. One manufacturer, Grundfos®, clearly states in the operational handbook that the pump must be completely disassembled, including removal of the motor shaft from the stator housing, and all components within the impeller housing. Care must be taken upon reassembly to insure that the cavity housing the motor shaft is *completely* refilled with distilled/deionized water. Care must also be taken with this pump during periods of cold weather to avoid freezing of the coolant water. Proper decontamination not only helps to ensure more reliable data; it also prolongs the life of any pump.

14. Field Blank Collection

When employing LFPS techniques, collection of the field blank must follow the same general rules for all groundwater sampling equipment. This includes the requirement that "all" sampling equipment, which comes in contact with the sample, must also come into contact with the field blank water. To overcome some of the difficulties that manual field blank collection through the inside of a pumping system creates, the following procedure is strongly recommended. Fill a 1000-ml decontaminated, graduated glass cylinder with method blank water supplied by the laboratory performing the analysis. Place a properly decontaminated pump into the graduated cylinder with sample tubing and plumbing fittings attached. Activate the pump and collect the required field blank samples. As the water is removed from the cylinder, replace it with additional method blank water. This procedure will require that the laboratory supply larger volumes of field blank water i.e., bulk water in liter or 4-liter containers. The traditional requirement that field blank water be supplied in the same identical containers as the sample being collected can not be practically satisfied when using LFSP. The identical bottle-to-bottle field blank requirement is waived for this sampling technique procedure only.

15. Tips

a). Temperature Measurement and Submersible Pumps

Variable-speed submersible pumps such as the Grundfos® Redi Flo 2 pump use water to cool the motor during operation. Sometimes, reduced flow rates may result in insufficient cooling of the motor and may elevate the temperature of the water to a point where it may begin to affect sample integrity. If the pump is used in low-yielding, two (2)- or four (4)- inch-diameter wells, temperature increases that do not stabilize may result. If this is observed, a field decision must be made to either discontinue or continue with LFPS. If all other WQIPs have stabilized, then collecting the sample and qualifying the water-quality data accordingly may be acceptable. If the temperature increase continues and eventually exceeds 40% of the initial recorded temperature (Celsius) and other WQIPs have not stabilized, sampling should be discontinued. Turning the pump off and on to control overheating is not acceptable. Always keep in mind that elevated temperature has a direct relationship with dissolved oxygen, specific conductance and, to a lesser degree, pH measurement. Higher temperatures may also reduce the concentrations of volatile organic compounds in groundwater samples due to their relatively high Henry's Law constants. If sampling with submersible pumps continues to result in elevated water temperature, other sampling alternatives should be discussed with the appropriate regulatory program.

When using some submersible pumps in large-diameter wells (six inch and greater), overheating of the motor, followed by mechanical shutdown and possible motor damage, may occur. This is the result of water being drawn to the pump intake in a more horizontal flow pattern which diminishes the design feature that normally moves cool water vertically across the motor (stator) housing. The use of specially designed shrouds may overcome this condition.

b). Control of Pump Speed

In order to achieve the high turning speeds, low-speed startup torque is generally lacking in some submersible pumps including the Grundfos® Redi Flo 2 pump. When attempting to control initial drawdown and/or sample flow rates, it is possible for the pump to cease pumping. Then, if a check valve has been installed, the pump may not have enough torque to overcome the head pressure when attempting to restart it. Sometimes, turning the pump to the highest speeds will overcome this situation or sometimes the pump may have to be pulled from the well and reinstalled. Neither of these corrective measures is conducive to LFPS. To avoid this scenario, make sure the control box comes equipped with a "ten turn pot" frequency adjustment knob. This will allow significantly greater control over pump speeds and the risk of losing pump flow will be reduced.

c). pH

Monitoring for stabilization of pH in groundwater is relatively straightforward and rarely requires serious troubleshooting. When calibrating for pH, do a two-point calibration, at a minimum. The calibration range should bracket the anticipated pH. If the pH is unknown, then a three-point calibration must be made. The temperature of the buffer solutions should be as close to the

temperature of the groundwater as possible. If the probe does not calibrate properly, check to make sure that the probe's electrical contact points are dry. As with preventative maintenance of any probe, make sure that the pH probe is rinsed with distilled/deionized water between use and cleaned periodically per the manufacturer's specifications. Overnight storage generally requires placement of the probe into a 2-molar (M) solution of potassium chloride. This solution may cause an unwanted build up of salt, therefore, frequent rinsing is necessary.

d). Temperature of Calibration Solutions

Correct field measurement of dissolved oxygen, conductivity and pH requires tight control over calibration solution temperature. Proper calibration calls for solution temperatures of these parameters to be the same as the groundwater being measured. This may be difficult to achieve when field sampling well-to-well as groundwater temperature can vary between wells based on depth, local setting (asphalt vs. open field) and other atmospheric and hydrogeological factors. In addition, it is logistically difficult to bring solutions to groundwater temperature at the point of pump intake without first installing the pump, collecting purge water and allowing sufficient time to bring calibration solutions to appropriate temperatures.

For the purposes of LFPS in New Jersey, calibration solution temperatures and the flow-through cell itself must be maintained at approximately 54° F (12° C ± 2° C) during calibration. When ambient conditions warrant, this will require the suspension of the solutions and flow-through cell in a container/bucket of water at the aforementioned temperature. When calibrating for dissolved oxygen, always make sure the cell is vented to the atmosphere by attaching short pieces of tubing to the inlet and outlet fittings while the cell is submerged.

During the purge phase, record the difference between the stabilized temperature and the temperature of the calibration solutions. This information must be presented to the end user. If the sampling event is extended for two or more days, appropriate adjustments can then be made to more accurately reflect the groundwater temperature during calibration.

16. Low Flow Purging and Sampling for Low Yielding Wells

The principal focus of water supply well installation is well yield. In contrast, the principal focus of monitor well installation is water quality; well yield is of secondary importance. In an attempt to locate and delineate groundwater contamination, monitor wells are frequently installed in low-yielding water-bearing zones.

Low-yield wells present challenges with respect to representative groundwater sample collection. The removal of water by bailers draws down the water level in the well by slug- type increments. Peristaltic pumps draw water out of the well by vacuum (negative pressure) which may result in degassing and VOC loss. The operation of variable-speed, submersible pumps at low flow rates may result in heating of the sample as it flows around and through the pump, which may also lead to degassing and VOC loss.

Wells that yield less than 0.1 l/min (100ml/min) frequently incur significant drawdown during well purging. If drawdown occurs across the screened interval or open borehole of a well, VOC loss may result. The increased stress on a well caused by significant drawdown may also result in an increase in water turbidity. In an effort to facilitate the collection of a representative groundwater sample from low-yielding wells, NJDEP will allow special sampling procedures to be used. This may include sample collection without regard to monitoring WQIPs associated with well stabilization.

At a minimum, water-quality data, well-construction data, water-level data, and accurate well-yield data for each low-yielding well will need to be submitted to the Department prior to the development of an acceptable sampling procedure. Since sample collection may begin almost as soon as purging is initiated, it is imperative that the exact interval where the sample will be collected along the screen be predetermined. Aside from the considerations for monitoring drawdown and WQIPs, all other LFPS considerations discussed above apply here as well. The owner of the well shall also propose possible explanations for the low yield of the well(s). Once the aforementioned information has been received, the Department will work with the well owner to formulate an acceptable sampling plan. The sampling plans will be approved on a case-by-case basis and will be well-specific. Implementation of any special sampling procedure or use of any special sampling equipment shall not be performed without prior NJDEP approval.

LOW FLOW SAMPLING DATA SHEET

SHEET ____ OF ____

SITE: _____ CONSULTING FIRM: _____
DATE: _____ FIELD PERSONNEL: _____
WEATHER: _____

MONITOR WELL #: _____ WELL DEPTH: _____ SCREENED/OPEN INTERVAL: _____
WELL PERMIT #: _____ WELL DIAMETER: _____ inches

PID/FID READINGS (ppm): BACKGROUND: _____
BENEATH OUTER CAP: _____
BENEATH INNER CAP: _____

PUMP INTAKE DEPTH: _____ ft below TOC
DEPTH TO WATER BEFORE PUMP INSTALLATION : _____ ft below TOC

TIME	PURGING	SAMPLING	pH (pH units)		SPECIFIC CONDUCTIVITY (mS/cm)		REDOX POTENTIAL (mv)		DISSOLVED OXYGEN (mg/l)		TURBIDITY (NTU)		TEMPERATURE (degrees C)		PUMPING RATE (ml/min)	DEPTH TO WATER (ft below TOC)
			READING	CHANGE*	READING	CHANGE*	READING	CHANGE*	READING	CHANGE*	READING	CHANGE*	READING	CHANGE*		
				NA		NA		NA		NA		NA		NA		

COMMENTS:

*INDICATOR PARAMETERS HAVE STABLIZED WHEN 3 CONSECUTIVE READINGS ARE WITHIN: ± 0.1 for pH; ± 3% for Specific Conductivity and Temperature; ± 10 mv for Redox Potential; and ± 10% for Dissolved Oxygen and Turbidity

NEW JERSEY DEPARTMENT OF ENVIRONMENTAL PROTECTION
FIELD INSTRUMENT AND CALIBRATION DATA

SITE:_____
DATE: _____
FIELD PERSONNEL:_____
START TIME:_____**STOP TIME:**_____

	METER	PROBE
DO	_____	_____
PH	_____	_____
SPEC. COND.	_____	_____
ORP	_____	_____
TURBIDITY	_____	_____

DISSOLVED OXYGEN Standard	TURBIDITY Reading	ORP

Water Temp_____	D.I. Water _____			Standard Temp._____	
Baro. Pres. _____	_____			Standard Conc._____	
Saturation _____	_____	_____		Initial Reading _____	
Init. Mtr. Rd. _____	_____	_____		Meter reset to _____	
Mtr. reset to _____	_____	_____			
O_2 Satur. % _____	_____	_____			

SPECIFIC CONDUCTANCE

	Conc.	Initial Reading	Meter Reset to	Temperature	Lot # and Exp. Date
Standard#1	_____	_____	_____	_____	_____
Standard#2	_____	_____	_____	_____	_____
Standard#3	_____	_____	_____	_____	_____
Standard#4	_____	_____	_____	_____	_____

pH CALIBRATION

Buffer	Temp.	Initial Reading	mV	Meter Reset To	Lot # and Exp. Date
4	_____	_____	_____	_____	_____
7	_____	_____	_____	_____	_____
10	_____	_____	_____	_____	_____

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APPENDIX A-C
STONE ENVIRONMENTAL, INC.
DOCUMENTS

STANDARD OPERATING PROCEDURE

SEI-6.37.0

Field Methods for Retrieval, Collection, Handling, and Preservation of Rock Samples to be Analyzed for VOCs and Physical Properties

NOTE: This SOP contains proprietary information – Do Not Distribute

SOP Number: SEI-6.37.0 | Date Issued: 06/30/08
Revision Number: 0 | Date of Revision: NA

1.0 OBJECTIVE

Rock cores are retrieved by a drilling team from fractured bedrock aquifers and sampled at approximately one foot intervals to be analyzed for volatile organic compounds (VOCs). Rock core samples will be collected for determination of VOC concentrations in the rock matrix, along with representative samples for physical property analyses including porosity, bulk density, organic carbon content, chloride diffusion coefficient and matrix permeability.

2.0 POLICIES

1. According to 40 CFR Part 160, Subpart E, Section 160.81 and the NELA standards, a testing facility shall have standard operating procedures in writing setting forth study methods that management is satisfied are adequate to insure the quality and integrity of the data generated in the course of a study.
2. Personnel will legibly record data and observations in the field to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

3.0 SAFETY ISSUES

1. If necessary and appropriate, a site-specific health and safety plan shall be created for each study site. A template for creating a proper health and safety plan is provided on the SEI network.
2. Care must always be taken when approaching a sampling location. Do not, under any circumstances, place yourself in danger to collect a sample.
3. If necessary and appropriate, all chemicals are required to be received with Material Safety Data Sheets (MSDS) or appropriate application label. These labels or MSDS shall be made available to all personnel involved in the sampling and testing.

4.0 PROCEDURES

4.1 Rock Core Sampling Equipment and Materials

1. Sharpies and Ball point pens
2. Several small coolers
3. Small Dry-Erase White Board (2)
4. Tough-book Field Computer (2)
5. USB Data Stick
6. Printer
7. Wash Tubs (4)
8. Folding Tables (4+)
9. Chairs (3)
10. Scissors
11. Scrub Brushes (several)
12. Squirt Bottles (4+)
13. Required field personnel protective equipment (PPE).
14. Paper towels
15. Teflon tape (heavy duty)
16. Duct tape
17. Rubber bands
18. Aluminum foil
19. Saran wrap
20. Parafilm
21. Phosphate free detergent
22. Distilled Water
23. Bubble wrap
24. Ziploc bags (large and small)
25. Ice Packs
26. Kim-wipes
27. Crack Hammer (2 or more)
28. Steel Chisel, 3" blade (3 or 4)
29. Pick – For removing o-rings
30. MeOH, Purge and Trap grade and Wash grade
31. Enerpac™ Hydraulic Press
32. Sample Trimming Cell
33. Complete Sample Crushing Cell
34. Stainless Rods (for knocking out pucks)
35. O-rings for Pucks
36. Spare Bolts for Base
37. Wood Blocks 6' Plank marked in 0.1 foot intervals.

4.2 Core Retrieval and Sample Collection

Core runs will be HQ-sized (2.5-inch diameter) in five-foot lengths, collected using a triple-tube core barrel system. Immediately upon arrival of the cores at ground surface, they will be removed from the core barrel and placed in aluminum foil-lined PVC trays (6 ft long, 4-inch diameter PVC pipe split lengthwise) and covered with a continuous sheet of clean aluminum foil to minimize volatilization of VOCs. Aluminum foil will be used to keep most of the core covered while the geologist and hydrogeologist inspect the core and select sample locations based on

presence of fractures, lithology, weathering and evidence of groundwater and/or DNAPL fluid flow. The hydrogeologist will flag the sections of the core to be subsampled for VOC analyses and physical property measurements (moisture content, porosity and bulk density, diffusion coefficient, matrix permeability, organic carbon content). The drilling pace should be controlled by the on-site hydrogeologist to ensure that core is not drilled or retrieved before the hydrogeologist and crusher are prepared to receive it (i.e. previous core run has been completely processed or nearly so).

4.2.1 VOC Sample Selection

Subsamples (i.e. core segments ~1 to 2-inches long) for determining CVOC concentrations willbe taken from the cores immediately to minimize chemical losses due to volatilization. The initial core logging will be performed to identify key features for subsampling purposes and samples will be quickly broken from the core using a hammer and chisel, and then wrapped in aluminum foil for VOC and moisture preservation and given a unique field ID. After these samples have been collected, the core will be logged in more detail by the geologist. Additional sample types will then be selected and removed from the core. For future reference, wood blocks will be placed in the core boxes where samples have been removed, indicating the sample ID, length and depth of the removed section and the type of sample.

Samples for CVOC analysis will be collected using three criteria. First, samples will be taken

immediately adjacent to (including fracture surface) and 6 to 12 inches away from identified fractures, both above and below these features. All breaks in core should be suspected as being

fractures insitu, however, emphasis will be made on features with additional lines of evidence for active fluid flow (i.e. secondary mineral coatings/staining, slickensides, report from drillers

regarding fluid loss/gain at specific depths during coring). These samples are intended for measuring the extent of diffusion into the matrix blocks away from fractures that may have once contained DNAPL phase or are pathways for solute transport. Second, additional subsamples will be collected at distinct changes in lithology /mineralogy. Samples will be collected from both sides of such boundaries, referred to as lithology pairs so that representative samples are collected from the different matrix materials. Third, field duplicate samples will be collected at a minimum frequency of 1/20, taken from the same length of core split lengthwise (along the core axis) to provide samples from the same depth

interval and lithology. The hydrogeologist will exercise judgment during sampling to provide an average sample frequency of one sample every 1 to 2 ft, which will likely provide more samples than specified in the Work Plan, to allow flexibility regarding which samples to analyze. Photographs of the core will be taken that identifies the top/bottom and depth interval of the core run, core location and date. The geologist then continues logging the details and the hydrogeologist selects the physical property samples.

4.2.2 Physical Properties Sample Selection

These samples will be collected approximately every 20 ft in the same manner as the CVOC samples described in Section 2.2.1. An effort will be made to obtain samples from different lithologies or where variations in lithology occur as assessed during core logging. Each sample will be a cylindrical disc of the same diameter as the HQ core retrieved from the core barrel, with a height of 2 to 6 inches. These sections of core will be obtained such that the insitu moisture conditions are retained: working quickly, keeping the core covered and out of direct sunlight, and immediately wrapping and sealing the samples. After the sample is broken from the core, it will be immediately wrapped tightly in clean aluminum foil twice around the circumference for complete coverage of the sample, followed by plastic wrap and tape. Finally, the sample will be completely wrapped in parafilm, labeled and placed in a sealable plastic bag. Each sample will be clearly labeled with the sample ID, core location, depth, and date. Details and a full lithology description of each sample will be recorded in the field notebook.

4.3 VOC Sample Preservation

VOC samples will be collected in 40 mL clear glass VOA vials with Teflon-lined septa and screw caps. The rock core sampling requires both purge and trap grade methanol for rock sample extraction/preservation, and wash grade methanol for decontamination, which should be ordered beforehand and available prior to start of drilling. Each labeled sample container and lid will be weighed empty, and re-weighed once the purge and trap grade methanol has been added to accurately determine the weight and volume of methanol extract. Sample vials are prepared with methanol on the day of sample collection where possible; otherwise the weight of the vial with methanol should be re-checked prior to use. At the end of each drilling day, each of the days vials used for rock samples will be weighed so the exact weight of each rock sample is known and then prepared for shipping. There should be a minimum of 15 g of rock in each sample jar. Sample bottles will be stored in a cooler with ice or fridge set to 4°C until they are shipped back to UW.

Following is a step-by-step outline of the process of rock core sampling for VOCs:

a. Lay core in an aluminum foil-lined split PVC tray. Note top/bottom of core and depth interval, measure length and quickly identify features (fractures, breaks, lithology/mineralogy changes, evidence of fluid flow (i.e. secondary minerals, precipitates, slickensides, etc.) to select sample locations. Cover with aluminum foil to minimize CVOC volatilization and evaporation of pore water.

b. Collect CVOC samples: break off a one to two-inch section of core using a rock hammer and chisel, inserting a wood spacer that specifies the sample ID, depth interval that was removed and the type of sample taken. If a field duplicate is planned, the disc length should be doubled and split lengthwise along the core axis to produce two samples from the same depth interval.

c. Completely wrap each sample in a piece of clean aluminum foil to minimize volatilization loss and place in a zip-loc bag with a unique field ID, and place in a cooler with ice for storage prior to crushing. Record sample information including a lithology description, sample type (e.g. lithology pair, duplicate), depth, position relative to nearby fractures and other pertinent details.

d. Following collection of samples for each core run, transport samples to the crushing area.

e. Crush CVOC samples: collect subsample from each CVOC sample using a hammer and chisel and steel chipping tray, with effort made to remove the outer rind of the sample exposed to drilling fluids. Place subsample in a clean, dry crushing cell and crush with the Enerpac™ system.

f. Empty crushed rock sample (minimum of 15 g) into 40 mL VOA sample vial containing a known volume (20 mL) of purge and trap grade methanol, taking care to avoid splashing of methanol out of the vial. Sample vials and screw caps with septum have been previously labeled and weighed both before and after filling with methanol so the mass/volume of methanol is accurately known. Bottle threads should be wiped clean with a kim-wipe prior to screwing on the cap to remove any rock fragments that prevent the cap from sealing properly.

g. Decontaminate the rock crusher components using the procedure outlined in Section 4.4. Multiple crushing cells will be used (at least four or five sets) to allow processing of all samples from most core runs before decontamination is required.

h. Equipment blanks and field duplicates will each be collected after every 20 samples.

i. After drilling is completed each day, weigh vials with the rock samples so the mass of rock is known. Wrap screw cap lid-bottle seam with Teflon tape, wrap vials with bubble wrap and place individually in zip-lock bags for shipping. Place in cooler with trip blanks, which remain with specific batches of samples until arrival at UW, and keep on ice.

j. Chain of custody (COC) forms will be filled out at the end of each day of sample collection and shipped with each cooler back to Stone Environmental.

4.4 Decontamination of Drilling and Subsampling Equipment

Decontamination procedures are designed to remove all traces of contaminants from the equipment to prevent cross-contamination. Core barrels will be cleaned between runs using clean water and pressure washer. The chisel will be sprayed off with wash grade methanol followed by distilled water, and dried with a clean cloth before re-use. Only those parts of the rock crusher that came into contact with the rock subsamples require cleaning, including the crushing cell with top/bottom plates and funnel used to direct the crushed rock sample into the VOA vial. Chipping trays will also be decontaminated between samples. At least four or five sets of crushing cells will be used, which in most cases should allow processing of all of the samples collected from a 5-ft core run. The procedure for cleaning these components consists of four steps. First, the parts will be cleaned in a phosphate-free detergent (e.g. Alconox) wash to get rid of obvious sediment. The second step is full immersion in a clean water rinse, followed by a methanol (wash grade) rinse using a squirt bottle to remove any traces of contaminants not removed previously. The final step is a rinse with distilled (organic free) water using a squirt bottle to remove all traces of methanol. Parts are then dried using clean paper towels. The soap and water baths will be changed on a regular basis, and equipment blanks will be collected after every twenty samples by wiping inside a clean crushing cell with a kim-wipe soaked in purge and trap grade methanol which is then placed in a 40 mL VOA vial to be submitted for analysis along with the rock samples. Decontamination fluids and remnant rock fragments will be barreled.

4.5 Field QA/QC

Equipment blanks, collected as described above, will be taken after every 20 samples. Trip blanks, consisting of purge and trap grade methanol in 40 mL vials will be placed with each batch of samples stored/shipped in coolers. These are filled with methanol at the same time as the sample vials, and handled with the prepared vials and methanol preserved VOC samples, and remain unopened until returned to Stone Environmental for analysis. Field duplicate samples will be taken every 20 samples on core sections of double length split along core axis, so that the samples are taken from the same depth interval and lithology. Methanol blanks, used to identify pre-existing contamination of the purge and trap grade methanol, are taken at the start and end of each bottle (typically 1 L) while filling sample vials and stored/shipped separately from the samples. All QA/QC samples will be identified on the chain of custody forms and tracked throughout the sample handling and analysis process.

5.0 RESPONSIBILITIES

1. All personnel will legibly record data and observations (including phone conversations) in accordance with this SOP to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

6.0 DEFINITIONS

1. *EPA* means the U.S. Environmental Protection Agency.

2. *Observations & Remarks Form (O&R):* A pre-printed form, which contains mostly blank space for general note taking. The form typically prompts the user for the study or project designation, the SEI project number, the client or sponsor name, the total number of pages (page *n* of n) and requires a signature and date. The form is generally used to capture notes of one person when another, more specific forms is not available.

3. *Raw data* means any laboratory worksheets, records, memoranda, notes, or exact copies thereof, that are the result of original observations and activities of a study and are necessary for the reconstruction and evaluation of the report of that study. In the event that exact transcripts of raw data have been prepared (e.g., tapes which have been transcribed verbatim, dated, and verified accurate by signature), the exact copy or exact transcript may be substituted for the original source as raw data. Raw data may include photographs, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

7.0 REFERENCES

40 CFR Part 160 Good Laboratory Practice Standards, August, 1989.

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

None

9.0 AUTHORIZATION

Written by:_____ Date:_____

Lee Rosberg, Staff Scientist

Approved by:_____ Date:_____

Chris Stone, President