Quality Assurance Project Plan for Sampling and Analysis – Groundwater and GAC Treatment System Monitoring

For the Reilly N.P.L. Site

St. Louis Park, Minnesota

December 2008

Revision 0

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List of Acronyms

Acronym	Definition
BFB	Bromofluorobenzene
C	Celsius
CAS Number	Chemical Abstracts Services
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CCC	Calibration Check Compounds
CCV	Continuing Calibration Verification
CD-RAP	Consent Decree Remedial Action Plan
cm	centimeter(s)
COC	Chain of Custody
COPC	Chemical of Potential Concern
CSLP	City of St.Louis Park
CSM	Conceptual Site Model
DFTPP	Decafluorotriphenylphosphine
DQI	Data Quality Indicators
DQL	Data Quality Level
DQO	Data Quality Objectives
EDD	Electronic Data Deliverable
EHS	Environmental Health and Safety
EML	Environmental Measurements Laboratory
F	Farenheit
FID	flame ionization detector
Ft	feet
FSP	Field Sampling Plan
g	gram
GAC	Granular Activated Carbon
GC	Gas Chromatography
GC/MS	Gas Chromatography/Mass Spectrometry
HASP	Health and Safety Plan
HAZWOPER	Hazardous Waste Operations and Emergency Response
HHRA	Human Health Risk Assessment
HRGC/LRMS	High Resolution Gas Chromatography-Low Resolution Mass Spectrometry
H&S	Health and Safety
ICAL	Initial Calibration
ICV	Initial Calibration Verification

List of Acronyms (Continued)

Acronym	Definition
IDL	Instrument Detection Limit
LCS	Laboratory Control Sample
LCS/LCSD	Laboratory Control Sample/Laboratory Control Sample Duplicate
LIMS	Laboratory Information Management System
MB	Method Blank
MDL	Method Detection Limit
mg/kg	milligrams/kilogram
MPCA	Minnesota Pollution Cotrol Agency
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NA	Not Available
N/A	Not Applicable
ND	Not Determined
NIST	National Institute of Standards and Technology
ng/L	Nanograms per Liter
NPL	National Priorities List
OSHA	Occupational Safety and Health Administration
OU	Operable Unit
oz	ounce
PAH	Polycyclic Aromatic Hydrocarbons
PE	Performance Evaluation
PID	Photoionization Detector
PM	Project Manager
PQO	Project Quality Objectives
ppb	parts per billion
ppt	parts per trillion
PRG	Preliminary Remediation Goals
QA	Quality Assurance
QAM	Quality Assurance Manual
QAPP	Quality Assurance Project Plan
QC	Quality Control
QL	Quantitation Limit
QMP	Quality Management Plan
% R	Percent Recovery
RCL	Recovery Control Limits
RCRA	Resource Conservation Recovery Act
RF	Response factor
RI	Remedial Investigation

List of Acronyms (Continued)

Acronym	Definition
RL	Reporting Limit
RRF	Relative Response Factor
RPD	Relative Percent Difference
RPM	Remedial Project Manager
RSD	Relative Standard Deviation
SDG	sample delivery group
S/N	Serial Number
SIM	Selective Ion Monitoring
SOP	Standard Operating Procedure
SOW	Statement of Work
SPCC	System Performance Check Compounds
SRM	Standard Reference Material
SSO	Site Safety Officer
SVOC	Semivolatile Organic Compounds
TA	TestAmerica, Inc.
TBD	To be determined
TIC	Tentatively Identified Compound
TPH	total petroleum hydrocarbons
UFP	Uniform Federal Policy
µg	micrograms
ug/L	micrograms per liter
USACE	United States Army Corps of Engineers
USEPA	United States Environmental Protection Agency
USGS	United States Geological Service

Introduction

This Quality Assurance Project Plan (QAPP) describes the implementation of the sampling, analysis, and associated Quality Assurance (QA) and Quality Control (QC) activities developed for the Groundwater and Granular Activated Carbon (GAC) Treatment System Monitoring at the Reilly Industries, Inc. Nation Priorities List (N.P.L.) site

This document adopts United States Environmental Protection Agency (USEPA) applicable Uniform Federal Policy (UFP) QAPP Worksheets [Publication Numbers: USEPA: EPA-505-B-04-900A; Department of Defense (DoD): DTIC ADA 427785] (USEPA 2005). Previously approved non-UFP QAPPs were utilized for compilation of the UFP QAPP content of this plan.

Background Information

Groundwater in the City of St.Louis Park has been found to contain polycyclic aromatic hydrocarbons (PAH) as a result of activities at a coal-tar distillation and wood-preserving plant (Site) operated from 1917 to 1972. Numerous previous studies have identified PAH in various aquifers beneath St. Louis Park and adjacent communities. Accordingly, the site of the plant operations was placed on the NPL and the federal and state governments sought remediation of environmental contamination via United States District Court Case No. Civil 4-80-469. As a result of this litigation, a Consent Decree Remedial Action (CD-RAP) was agreed to by the USEPA, the Minnesota Pollution Control Agency (MPCA), Minnesota Department of Health (MDH), the City of St. Louis Park (CSLP), the Reilly Tar and Chemical Corporation, and other parties in April of 1986. This CD-RAP requires sampling and analysis of groundwater and treated groundwater at the site.

The groundwater contamination is being monitored and controlled by a number of wells throughout the City. There are monitoring wells in addition to pumping wells. The pumping wells serve as source and gradient control wells. These wells are screened within the four uppermost aquifers beneath the City. The aquifers, in order of shallow to deep are: the Drift, Platteville, St. Peter, and Prairie du Chien-Jordan aquifers. The City water supply is obtained from the Prairie du Chien-Jordan aquifer.

Well placement was determined by previous studies that identified areas of contamination extending from the Reilly Site. Groundwater is controlled by a series of pumping wells that capture the groundwater flowing from the site. These wells pump the water through a treatment facility that discharges to the sanitary sewer. This limits the further spread of contamination in the groundwater. A number of additional monitoring wells are sampled regularly in accordance with the CD-RAP to determine the rate of natural attenuation. Most of the wells sampled have shown a significant decrease in PAH concentrations since monitoring began in 1988.

A more detailed explanation of site background is contained on Pages 3 through 9 of the Consent Decree. The City's consulting company is ENSR (now AECOM). AECOM works with the City to address issues concerning the CD-RAP which includes work plan development and implementation for various tasks, groundwater sampling, and compliance to the CD-RAP.

A summary of the aquifers which underlie the former wood preserving plant site, their approximate location below the surface level, the general use of the aquifers, and the relative maximum historical PAH

concentration measured in each unit (as indicated by historical records and the federal government's Record of Decision in Case No. Civil 4-80-469) are as follows:

Aquifer	Approximate Depth (ft.)	Use	Approximate Upper Concentration of Total PAH
Drift-Platteville	0 - 90	Industrial/Monitor wells	1,000 µg/l off site
St. Peter	90 - 200	Municipal/Private drinking water wells	10 µg/l off site
Prairie du Chien-Jordan	250 - 500	Municipal drinking water wells	10 µg/l off site
Ironton-Galesville	700 - 750	Industrial	5 µg/l on site
Mt. Simon-Hinckley	800 – 1,100	Municipal drinking water wells	0.080 µg/l off site

More extensive information relative to the identified level of PAHs in the various aquifers is provided in the following reports:

- Annual Monitoring Reports for 1988 through 2007
- St. Peter Aquifer Remedial Investigation Report (March 30, 1989)
- Drift-Platteville Aquifer (Northern Area) Remedial Investigation Report (March 30, 1989)

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

Document Title: Quality Assurance Project Plan for Sampling and Analysis – Groundwater and GAC Treatment System Monitoring for Reilly Industries, Inc. N.P.L Site, St.Louis Park, Minnesota

Lead Organization: AECOM, Inc. (formerly ENSR), representing The City of St. Louis Park, St. Louis Park, Minnesota 55416

Preparer's Name and Organizational Affiliation: Robert Kennedy, AECOM

Preparer's Address, Telephone Number, and E-mail Address:

2 Technology Park Drive, Westford, MA 01886-3140

978-589-3000

Robert.Kennedy@aecom.com,

Preparation Date (Day/Month/Year): December, 2008

Investigative Organization's Project Leader

William Gregg / AECOM, Inc./ December 2008

Regulatory Organization's Project Leader

Remedial Project Manager-Superfund

Matthew J. Ohl / USEPA Region 5/ December 2008

Regulatory Organization's Project Leader

Nile Fellows / MCPA / December 2008

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

Site Name/Project Name: Reilly Industries, Inc. N.P.L. Site / Groundwater and GAC Treatment System
Site Location: St.Louis Park, Minnesota

Site Number/Code: **EPA ID# MND980609804**
Operable Unit: **OU-2, OU-3, OU-4, OU-5**
Contractor Name: **AECOM,Inc.**
Contractor Number: **Not Applicable (N/A)**
Contract Title: **N/A**
Work Assignment Number: **N/A**

1. Identify guidance used to prepare QAPP:

   Uniform Federal Policy for Quality Assurance Project Plans. Evaluating, Assessing, and Documenting Environmental Data Collection and Use Programs. Part 1: UFP-QAPP Manual. Final Version 1. March 2005. Intergovernmental Data Quality Task Force (US Environmental Protection Agency, US Department of Defense, US Department of Energy). USEPA 505-B-04-900A.

2. Identify regulatory program: Comprehensive Environmental Response Compensation, and Liability Act. (CERCLA)

3. Identify approval entity: USEPA Region 5

4. Indicate whether the QAPP is a generic or a project-specific QAPP. (circle one)

5. List dates of scoping sessions that were held:

   No scoping sessions were held.

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

Title
ENSR, 2007. 2008 Sampling Plan, Reilly Tar & Chemical Corp., N.P.L. Site, St. Louis Park, Minnesota. Submitted October 31, 2007
ENSR, 2008. 2009 Sampling Plan, Reilly Tar & Chemical Corp., N.P.L. Site, St. Louis Park, Minnesota. Submitted October 31, 2007

7. List organizational partners (stakeholders) and connection with lead organization:

   This work will be performed under the requirements of the CD-RAP for the Reilly Tar and Chemical Corporation N.P.L Site in St. Louis Park, Minnesota., as well as requirements from USEPA Region 5, the MPCA, and the MDH.

8. List data users: See item #7 above.

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

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

   Worksheet #9 is omitted because project specific scoping session information is not available. Project scope is defined by the requirements of the CD-RAP.

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

Required QAPP Element(s) and Corresponding QAPP Section(s)	Required Information	Crosswalk to QAPP Worksheet No. 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 Planning Session Documentation (including Data Needs tables) - Project Scoping Session Participants Sheet - Problem Definition, Site History, and Background - Site Maps (historical and present)	NA NA 10 and Introduction Reports submitted March 15 Annually
2.6 Project Quality Objectives (PQOs) and Measurement Performance Criteria 2.6.1 Development of PQOs Using the Systematic Planning Process 2.6.2 Measurement Performance Criteria	- Site-Specific PQOs - Measurement Performance Criteria Table	11 12
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

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

Required QAPP Element(s) and Corresponding QAPP Section(s)	Required Information	Crosswalk to QAPP Worksheet No. or Related Documents
Measurement/Data Acquisition		
3.1 Sampling Tasks 3.1.1 Sampling Process Design and Rationale 3.1.2 Sampling Procedures and Requirements 3.1.2.1 Sampling Collection Procedures 3.1.2.2 Sample Containers, Volume, and Preservation 3.1.2.3 Equipment/Sample Containers Cleaning and Decontamination Procedures 3.1.2.4 Field Equipment Calibration, Maintenance, Testing, and Inspection Procedures 3.1.2.5 Supply Inspection and Acceptance Procedures 3.1.2.6 Field Documentation Procedures	- Sampling Design and Rationale - Sample Location Map - Sampling Locations and Methods/ SOP Requirements Table - Analytical Methods/SOP Requirements Table - Field QC Sample Summary Table - Sampling SOPs - Project Sampling SOP References Table - Field Equipment Calibration, Maintenance, Testing, and Inspection Table	17 SAP 18 19 20 Appendix B 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	Appendix C 23 24 25
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 - Example Chain-of-Custody Form and Seal	26 Appendix B 27 27 Appendix B
3.4 QC Samples 3.4.1 Sampling QC Samples 3.4.2 Analytical QC Samples	- QC Samples Table	28

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

Required QAPP Element(s) and Corresponding QAPP Section(s)	Required Information	Crosswalk to QAPP Worksheet No. or Related Documents
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 Procedures	29 30 29
Assessment/Oversight		
4.1 Assessments and Response Actions 4.1.1 Planned Assessments 4.1.2 Assessment Findings and Corrective Action Responses	- Planned Project Assessments Table - Assessment Findings and Corrective Action Responses Table	31 32
4.2 QA Management Reports	- QA Management Reports Table	33
4.3 Annual Report	To be completed following data collection	Not Available (NA)
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	To be completed following data evaluation	NA

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

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

QAPP Recipients	Title	Organization	Telephone Number	Address	Document Control Number
Matthew Ohl	Remedial Project Manager (RPM), USEPA	USEPA	312-886-4442	77 West Jackson Mail Code HSR-6J Chicago, Illinois 60604	
Nile Fellows	Project Leader	MPCA	651-296-7299	520 Lafayette Road North St. Paul, Minnesota 55155	
John Jones	Director of Regulatory Management	Vertellus Specialties, Inc.	317-248-6427	300 North Meridian Street, Suite 1500 Indianapolis, Indiana 46204-1763	
Lisa Uriell	Laboratory Project Manager	TestAmerica(TA) - Denver	303-736-0157	4955 Yarrow Street Arvada, Colorado 80002	
William Gregg	Project Manager/Field Coordinator	AECOM	651-222-0841	413 Wacouta Street St. Paul, MN 55101	
Scott Anderson	Superintendant of Utilities	CSLP	952-924-2557	5005 Minnetonka Boulevard St. Louis Park, Minnesota 55416	
Andrew Tarara	Field QA Manager	AECOM	651-222-0841	413 Wacouta Street St. Paul, MN 55101	
Lori Herberich	Project QA Officer	AECOM	978-589-3383	2 Technology Park Drive Westford, MA 01886-3140	
James Herberich	Data Management Task Manager	AECOM	978.589.3193	2 Technology Park Drive Westford, MA 01886-3140	
Karen Kuoppala	Laboratory QA Manager	TA-Denver	303-736-0157	4955 Yarrow Street Arvada, Colorado 80002	

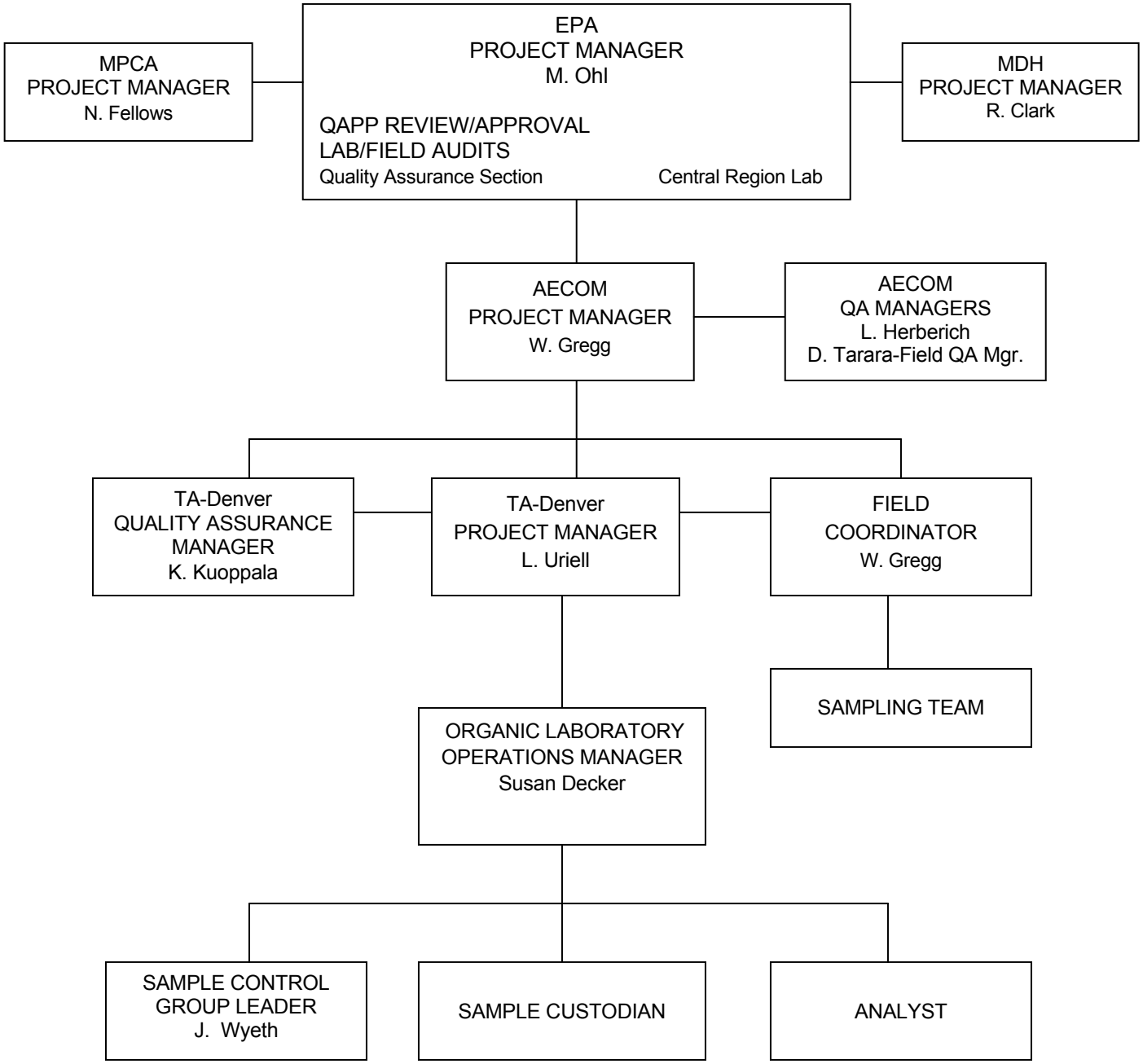
QAPP Worksheet #4 (UFP-QAPP Manual Section 2.3.2) Project Personnel Sign-Off Sheet

Organization: A completed sign-off sheet will be maintained in the files for each organization executing tasks for this project.

Project Personnel	Title	Telephone Number	Signature*	Date QAPP Read

*Signature indicates that personnel have read the applicable QAPP sections and will perform the tasks as described.

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



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

Communication Drivers	Responsible Entity	Name	Telephone Number	Procedure (timing, pathways, etc.)
Field activities status and issues	AECOM Field Coordinator	William Gregg		Communicate daily, or as needed, with AECOM field personnel, subcontractors, and AECOM Field QA Manager directly, or via e-mail or phone. Minor work plan deviations and/or proposed revisions will be documented and communicated in writing, with a copy sent to USEPA.
Sampling progress/laboratory coordination	AECOM Field Coordinator	William Gregg		Communicatedaily, or as needed, with AECOM Project Field QA Manager and Laboratory PM via e-mail or phone.
Health and safety briefings and updates	AECOM Field Coordinator	William Gregg		Communicate daily during samplingwith field personnel.
Significant health and safety concerns or incidents	AECOM Field Coordinator	William Gregg		Communicate immediately with AECOM Regional EHS Manager.
Analytical laboratory issues, including coordination with field, schedule, and technical issues	AECOM Project Field QA Manager	Andrew Tarara		Communicate with AECOM Field Coordinator and Laboratory PM as needed via phone or e-mail.
Analytical data validation issues	AECOM Project QA Officer	Lori Herberich		Communicate with Laboratory PM as needed via phone or email; significant issues will be communicated to AECOM PM and AECOM Project Field QA Manager.
Audit findings (field and/or laboratory)	AECOM Project QA Officer/ AECOM Project Field QA Manager	Lori Herberich/ Andrew Tarara		Communicate findings to AECOM PM or Laboratory PM (as appropriate); transmit final audit reports, including corrective actions to AECOM PM

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

Communication Drivers	Responsible Entity	Name	Telephone Number	Procedure (timing, pathways, etc.)
Project status and issues (internal)	AECOM PM	William Gregg		Communicate with CSLP as needed, via email or phone.
Project status and issues (external)	AECOM PM	William Gregg		Communicate with USEPA RPM and MPCA PM as needed via e-mail or phone; submit monthly status reports.
Data management	Laboratory PM	Mike Phillips		Transmit EDDs to Data Management Task Manager
	AECOM Project QA Officer	Lori Herberich		Communicate with Data Management Task Manager regarding final data qualifiers.
Stop Work (technical non-compliance)	AECOM Field team, Project Field QA Manager	Andrew Tarara		Any personnel believing that a work stoppage is necessary shall first verbally notify the AECOM PM, who will in turn verbally notify CSLP, if necessary. Given the potential significance of such communications, this should occur as quickly as possible.

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

Name	Title	Organizational Affiliation	Responsibilities	Education and Experience Qualifications[1]
William Gregg	Project Manager	AECOM	Overall responsibility for completion of project in accordance with CD-RAP and HASP requirements, including technical, financial, and scheduling. All deviations from approved project plans will be discussed with and approved by the Project Manager. Primary point of contact with the USEPA and MPCA.	BS, Geology, 31 years experience
Andrew Tarara	Project Field QA Manager	AECOM	Responsible for the execution and completion of the monitoring program, including procurement of analyticalsubcontractors, review of task deliverables, and serving as the focus for coordination of all field and laboratory tasks. The Task Manager will keep the AECOM PM apprised of the status of the task, as well communicate any issues with the schedule, budget, or achievement of the task objectives.	BA, Geology, 13 years experience
William Gregg	Field Coordinator	AECOM	Responsible for implementing field sampling activities in accordance with the approved plans. Primary responsibilities will include directing activities on site, monitoring subcontractor performance in the field, reviewing field records, and communicating status, quality issues, or delays with CSLP or USEPA as needed..	BS, Geology, 31 years experience
Lori Herberich	Project QA Officer	AECOM	Responsible for reviewing and approving QA procedures, ensuring that planned QA assessments (e.g., technical surveillance audits) are conducted according to the QAPP, and coordinating data validation.	BS, Forest Biology, 21 years experience

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

Name	Title	Organizational Affiliation	Responsibilities	Education and Experience Qualifications[1]
Peter Sullivan	Regional EHS Manager	AECOM	Responsible for ensuring that the objectives of AECOM Health and Safety Program are met and for monitoring task activities for conformance to the HASP.	BS, Chemical Engineering, 8 years experience
James Herberich	Data Management Task Manager	AECOM	Responsible for data management activities for project.	BA, Engineering Sciences, 22 years experience.
Lisa Uriell	Laboratory PM	TA	Acts as the primary point of contact at TA facilities for the AECOM Project Chemist to communicate and resolve sampling, receipt, analysis, and storage issues.	BS, Biology, 3 years experience
Karen Kuoppola	Laboratory QA Manager	TA	Responsible for reviewing and approving laboratory QA procedures, ensuring that planned QA assessments are conducted according to the QAPP or SAP and relevant EPA methods and associated QA/QC requirements.	BA, Chemistry/Geology, 25 years experience

[1] Resumes of all individuals are available upon request.

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

Project Function	Specialized Training by Title or Description of Course	Training Provider	Training Date	Personnel/Groups Receiving Training	Personnel Titles/ Organizational Affiliation	Location of Training Records/Certificates
PM/ Field Coordinator	40 hour HAZWOPER [a]	AECOM	1/12/84	William Gregg	Project Manager/ AECOM	AECOM St. Paul
	8 hour HAZWOPER Manager/Supervisor	AECOM	5/7/88			
	HAZWOPER 8-hr Refresher	AECOM	12/13/07			
	Hazard Communication	AECOM	10/05/05			
	Hazmat awareness	AECOM	10/20/06			
	Hazmat shipping	AECOM	10/10/06			
	First Aid	ARC	12/18/07			
Project Field QA Manager	40 hour HAZWOPER	Compliance Solutions	3/31/00	Andrew Tarara	On-Site Field Coordinator/AECOM	AECOM St. Paul
	HAZWOPER 8-hr Refresher	AECOM	10/02/08			
	Hazard Communication	AECOM	10/05/05			
	Hazmat awareness	AECOM	10/19/06			
	Hazmat shipping	AECOM	1/10/08			
	First Aid/CPR	ARC	12/18/07			
Field Sampling Personnel	Hazmat awareness	AECOM	Various	Field Sampling Team	AECOM Field Technicians	AECOM St. Paul and Plymouth
	40 hour HAZWOPER	AECOM	Various			
	HAZWOPER 8-hr Refresher	AECOM	Various			

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

[a] Hazardous Waste Operations and Emergency Response

All AECOM and CSLP personnel working on the project will be properly trained and qualified individuals. Prior to commencement of work, personnel will be given instruction specific to this project, covering the following areas:

- Organization and lines of communication and authority
- Overview of the Site Management Plan and QAPP
- Documentation requirements
- Decontamination requirements
- Health and Safety considerations

Training of field personnel will be provided by the Field Coordinator or a qualified designee.

The analysts performing chemical analyses of samples will be trained in and will have exhibited proficiency in the analytical methods to be employed. AECOM, CSLP, and TA are responsible for maintaining documentation of training for their respective employees working on the project.

QAPP Worksheet #10 (UFP-QAPP Manual Section 2.5.2) Problem Definition and
QAPP Worksheet #11 (UFP-QAPP Manual Section 2.6.1) Project Quality Objectives/Systematic Planning Process Statements

Step 1 - The problem to be addressed by the project. Groundwater in five aquifers in the CSLP contains PAHs as a result of historical coal-tar distillation and wood-preserving activities at the Reilly site from 1917 to 1972. The source controls at the site are designed to limit the spread of the PAH plume and drinking water treatment is provided to ensure an adequate supply of drinking water. The concentration of PAH needs to monitored continually to ensure that the public water supply meets the PAH criteria outlined in the CD-RAP and that the PAH concentrations in groundwater are not increasing.

Step 2 – Identify the Decision. Groundwater and treated drinking water samples will be periodically collected and analyzed for PAH. The decision to made based on the data generated during these activities is whether the drinking water meets the PAH criteria and whether PAH is spreading in the aquifers.

Step 3 – Identify Inputs to the Decision. The groundwater monitoring network in St. Louis Park is well established based on monitoring since 1987. A series of wells in each aquifer as discussed in section A.6 are to provide sampled data for this program. The samples will be analyzed for PAH to evaluate contaminant concentrations.

Step 4 – Define the Boundaries of the Study. The spatial boundaries of the study are based on the plume geometry presented in the Annual Monitoring Reports. The spatial boundary varies between aquifers; however, the plume is generally contained within the CSLP city limits.

Step 5 – Develop a Decision Rule. Decisions affecting public health involve samples of municipal drinking water wells and comparing the results to the drinking water criteria outlined in the CD-RAP. If municipal drinking water sample exceed the Advisory Level for PAH, then it will be re-sampled according to the procedures described in the CD-RAP. Monitoring conducted since 1988 has revealed a stable PAH plume. For example, PAH concentrations at individual wells have shown little fluctuation over time. The drinking water wells have an established re-sampling action plan described in sections 4.3.2 and 12.1 of the CD-RAP. Groundwater sampling frequency is defined in Tables B-2 and B-3. Worksheet # 18

Step 6 – Specify Limits on Decision Errors. The only limitations are the data quality limitations based on the analytical methodologies used. Because of the re-sampling provisions outlined in the CD-RAP and because of the extensive historical data, the chance of a decision error based on sampling results is very low.

Step 7 – Optimize the Design for Obtaining Data. The wells to be sampled for this program were selected to be representative of each of the five aquifers being monitored. The sampling design has been developed to support the objective of comparing PAH concentrations to drinking water criteria and historic water quality results.

QAPP Worksheet #10 (UFP-QAPP Manual Section 2.5.2) Problem Definition and
QAPP Worksheet #11 (UFP-QAPP Manual Section 2.6.1) Project Quality Objectives/Systematic Planning Process Statements

AECOM works with CSLP to address issues concerning the CD-RAP that includes work plan development and implementation for various tasks, groundwater sampling, and compliance to the CD-RAP. Annual reports will include calculations of the sums of specific PAHs described below.

For active drinking water wells, TA will notify CSLP by telephone, within 24 hours of completing an analysis, whenever a sample analysis is shown to exceed the following Advisory Levels or Drinking Water Criterion:

Parameter	Advisory Level	Drinking Water Criterion
Sum of Benzo(a)pyrene and Dibenz(a,h)anthracene[1]	3.0 ng/L[1]	5.6 ng/L
Total Carcinogenic PAH[2]	15 ng/L[3]	28 ng/L[3]
Total Other PAH	175 ng/L	280 ng/L

[1] **Or the detection limit, whichever is largest**
[2] **See Table A-3**
[3] **Different concentrations for additional carcinogenic PAH may be established in accordance with the procedure specified in Part D.1 of the Consent Decree**

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

Matrix	Sediment				
Analytical Group[a]	PAHs and select SVOCs (HRGC/LRMS)				
Concentration Level	Low (part per billion)				
Sampling Procedure[b]	**Analytical Method/SOP**[c]	**DQIs**	**Measurement Performance Criteria**[d]	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
SOP 7130	DV-MS-0011	Accuracy/Bias-Contamination	No target compound >RL	Method Blank/Instrument Blank	A
	DV-MS-0011	Accuracy/Bias-Contamination	No target compound >RL	Equipment Rinsate Blanks	S & A
	DV-MS-0011	Accuracy/Bias	Compound-specific, see Appendix A	LCS	A
	DV-MS-0011	Accuracy/Bias	Compound-specific, see Appendix A	MS	S & A
	DV-MS-0011	Accuracy/Bias	Compound-specific, see Appendix A	Surrogates	A
	DV-MS-0011	Accuracy/Bias	Supplier Certified Limits	PE Sample	A
	DV-MS-0011	Precision	Compound-specific, see Appendix A	MSD	S & A
	DV-MS-0011	Precision	RPD ≤ 30% if both samples are > 5x RL	Field Duplicate	S & A
	DV-MS-0011	Completeness	≥ 90%	Data Completeness Check	S & A

[a] Refer to QAPP Worksheet #15 for a complete list of analytes for each analytical group
[b] Refer to QAPP Worksheet #21
[c] Refer to QAPP Worksheet #23
[d] Analyte specific limits may be found in Appendix A
[e] HRGC/LRMS: High Resolution Gas Chromatography/Low Resolution Mass Spectrometry – Selective Ion Monitoring

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

Matrix	Sediment				
Analytical Group[a]	PAHs (HRGC/LRMS – SIM)[e]				
Concentration Level	Low (part per trillion)				
Sampling Procedure[b]	**Analytical Method/SOP**[c]	**DQIs**	**Measurement Performance Criteria**[d]	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
SOP 7130	DV-MS-0005	Accuracy/Bias-Contamination	No target compound >RL, No carcinogenic PAH > MDL No non-carcinogenic PAH >5x MDL	Method Blank/Instrument Blank	A
	DV-MS-0005	Accuracy/Bias-Contamination	No target compound >RL, no common lab contaminants >5x RL	Equipment Rinsate Blanks	S & A
	DV-MS-0005	Accuracy/Bias	Compound-specific, see Appendix A	LCS	A
	DV-MS-0005	Accuracy/Bias	Compound-specific, see Appendix A	MS	S & A
	DV-MS-0005	Accuracy/Bias	Compound-specific, see Appendix A	Pre-extraction Internal Standards	A
	DV-MS-0005	Accuracy/Bias	Supplier Certified Limits	PE Sample	A
	DV-MS-0005	Precision	Compound-specific, see Appendix	MSD	S & A
	DV-MS-0005	Precision	RPD ≤ 30% if both samples are > 5x RL	Field Duplicate	S & A
	DV-MS-0005	Completeness	≥ 90%	Data Completeness Check	S & A

[a] Refer to QAPP Worksheet #15 for a complete list of analytes for each analytical group
[b] Refer to QAPP Worksheet #21
[c] Refer to QAPP Worksheet #23
[d] Analyte specific limits may be found in Appendix A
[e] HRGC/LRMS-SIM: High Resolution Gas Chromatography/Low Resolution Mass Spectrometry – Selective Ion Monitoring

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

The principal objectives of the QAPP pertain to the collection of data that are sufficient to monitor the effectiveness of the GAC treatment system and to detect changes in groundwater quality. The Data Quality Objectives for this project are defined primarily in Worksheets #10, 12, and the Appendix A. The quality of the data gathered in this project can be defined in terms of the following elements:

- Precision – Precision is the degree of agreement among repeated measurements of the same characteristic (analyte, parameter, etc.) under the same or similar conditions. Precise data will be achieved through the use of sampling and analytical procedures that minimize biases, through the use of standard procedures, through the meticulous calibration of analytical equipment and by implementing corrective action whenever measured accuracy and precision exceed pre-established limits.

  Field precision is assessed through the collection and measurement of field duplicates at a rate of one duplicate per 10 analytical samples (ten percent duplication), or Sample Delivery Group (SDG), whichever is less. Precision will be measured through the calculation of relative percent difference (RPD). The RPD is calculated as follows:

  where: RPD = relative percent difference
  D_1 = first sample value
  D_2 = second sample value (duplicate)

$$RPD = \frac{1 D_1 - D_2 1}{(D_1 + D_2)/2} \times 100$$

  Precision in the laboratory is assessed through the calculation of RPD for duplicate samples, either as matrix spike/matrix spike duplicates (MS/MSDs) or as laboratory duplicates, depending on the method. Precision control limits for laboratory analyses are discussed in Appendix A, Table A-1.

- Accuracy – Accuracy is the extent of agreement between an observed value (sampleresult) and the accepted, or true, value of the parameter being measured. The determination of the accuracy of a measurement requires knowledge of the true or accepted value for the signal being measured. Laboratory and method accuracy are calculated as a percentage using the following equation:

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

$$Accuracy = \frac{X}{T} x\,100$$

where: X = the observed value of measurement
T = "true" value

Laboratory accuracy is assessed through the analysis of MS/MSDs, laboratory control samples (LCSs), and surrogate compounds, and the subsequent determination of percent recoveries (%Rs). Accuracy control limits are given in Table A-3. The spike recovery is calculated in percentage using the following equation:

$$\%\,\mathrm{Re}\,\mathrm{cov}\,ery_Accuracy = \frac{SS - US}{C} x\,100$$

where: SS = Spiked Sample Concentration
US = Unspiked Sample Concentration
C = Spike Concentration added

- Representativeness – Representativeness is the extent to which reported analytical results truly depict the PAH concentration in the sampled environment. Representativeness is optimized through proper selection of sampling sites, times and procedures, through proper sample preservation, and through prompt extraction and analysis.

  It is essential that representative groundwater samples be retrieved for laboratory analyses. Accuracy and precision in the measurement of parameters used to monitor groundwater as it is purged from monitor wells and piezometers will be achieved through the use of standard monitoring procedures carried out continuously during the sample retrieval task. Field measurement equipment will be calibrated in accordance with the manufacturer's recommendations, and appropriate corrective action will be initiated whenever measured accuracy and precision do not meet pre-established limits. Precision and accuracy of field measurement devices will be tested by taking duplicate samples and calculating the relative percent difference using the formula presented above. Duplicate field readings will be completed at a 10 percent frequency.

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

- Comparability – Comparability is the extent to which comparisons among separate measurements will yield valid conclusions. Comparability among measurements in the monitoring program will be achieved through the use of rigorous standard sampling and analytical procedures outlined in the SOPs in Appendices B and C.
- Completeness – Completeness is assuring that a sufficient number of successful (valid) measurements have been collected to characterize the concentrations of PAH in the influent and effluent of the treatment system and in the aquifers of interest over a period of time. For this project, the completeness objective is that 95 percent of the laboratory analyses and 95 percent of the field measurements will produce valid data. Field data will be supplemented by re-sampling if necessary to ensure completeness.
- Sensitivity – Sensitivity is the ability of the method or instrument to detect the contaminant of concern at the level of interest. Determination of instrument sensitivity is accomplished by calibration using multiple concentrations of the analytes of interest. Once instrument sensitivity is demonstrated, analysis of replicate spiked samples of deionized reagent water at a concentration of one to five times the instrument sensitivity is used to determine method sensitivity (i.e. method detection limit).
- Traceability - the extent to which results can be substantiated by hard-copy documentation. Traceability documentation exists in two forms: that which links final numerical results to authoritative measurement standards, and that which explicitly describes the history of each sample from collection to analysis.

The fundamental mechanisms that will be employed to achieve these quality goals can be categorized as prevention, assessment and correction, as follows:

1. Prevention of defects in the quality through planning and design, documented instructions and procedures, and careful selection and training of skilled, qualified personnel.

2. Quality assessment through a program of regular audits and inspections to supplement continual informal review (refer to Section C.1 of this QAPP).

3. Permanent correction of conditions adverse to quality through a closed-loop corrective action system.

The CSLP sampling program QAPP has been prepared in direct response to these goals. The QAPP describes the quality assurance program to be implemented and the quality control procedures to be followed by TA during the course of laboratory analyses in support of the various site investigation studies for the CSLP Site. The Quality Assurance objectives will require the use of field blanks, method blanks, field duplicates, surrogate spikes, matrix spikes and matrix spike duplicates. The overall required frequency of these QC sample types is provided in the table below.

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

Objective	Frequency (%)
Field Duplicates	10
Field Blanks	10
Method Blanks	5
Surrogate Spikes	100
Matrix Spikes/Duplicates	5

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

Secondary Data	Data Source (Originating Organization, Report Title, and Date)	Data Generator(s) (Originating Org., Data Types, Data Generation/Collection Dates)	How Data Will Be Used	Limitations on Data Use
Most relevant site data has been collected and analyzed under the previous versions of this QAPP. This data is summarized in Annual Reports from 1988 to the present. Some historical data collected before the RAP was used to make decisions about well placement and RAP development.	AECOM USEPA CD_RAP,, September 4, 1986	TA-Denver laboratory (formerly STL-Denver, and Rocky Mountain Analytical)	Evaluation of ongoing site cleanup	None

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

Sampling and Analysis Tasks - The USEPA, MPCA, MDH, CSLP, and Reilly have agreed to acceptable drinking water quality criteria for PAH. QAPP Worksheet #15 lists the nominal reporting limits for the target compounds listed in the CD-RAP. Currently, only TA-Denver has conducted the laboratory analyses of groundwater samples at the site.

The list of chemicals of concern was negotiated as part of the Consent Decree. Based on the raw materials used and the products produced at the Reilly plant, mainly coal tar and creosote, PAH compounds and phenols were identified in the CD-RAP as compounds of concern. PAH has driven the risk associated with the Reilly site since 1978 when the Minnesota Department of Health developed a parts per trillion PAH analytical technique.

QAPP Worksheet #15 lists the 31 PAH compounds that all samples are analyzed for. Generally not all of these compounds are detected in every aquifer. The Drift and Platteville aquifer wells are analyzed for a parts per billion reporting limit. At this higher level, most of the compounds are not detected. The other aquifers are analyzed using the lower parts per trillion analyses. W23 is a Prairie du Chien-Jordan aquifer well located on the site. Historical concentrations for this well can be found in the Annual report in Table 4-1. Typical concentrations in wells off-site are summarized in the table in the Intorduction section of this QAPP.

Many of the wells sampled for this program were municipal drinking water wells or private/industrial production wells pre-dating the study of the site. Site specific studies have resulted in numerous monitoring wells and piezometers being installed in key down gradient locations at various depths.

In conjunction with the implementation of remedial measures to limit the spread of contaminants, a granular activated carbon (GAC) treatment system has been installed to treat water from City wells (identified - SLP) 10 and 15. Further provisions of the RAP call for long-term monitoring of the influent and effluent of the GAC treatment system and the major aquifers underlying the region. The general objective of the monitoring program is to identify the distribution of PAH in the groundwater and compare the analytical data with water quality criteria established in the CD-RAP. Currently, both CSLP and ENSR are collecting the groundwater samples. Typically, CSLP collects water samples from pumping wells (i.e. City owned wells) and ENSR collects water samples from non-pumping wells (i.e. monitoring wells). The specific objectives of the sampling and analysis program, and therefore, the intended end use of the data vary slightly for the different aquifers (Mt. Simon-Hinckley, Ironton-Galesville, Prairie du Chien-Jordan, St. Peter, and Drift-Platteville) monitored in accordance with the CD-RAP.

If any new Ironton-Galesville Aquifer drinking water wells are installed within one mile of well W23 (CD-RAP Section 6.2.1), then those wells will be sampled and analyzed for PAH to meet the objective of assuring protection of the wells from PAH resulting from the activities of Reilly at the Site. The analytical data would be used to compare the levels of PAH found in potential Ironton-Galesville Aquifer drinking water wells to the drinking water criteria established in the CD-RAP.

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

The objectives of monitoring the many Prairie du Chien-Jordan Aquifer wells, including municipal drinking water wells, private or industrial wells, and monitoring wells are to (CD-RAP Section 7.3): 1) monitor the distribution of PAH in the aquifer, thus evaluating the source and gradient control system, and 2) assure the continued protection of drinking water wells from PAH resulting from the activities of Reilly at the Site. The analytical data will be used to compare the levels of PAH in the Prairie du Chien-Jordan Aquifer to historical PAH data and to various criteria established in the CD-RAP (e.g., drinking water criteria for drinking water wells and a cessation criterion of 10 micrograms per liter of total PAH for source control well W23).In addition to water quality data generation, water level data will be used for the purpose of determining groundwater flow patterns in the Prairie du Chien-Jordan Aquifer.

The objectives of monitoring St. Peter Aquifer wells are to (CD-RAP Section 8.1.3): 1) monitor the distribution of PAH in the aquifer, thus evaluating a gradient control system installed at W410 in 1990, and 2) assure the continued protection of drinking water wells from PAH resulting from the activities of Reilly at the Site. Upon its receipt, analytical data will be used to compare the levels of PAH in the St. Peter Aquifer to historical PAH data, to drinking water cessation criteria for well W410 and to drinking water criteria established in the CD-RAP. Water level data will be used to evaluate groundwater patterns in the St. Peter Aquifer.

The objective of monitoring the Drift-Platteville Aquifer wells (CD-RAP Section 9.6) is to monitor the distribution of PAH in the aquifer, thus evaluating the source and gradient control systems. Groundwater analytical data will be used to compare levels of PAH in the Drift-Platteville Aquifer with historical water quality data for the aquifer and with various criteria established in the CD-RAP for PAH. Water level data will be used to evaluate groundwater flow patterns in the Drift-Platteville Aquifer.

In addition to the objectives for laboratory analytical data described above, field measurement data will be collected to aid in the groundwater sampling procedure. In accordance with MPCA Guidelines (January 1995), field measurements of temperature, pH, and specific conductance will be made for the purpose of determining that a sufficient volume of water has been purged from the well prior to sampling. The objective of those field measurements is to determine when three successive well volumes exhibiting equivalent temperature pH, and specific conductance have been purged from each monitoring well, so that representative samples may be collected.

Quality Control Tasks: QC samples have been defined for the field and laboratory efforts. Field QC samples are summarized on Worksheet #20; laboratory QC samples are summarized on Worksheet #28.

Secondary Data: All relevant secondary/historical data are summarized on Worksheet #13.

Data Management Tasks: The handling of records and data is summarized on Worksheet #29.N

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

Documentation and Records: Project related records (field, sample transfer/chain of custody, laboratory) are summarized on Worksheet #29.

Assessment/Audit Tasks: Field and laboratory audits are scheduled in accordance with Worksheet #31.

Data Review Tasks: Field data will be reviewed as described in Worksheet #34. Laboratories are contractually required to verify all laboratory data including electronic data deliverables (EDDs) as summarized in Worksheet #34. Data validation and usability assessments will be conducted as detailed in Worksheets #35, 36, and 37.

Management Responsibilities

This project is being conducted in accordance with the CD-RAP for the Reilly Tar & Chemical Corporation N.P.L Site in St. Louis Park, Minnesota. The parties to the Consent Decree include the Reilly Industries, Inc. (formerly Reilly Tar & Chemical Corporation - Reilly), City of St. Louis Park, Minnesota (CSLP), United States Environmental Protection Agency (EPA), Minnesota Pollution Control Agency (MPCA), and Minnesota Department of Health (MDH). In accordance with Section O of the Consent Decree, each party is required to identify a Project Leader. The current Project Leaders for the responsible parties are as follows; Mr. William Gregg (City), Mr. Nile Fellows (MPCA), Mr. Matthew Ohl (USEPA), Mr. Richard Clark (MDH), and Mr. John Jones (Vertellus). The responsibility for the overall QA/QC on the project is AECOM. Both CSLP and AECOM are responsible for the completion of the monitoring tasks described in the Sampling Plan and this QAPP. CSLP is assisted in the retrieval and laboratory analysis of water samples by AECOM and Test America Laboratories (TA), respectively. AECOM is responsible for the field sampling QA/QC and will perform the biennial audit of TA.

The EPA and MPCA are responsible for review and approval of the Sampling Plan, including the QAPP. In addition, laboratory and field audits may be completed by appropriate agency representatives. Responsibilities of the key positions in the EPA and MPCA are described below:

- EPA Project Manager (Mr. Matthew Ohl): The EPA Project Manager, EPA Region 5, is responsible for the review and approval of the QAPP on behalf of the EPA. Mr. Ohl is responsible for obtaining input, as needed from the EPA Quality Assurance Officer, and the EPA Central Regional Laboratory, EPA Region 5.
- MPCA Project Manager (Mr. Nile Fellows): The MPCA Project Manager shall be responsible for review and approval of the Sampling Plan, and review of laboratory and field procedures described in the QAPP.

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

QA Responsibilities

As discussed above, EPA and MPCA Project Managers (Messrs. Ohl and Fellows) are responsible for QAPP review and approval. The overall data validation and assessment program will be managed by AECOM's project manager Mr. William Gregg. The technical staff responsible for completing the validations and assessments is AECOM's Project QA Officer, Ms. Lori Herberich. The laboratory will also conduct data reviews prior to the release of any data. The TA staff responsible for reviewing the data is Ms. Karen Kuoppala, Quality Assurance Manager. CSLP and the Agencies are responsible for internal and external performance and system audits.

Field Responsibilities

AECOM and CSLP will be responsible for the coordination of all field sample collection under direction of the Field Coordinator (AECOM Project Manager). CSLP has contracted AECOM to assist in sampling. In general CSLP collects samples from municipal pumping wells, and AECOM collects samples from monitoring wells. The Sampling Team shall consist of employees of CSLP and AECOM. The team shall be responsible for sample collection, conducting field measurements (i.e. water level), and maintaining proper decontamination procedures stated in the QAPP. The person responsible for identifying and documenting nonconformance and subsequent corrective action is the AECOM Project Manager.

Lab Responsibilities

The TA analytical facilities in Arvada, Colorado, will be responsible for the coordination and completion of all laboratory groundwater and treatment water analyses for polynuclear aromatic hydrocarbons (PAH) and semi-volatile organic compounds. This facility is certified in Minnesota by MDH (MN certificate #008-999-342). Responsibilities of the key positions in the organization of TA are described below:

- Laboratory Project Manager: The Laboratory Project Manager is ultimately responsible for all laboratory analyses and is the primary point of contact for issues surrounding this QAPP, resolving technical problems, modifications to SOPs, etc. The Laboratory Project Manager is responsible for the coordination of routine day-to-day project activities including project initiative, status tracking, data review and requests, inquiries and general communication related to the project. Final approval of data packages is the responsibility of the Laboratory Project Manager.
- Operations Manager: The Operations Manager is responsible for oversight of all aspects of log in, preparation, and analysis of samples to ensure that project objectives, requirements and QA/QC criteria are met. The evaluation of data, as it is compiled and organized in accordance with the requirements of the QAPP, is the responsibility of the section supervisors who report to the Operations Manager.

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

Additional review, evaluation, and assessment of the data as it pertains to the QAPP is performed by an experienced second party reviewer, also reporting to the Operations Manager.

- Analyst: The Analyst is responsible for the analysis of water samples for the requested parameters utilizing the methods prescribed by the QAPP.
- Technician: The Technician is responsible for sample extraction. This requires practical experience and knowledge in the techniques of liquid - liquid solvent extraction, Kuderna - Danish evaporation, and the quantitative preparation of sample extracts for analysis.
- Quality Assurance Manager: The Quality Assurance Manager is responsible for overall quality control oversight, including internal audits and corrective actions. The Quality Assurance Manager supervises an independent QA/QC department and reports directly to the Laboratory Director and Corporate QA Director.
- Sample Control Group Leader: The Sample Control Group Leader is responsible for overseeing the sample control staff in sample receipt by the laboratory. This includes inspection of sample containers and chain of custody records, noting of errors, omissions, or out of control items, and ultimately logging samples into the laboratory database. The Sample Control Group Leader communicates with the Laboratory Project Manager and reports to the Operations Manager.

QAPP Worksheet #15 (UFP-QAPP Manual Section 2.8.1) Data Quality Levels and Analytical Method Evaluation

Matrix: Water
Analytical Group: PAHs, Method DV-MS-0005, HRGC/LRMS-SIM, TA, Denver, CO
Concentration Level: Low (part per trillion)

Analyte	CAS Number	Project RL Goal (ng/L) [a]	Analytical Method[b]		Achievable Laboratory Limits[c]	
			MDLs (ng/L)	Method RLs (ng/L)	MDLs (ng/L)	RLs (ng/L)
1H-Indene	95-13-6	4.7	NA	NA	0.66	4.7
1H-Indole	120-72-9	4.7	NA	NA	1.73	4.7
1-Methylnaphthalene	90-12-0	5.6	NA	NA	0.89	5.6
2,3-Benzofuran	271-89-6	5.4	NA	NA	0.54	5.4
2,3-Dihydroindene	496-11-7	5	NA	NA	0.61	5
3-Methylcholanthrene [g]	56-49-5	4.4	NA	NA	5.04	20
7,12-Dimethylbenz(a)anthracene [g]	57-97-6	2.8	NA	NA	2.27	2.8
Acenaphthene	83-32-9	5.7	NA	NA	0.80	5.7
Acenaphthylene	208-96-8	4.8	NA	NA	0.66	4.8
Acridine	260-94-6	6.2	NA	NA	6.50	6.2
Anthracene	120-12-7	4.2	NA	NA	0.80	4.2
Benzo(a)anthracene	56-55-3	4.3	NA	NA	0.92	4.3
Benzo(a)pyrene	50-32-8	2.5	NA	NA	1.24	2.5
Benzo(b)fluoranthene	205-99-2	4.7	NA	NA	1.39	4.7
Benzo(b)thiophene	4565-32-6	5.2	NA	NA	0.75	5.2
Benzo(c)phenanthrene[f g]	195-19-7	NA	NA	NA	NA	20
Benzo(e)pyrene	192-97-2	4.3	NA	NA	1.14	4.3
Benzo(g,h,i)perylene	191-24-2	6.2	NA	NA	1.17	6.2
Benzo(j)fluoranthene[e]	205-82-3	NA	NA	NA	NA	20
Benzo(k)fluoranthene	207-08-9	4.1	NA	NA	0.39	4.1
Biphenyl	92-52-4	5.6	NA	NA	1.05	5.6
Carbazole	86-74-8	3.8	NA	NA	0.99	3.8

QAPP Worksheet #15 (UFP-QAPP Manual Section 2.8.1) Data Quality Levels and Analytical Method Evaluation

Analyte	CAS Number	Project RL Goal (ng/L) [a]	Analytical Method[b]		Achievable Laboratory Limits[c]	
			MDLs (ng/L)	Method RLs (ng/L)	MDLs (ng/L)	RLs (ng/L)
Chrysene	218-01-9	5.6	NA	NA	1.24	5.6
Dibenz(a,c)anthracene[d g]	215-58-7	NA	NA	NA	NA	4.3
Dibenzo(a,e)pyrene[f g]	192-65-4	NA	NA	NA	1.69	20
Dibenzo(a,h)anthracene[d]	53-70-3	5.9	NA	NA	1.04	5.9
Dibenzo(a,h)pyrene[f g]	189-64-0	NA	NA	NA	NA	20
Dibenzo(a,i)pyrene[f g]	189-55-9	NA	NA	NA	5.63	20
Dibenzofuran	132-64-9	5.7	NA	NA	0.99	5.7
Dibenzothiophene	132-65-0	4.1	NA	NA	0.98	4.1
Fluoranthene	206-44-0	4.6	NA	NA	1.69	4.6
Fluorene	86-73-7	4.1	NA	NA	0.85	4.1
Indeno(1,2,3-cd)pyrene	193-39-5	5.4	NA	NA	1.26	5.4
Naphthalene	91-20-3	8.6	NA	NA	1.14	8.6
Perylene	198-55-0	3.3	NA	NA	3.81	3.3
Phenanthrene	85-01-8	6.3	NA	NA	3.21	6.3
Pyrene	129-00-0	4.2	NA	NA	0.99	4.2
Quinoline	91-22-5	9	NA	NA	5.65	9.0

[a] Project RL Goals are based on the achievable lab limits.

[b] Analytical MDLs and QLs are not available in validated reference methods for the HRGC/LRMS-SIM method.

[c] Achievable MDLs are updated annually by the laboratory. Values provided here were derived from the 11/20/07 MDL study performed by TA-Denver. RLs are project defined and not equivalent to the QL. All target analyte detections between the RL and MDL are reported by the laboratory as estimated values. Actual sample MDLs and RLs will vary based on sample volume and other sample-specific factors such as dilutions. MDLs studies are performed on an annual basis and MDL values may be updated annually.

[d] Dibenzo(a,h)anthracene and Dibenzo(a,c)anthracene coelute.

QAPP Worksheet #15 (UFP-QAPP Manual Section 2.8.1) Data Quality Levels and Analytical Method Evaluation

[e] Laboratory studies have shown that Benzo(j)fluoranthene will coelute with either benzo(b)fluoranthene or benzo(k)fluoranthene depending on the relative concentration of these two compounds in solution. Benzo(j)fluoranthene cannot be consistently separated by this method. Therefore, if present, it will be detected and reported as benzo(b) and/or benzo(k)fluoranthene.

[f] Analytical standards not consistently available.

[g] These “extended list” analytes are only reported on specific samples as defined in the SAP.

QAPP Worksheet #15 (UFP-QAPP Manual Section 2.8.1) Data Quality Levels and Analytical Method Evaluation

Matrix: Water
Analytical Group: PAHs, Method DV-MS-0011, HRGC/LRMS-full scan, TA, Denver, CO
Concentration Level: Low (part per billion)

Analyte	CAS Number	Project RL Goal (ug/L) [a]	Analytical Method[b]		Achievable Laboratory Limits[c]	
			MDLs (ug/L)	Method RLs (ug/L)	MDLs (ug/L)	RLs (ug/L)
1H-Indene	95-13-6	10	NA	NA	2.66	10
1H-Indole	120-72-9	10	NA	NA	1.30	10
1-Methylnaphthalene	90-12-0	10	NA	NA	3.36	10
2,3-Benzofuran	271-89-6	10	NA	NA	1.97	10
2,3-Dihydroindene	496-11-7	10	NA	NA	3.62	10
3-Methylcholanthrene [g]	56-49-5	10	NA	10	1.73	10
7,12-Dimethylbenz(a)anthracene [g]	57-97-6	10	NA	10	1.20	10
Acenaphthene	83-32-9	10	NA	10	2.46	10
Acenaphthylene	208-96-8	10	NA	10	2.29	10
Acridine	260-94-6	10	NA	NA	2.21	10
Anthracene	120-12-7	10	NA	10	0.51	10
Benzo(a)anthracene	56-55-3	10	NA	10	0.89	10
Benzo(a)pyrene	50-32-8	10	NA	10	0.56	10
Benzo(b)fluoranthene	205-99-2	10	NA	10	1.10	10
Benzo(b)thiophene	4565-32-6	10	NA	10	2.15	10
Benzo(c)phenanthrene [f g]	195-19-7	NA	NA	NA	NA	10
Benzo(e)pyrene	192-97-2	10	NA	NA	0.54	10
Benzo(g,h,i)perylene	191-24-2	10	NA	10	0.69	10
Benzo(j)fluoranthene[e]	205-82-3	NA	NA	NA	NA	10
Benzo(k)fluoranthene	207-08-9	10	NA	10	0.88	10
Biphenyl	92-52-4	10	NA	10	3.29	10
Carbazole	86-74-8	10	NA	NA	0.66	10

QAPP Worksheet #15 (UFP-QAPP Manual Section 2.8.1) Data Quality Levels and Analytical Method Evaluation

Analyte	CAS Number	Project RL Goal (ug/L) [a]	Analytical Method[b]		Achievable Laboratory Limits[c]	
			MDLs (ug/L)	Method RLs (ug/L)	MDLs (ug/L)	RLs (ug/L)
Chrysene	218-01-9	10	NA	10	0.88	10
Dibenz(a,c)anthracene[d g]	215-58-7	-	NA	NA	NA	10
Dibenzo(a,e)pyrene[f g]	192-65-4	NA	NA	10	0.67	10
Dibenzo(a,h)anthracene[d]	53-70-3	10	NA	10	0.75	10
Dibenzo(a,h)pyrene[f g]	189-64-0	NA	NA	NA	2.12	10
Dibenzo(a,i)pyrene[f g]	189-55-9	NA	NA	NA	1.00	10
Dibenzofuran	132-64-9	10	NA	10	2.05	10
Dibenzothiophene	132-65-0	10	NA	NA	0.74	10
Fluoranthene	206-44-0	10	NA	10	0.61	10
Fluorene	86-73-7	10	NA	10	1.35	10
Indeno(1,2,3-cd)pyrene	193-39-5	10	NA	10	0.72	10
Naphthalene	91-20-3	10	NA	10	2.95	10
Perylene	198-55-0	10	NA	NA	0.77	10
Phenanthrene	85-01-8	10	NA	10	0.64	10
Pyrene	129-00-0	10	NA	10	0.60	10
Quinoline	91-22-5	10	NA	NA	1.85	10
2-Methylphenol[g]	95-48-7	NA	NA	.10	0.98	10
4-Methylphenol[g]	106-44-5	NA	NA	10	0.25	10
2-Chlorophenol[g]	95-57-8	NA	NA	10	2.0	10
2-Nitrophenol[g]	88-75-5	NA	NA	10	0.39	10
2,4-Dimethylphenol[g]	105-67-9	NA	NA	10	0.58	10
2,4-Dichlorophenol[g]	120-83-2	NA	NA	10	0.64	10
4-Chloro-3-methylphenol[g]	59-50-7	NA	NA	20	0.90	10
2,4,6-Trichlorophenol[g]	88-06-2	NA	NA	10	0.29	10
2,4,5-Trichlorophenol[g]	95-95-4	NA	NA	10	0.45	50

QAPP Worksheet #15 (UFP-QAPP Manual Section 2.8.1) Data Quality Levels and Analytical Method Evaluation

Analyte	CAS Number	Project RL Goal (ug/L) [a]	Analytical Method[b]		Achievable Laboratory Limits[c]	
			MDLs (ug/L)	Method RLs (ug/L)	MDLs (ug/L)	RLs (ug/L)
2,4-Dinitrophenol [g]	51-28-5	NA	NA	50	10	50
4-Nitrophenol [g]	100-02-7	NA	NA	50	1.23	50
4,6-Dinitro-2-methylphenol [g]	534-52-1	NA	NA	50	4.0	50
Pentachlorophenol [g]	87-86-5	NA	NA	50	20	50

[a] Project RL Goals are based on the achievable lab limits.

[b] Analytical MDLs and QLs are not available in validated reference methods for the HRGC/LRMS-SIM method.

[C] Achievable MDLs are updated annually by the laboratory. Values provided here were derived from the 11/20/07 MDL study performed by TA-Denver. RLs are project defined and not equivalent to the QL. All target analyte detections between the RL and MDL are reported by the laboratory as estimated values. Actual sample MDLs and RLs will vary based on sample volume and other sample-specific factors.

[d] Dibenzo(a,h)anthracene and Dibenzo(a,c)anthracene coelute.

[e] Laboratory studies have shown that Benzo(j)fluoranthene will coelute with either benzo(b)fluoranthene or benzo(k)fluoranthene depending on the relative concentration of these two compounds in solution. Benzo(j)fluoranthene cannot be consistently separated by this method. Therefore, if present, it will be detected and reported as benzo(b) and/or benzo(k)fluoranthene.

[f] Analytical standards not consistently available.

[g] These “extended list” analytes are only reported on specific samples as defined in the SAP. Tentatively identified compounds (TICs) may be requested on the acid fraction when phenolic compounds are requested.

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

Activities	Organization	Dates (MM/DD/YY)		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
Project Status	AECOM	Monthly	Monthly	Progress report	
QAPP and SAP submission	AECOM	March each year	June each year	QAPP and SAP	June 15 each year
Collection of Samples and Submission for Analysis	AECOM	January each year	December each year	Sample submission to laboratories	At time of collection per SOP
Laboratory Analysis	TA-Denver	January each year	December each year	Analytical data to AECOM	Beginning at 30 days after collection. See Worksheet #30 for turnaround times.
Data Validation and Verification of Sediment Data	AECOM	January each year	March each year	Validated data with progress report	When completed.
Evaluation of Sample Data	AECOM	January each year	March each year	Annual Monitoring Report	January each year
Preparation and Delivery of Annual Report to USEPA/MCPA	AECOM	January each year	March each year	Annual Monitoring Report	March 15 each year

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

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

Samples will be collected by AECOM and CSLP personnel in accordance with the 1995 MPCA Ground-water Sampling Guidance Document.

The Sampling Plan outlines the scope of work to be performed in order to monitor the groundwater in the St. Louis Park, Minnesota, area in accordance with the CD-RAP and the Agencies' October 3 and 19, 2000, letters related to the Reilly N.P.L. Site. Included in this Plan are: 1) the identity of wells to be monitored, 2) the schedule for groundwater monitoring, and 3) a description of the procedures that will be used for sample collection, water level measurement, sample handling, sample analysis, and reporting. The overall sampling program is summarized in Worksheet# 18.

Groundwater Sampling and Water Level Measurements

Groundwater samples will be collected and water levels measured in accordance with the procedures outlined in this QAPP. The wells involved in the monitoring program include municipal and commercial wells, piezometers and groundwater monitoring wells as defined in Worksheet#18. Sampling procedures to accommodate the dimensions and configuration of each type of well are described below. Further details on well dimensions, water level measurements and sample acquisition strategies are given in the Site Management Plan.

The importance of proper sampling of wells cannot be over emphasized. Even though the well being sampled may be correctly located and constructed, special precautions must be taken to ensure that the sample taken from that well is representative of the groundwater at that location and that the sample is neither altered nor contaminated by the sampling and handling procedure. Sample collection will always proceed from the less contaminated sampling points to the monitoring points containing progressively higher concentration of PAH.

Sample Collection - Monitoring Wells and Piezometers

Because unanticipated or changed conditions may cause difficulty in purging the monitoring wells and piezometers, flexibility in the approach to the method of well purging is necessary. This QAPP proposes that the sampling team be given latitude in the selection of purge equipment necessary to complete the task (various pumping equipment and procedures that may be used for purging monitoring wells are described in SOP 7130 and MPCA's 1995 Guidelines). In all cases where no dedicated pump exists, samples will be retrieved using laboratory-cleaned, stainless steel or Teflon bailers as described below.

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

Worksheet#18 specifies that Prairie St. Peter Aquifer monitor well W24 be sampled. This well is equipped with a dedicated submersible pump and it will be the responsibility of the sampling team to determine if the pump is operable. In the event the dedicated pump within any individual well is operable, well purging and sample retrieval tasks will be completed with the aid of the pump in conformance with monitoring parameters established herein. In the event the dedicated pump within any individual well is inoperable, the pump will be removed, if possible, and purging/sampling procedures will be as established below.

Monitoring wells and piezometers not equipped with dedicated submersible pumps will be purged using a non-dedicated submersible pump, suction pump or bailer. During the purging of each well, temperature, pH and specific conductance of the purge water will be monitored using a Quanta Hydrolab water quality monitor (or equivalent). Readings will be taken once per well volume. Stabilization of these readings will indicate that purging is complete and sampling may commence. Upon completion of well purging, samples will be collected from each well using a stainless steel or Teflon bailer and a new length of nylon or polyester rope. All non-dedicated purging and sampling equipment will be decontaminated before use and between sampling points. Equipment blanks will be collected at the frequency of one for each 10 samples collected from wells that have non-dedicated sampling equipment.

Samples will be collected by filling each of the appropriate sample containers in rapid succession, without pre-rinsing the containers with sample. The bottle will be held under the sample stream without allowing the mouth of the bottle to come in contact with the bailer and filled completely, and the cap securely tightened. All sample labels will be checked for completeness, sample custody forms completed and a description of the sampling event recorded in the field notebook.

Sample Collection - Pumping Wells

At active pumping wells, the sampling team will first determine that the wells have actually been pumping during the period preceding sampling. This information may be derived from inspecting flow recorders or from interviewing knowledgeable persons regarding the wells (water department employees, well owners, etc.). The information will be documented in the field notes of the sampling team. Water level measurements will then be made, if practical. The normal operation of the well will not be interrupted for the purpose of measuring water levels. A clean electric tape will be used to measure water levels in pumping wells. Sampling will proceed by filling the required containers with water from the sampling tap as near to the well head as possible, and before any holding tanks or treatment is encountered.

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

If it cannot be determined that a well has been pumping at some time during the 24-hour period preceding sampling, or if it is known the well was not pumping, then the well shall be purged until field measurements of temperature, pH, and specific conductance have stabilized after at least three well volumes have been removed from the well. These measurements, water levels, and the amount of water pumped will be recorded in the field notes.

Sampling Procedures - GAC Treatment System

Sampling points will be flushed for at least five minutes before collecting a sample. Each PAH sample and matrix spike sample will be collected in six 1-liter amber glass bottles, which should be filled and capped in succession. PAH sample bottles will not be rinsed before being filled.

The GAC treated water samples will have to be collected from two sample taps, one for each column (see Figure B-1). This will be done by filling three 1-liter bottle from the first column sample tap and then three more bottles from the second (three from each for duplicate samples). No notations distinguishing the two taps will be made on the labels.

Field blank samples will be prepared by transferring contaminant-free deionized water provided by TA into sample bottles in a fashion as closely similar to actual sample collection as possible. Field blank sample bottles will be filled and capped in succession with individual bottles open to the atmosphere for an equal time as for actual process samples. Field blanks will be prepared in the area in which GAC treated water samples are collected.

Field duplicate and matrix spike duplicate samples will be obtained by filling six 1-liter bottles at the sampling points by the procedure described above and assigning a different sample number to each of the resulting two bottle samples.

Chain-of-custody forms will be completed and all samples collected for this program will be packed, cooled to a temperature less than 4°C, and shipped overnight to TA on the day they are collected.

The sampling team must recognize that great care is required to collect samples for part per trillion level PAH analyses that are free from outside contamination. PAH compounds are present in cigarette smoke, engine exhaust and many petroleum derived oils, among other sources. There will be no smoking anywhere in the GAC treatment building for at least 72 hours prior to the day on which PAH samples are to be collected. Similarly, no vehicles will enter the GAC treatment building and the large access door will stay closed for at least 72 hours prior to the sampling day. Disposable gloves will be worn when collecting, handling and packaging samples. Sample bottles will remain in closed shipping coolers until they are needed, and will be packaged and sealed for shipment as soon as possible after sampling.

QAPP Worksheet #18 (UFP-QAPP Manual Section 3.1.1) Sampling Locations and Monitoring Schedules Tables

Sampling Plan GAC Treatment System Monitoring Schedule[a]				
RAP Section	**Sampling Points**	**Start of Monitoring**	**Sampling Frequency**	**Analyses[b]**
4.3.1(C)	Treated water (TRTD)	Date of plan approval	Quarterly	PAH (ppt)[c]
4.3.3(D)	Feed water (FEED)	Date of plan approval	Annually	PAH (ppt)[c]
4.3.4	Treated water	Date of plan approval	Annually	Extended PAH (ppt)[c]
4.3.4	Treated or Feed water	Date of plan approval	Annually	Acid fraction compounds (ppb)[c]

[a] This schedule does not include certain contingencies (e.g. exceedance monitoring) and, therefore, represents the minimum program that is likely to occur between the date this Plan is approved and December 31, 2009. Sections 4 and 12 of the CD-RAP outline the additional monitoring that will be conducted if PAH criteria are exceeded. The first samples will be collected during the period indicated by the monitoring frequency following the date of the start of monitoring.

[b] Lists of parameters and methods for analysis of PAH, extended PAH, and acid fraction compounds are provided in Worksheet#15 and Appendix A. Field blanks will be collected and analyzed at a frequency of one every 10 samples or fewer. Treated and Feed water duplicate samples will be collected and analyzed at a frequency of one per 10 samples.

c ppt = parts per trillion (ng/L). ppb = parts per billion (ug/L)

QAPP Worksheet #18 (UFP-QAPP Manual Section 3.1.1) Sampling Locations and Monitoring Schedules Tables

Sampling Plan Groundwater Monitoring Schedule[a]					
Source of Water	**CD-RAP References**	**Sampling[b] Points**	**Start of Monitoring**	**Sampling Frequency**	**Analyses[c]**
Mt. Simon-Hinckley Aquifer	5.1	SLP11, SLP12, SLP13, SLP17	Date of plan approval	Annually	PAH (ppt) [d]
	5.3.2	New municipal wells within one mile of well W23	At the time of installation	Annually	PAH (ppt)
Ironton-Galesville Aquifer	6.1.4	W105	Date of plan approval	Every even numbered year [e]	PAH (ppt)
	6.2.1	New municipal wells within one mile of well W23	At the time of installation	Annually	PAH (ppt)
Prairie du Chien-Jordan Aquifer	Table 1 [f]	SLP6, W48, W119 [g], W413 [g]	Date of plan approval	Quarterly	PAH (ppt)
	Table 1	SLP4, SLP10 or SLP15, W23, W29, W40, W70, W401, W402, W403, E2, E3, E7, E13, E15	Date of plan approval	Annually	PAH (ppt)
	Table 1	E4, SLP5, SLP8, W32	Date of plan approval	Semi-annually	Water level monitoring [h]
	Table 1	H6, MTKA6, SLP7 or SLP9 [g], SLP14, SLP16, W405 or W406 [i]	Date of plan approval	Every even numbered year	PAH (ppt)
St. Peter Aquifer	Table 2 [f]	SLP3, W24, W33, W122, W133, W410, W411, W412	Date of plan approval	Semi-annually	PAH (ppt)
	Table 2	W409	Date of plan approval	Semi-annually	PAH (ppb) [d]
	Table 2	P116, W129, W408	Date of plan approval	Semi-annually	Water level monitoring

QAPP Worksheet #18 (UFP-QAPP Manual Section 3.1.1) Sampling Locations and Monitoring Schedules Tables

Sampling Plan Groundwater Monitoring Schedule[a]					
Source of Water	**CD-RAP References**	**Sampling[b] Points**	**Start of Monitoring**	**Sampling Frequency**	**Analyses[c]**
Platteville Aquifer	9.2.3	W421	Date of plan approval	Quarterly	PAH (ppb)
	Table 2	W20, W27, W101, W131, W143, W426, W428, W431, W433, W120, W434, W437, W438	Date of plan approval	Semi-annually	PAH (ppb)
	Table 2	W1, W18, W19, W100, W121, W124, W130, W424	Date of plan approval	Semi-annually	Water level monitoring
Drift Aquifer	9.1.3	W420	Date of plan approval	Quarterly	PAH (ppb)
	Table 2	P109, P112, P307, P308, P309, P310, P312, W117, W128, W136, W422, W427, W439	Date of plan approval	Semi-annually	PAH (ppb)
	Table 2	P47, W2, W10, W15, W116, W135	Date of plan approval	Semi-annually	Water level monitoring

a This schedule does not include certain contingencies (e.g. exceedance monitoring) and, therefore, represents the minimum program that is likely to occur between the date this Plan is approved and December 31, 2009. Section 12 of the CD-RAP outlines the additional sampling that will be conducted if the drinking water criteria are exceeded in samples from water supply wells. The first samples will be collected during the period indicated by the monitoring frequency following the date of the start of monitoring..

b Sampling points are located on the maps shown in the . Letter prefixes to well codes are defined as follows:

- W 4-inch monitoring well
- P Monitoring piezometer
- SLP St. Louis Park supply well
- E Edina supply well
- H Hopkins supply well
- MTK Minnetonka supply well

c Lists of parameters and descriptions of the methods for analysis of PAH and expanded analyses are provided in the Worksheet#15 and Appendix A. Water levels will be measured each time samples are collected for analyses, except for those wells which prove to be inaccessible for such measurements.

d ppt = parts per trillion (ng/L). ppb = parts per billion (ug/L)

QAPP Worksheet #18 (UFP-QAPP Manual Section 3.1.1) Sampling Locations and Monitoring Schedules Tables

e Wells sampled every two years shall alternate the season the well is sampled. For example, SLP14 will be sampled in spring 2006, fall 2008, spring 2010, etc.

f Wells will be sampled in accordance with Table 2 from the Agencies' October 3, 2000, letter, or Table 1 from the Agencies' October 19, 2000, letter, in lieu of the requirements given in the Consent Decree. Wells W19 and W130 were listed in the Agencies' Table 2 for both the Drift and Platteville Aquifers. Wells W19 and W130 are Platteville Aquifer wells.

g Well unavailable for groundwater monitoring. Wells have been destroyed, removed, or are not in service.

h Water levels will be measured semi-annually at these wells in addition to the sampled wells, except for those wells that prove to be inaccessible for such measurements.

W405 = American Hardware Mutual, W406 = Minikahda Golf Course.

QAPP Worksheet #18 (UFP-QAPP Manual Section 3.1.1) Sampling Locations and Monitoring Schedules Tables

Matrix	Analytical Group	Concentration Level	Analytical and Preparation Method/SOP Reference[a]	Sample Size[b]	Containers (number, size, and type)	Preservation Requirements	Maximum Holding Time[c] (preparation/ analysis)
Groundwater	PAHs	Low (ppt)	DEN-MS-0005	6 liters	6 1-liter amber glass bottles, Teflon-lined caps	0-6°C; store in the dark	7 calendar days to preparation; 40 calendar days from preparation to analysis
Groundwater	PAHs and phenolics	Low (ppb)	DEN-MS-0011 DV-OP-0008	2 liters	2 1-liter amber glass bottles, Teflon-lined caps	0-6°C; store in the dark	14 calendar days to preparation; 40 calendar days from preparation to analysis

[a] Refer to Worksheet #23 for SOP titles.

[b] Sample size is the minimum requested by the laboratory to perform the requested analysis. Triple the minimum sample volume is needed for samples designated for MS/MSD analysis.

[c] Samples will be analyzed as soon as possible after sample receipt at the laboratory. The holding times listed are the maximum times that samples may be held before extraction.

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

Example Summary of 2008 Sampling and Analytical Program[1]								
Sample Matrix	**Field Parameter**	**Laboratory Parameters**	**Estimated Number of Samples**	**Field Blanks**	**Field Duplicates**	**Matrix Spike[1]**	**Matrix Spike Duplicate[1]**	**Matrix Total**
GAC Treated Water	None	PAH (ppt)	8	4	8	4	4	28
		Acid Fraction compounds[2]	2	2	1	1	1	7
GAC Feed Water	None	PAH (ppt)	2	2	1	1	1	7
Groundwater	pH, temperature, Specific Conductance	PAH (ppt)	46	9	9	9	9	82
		PAH (ppb)	58	6	6	6	6	82

[1] Annual tables for field quality control sample summary information will be provided in the specific Sampling and Analysis Plans.

[2] Matrix spike sample/matrix spike duplicate sample shall consist of the same matrix being analyzed. Triple the normal volume when related matrix spike/matrix spike duplicate samples are to be retrieved.

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

The following is a list of all Standard Operating Procedures (SOPs) associated with project sampling including, but not limited to, sample collection, sample preservation, equipment cleaning and decontamination, equipment testing, inspection and maintenance, supply inspection and acceptance, and sample handling and custody.

Reference Number	Title, Revision Date and/or Number	Originating Organization	Equipment Type	Modified for Project Work? (Y/N)	Comments
SOP 7130	Groundwater Sample Collection from Monitoring Wells / Rev. 2 Nov.1999	AECOM	Various – see Appendix B	No	Appendix B
SOP 7510	Packaging and Shipment of Environmental Samples / Rev. 4 4th Qtr. 1999	AECOM	Various – see Appendix B	No	Appendix B
SOP 1007	Chain-of-Custody Procedures / Rev.2 Feb 2008	AECOM	Various – see Appendix B	No	Appendix B
SOP 7121	Field and Laboratory Measurement of pH / Rev.0 July 1998	AECOM	Various pH meters	No	Appendix B
SOP 7123	Field and Laboratory Measurement of Temperature / Rev.0 July 1998	AECOM	Various thermometers	No	Appendix B
SOP 7124	Field and Laboratory Measurement of Specific Conductance / Rev.0 July 1998	AECOM	Various EC meters	No	Appendix B
SOP 7500	Decontamination of Field Equipment / Rev.4 Q4 1994	AECOM	Various – see Appendix B	Yes	See modification details below
SOP 7721	Water Level Measurements / Rev.0 Feb.2008	AECOM	Various – see Appendix B	No	Appendix B

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

The field decontamination procedure to be used on sampling equipment, which comes into contact with groundwater samples, is as follows:

- Disassemble equipment, if applicable
- Wash with Alconox and potable water
- Rinse with potable water

Sampling equipment, such as pumps, are decontaminated prior to sampling each well. The laboratory decontamination procedure to be used on sampling equipment, such as bailers, which comes into contact with groundwater samples, is as follows:

- Disassemble equipment
- Rinse with methanol
- Scrub with hot soapy water
- Rinse three times with hot deionized water
- Set on aluminum foil, dull side up, air dry
- Bake for one hour at 200°C
- Wrap with aluminum foil, dull side in

Pre-cleaned 1-liter amber bottles are supplied by TA for sample containerization. In order to minimize the possibility of sample destruction during shipment, six 1-liter bottles will be taken for all ppt samples. For all samples, field blanks and matrix spikes and duplicates, subsequent extraction and analysis will be conducted on four intact 1-liter bottles. All field blank duplicates will be extracted and held. In the event that the field blank is lost during analysis or invalidated, the duplicate field blank will be analyzed and reported. Additional sample matrix will be required for matrix spike analyses.

If less than four liters of a sample remains after shipment and storage for analysis, the Program Manager will be notified and another sample will be collected and shipped to the laboratory for analysis. The analysis report for the sample batch containing the affected sample will clearly note in the discussion section that a replacement sample was taken.

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

If a sample extract is broken or lost during analysis, the Program Manager will be notified and will be responsible for determining the need for replacing the lost sample. The analysis report for the sample batch containing the affected sample will clearly note in the discussion section the action taken.

If a method blank, or matrix spike and its duplicate is lost or broken during analysis, a replacement QC sample will be sampled and analyzed. The analysis report will clearly note that a replacement QC sample was analyzed.

If a field blank is lost or broken during shipment, storage, or analysis, its duplicate will be analyzed. The analysis report for the sample batch associated with the field blank will clearly note the occurrence in the discussion section.

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

Parameter	Device	Calibration	Routine Check		Acceptance Criteria	Responsible Person	SOP Reference[1]
			Technique	Frequency			
pH	Hydrolab, Quanta Water Quality Meter or equivalent	Standardize in three or more standard buffer solutions, or use the auto calibration with factory solution	Calibration check-analyze standard buffer solution	1/10 samples	±0.1 pH units	Field Coordinator or designee	7121
			Analyze duplicates	1/10 samples	±0.1 pH units		
			Auto calibration	1/10 samples	±0.1 pH units		
Specific Conductance	Hydrolab, Quanta Water Quality Meter or equivalent	Standardize using two or more KCL solutions or use the auto calibration with factory solution	Calibration check-analyze standard KCL solution	1/10 samples	±1 percent of range	Field Coordinator or designee	7124
			Analyze duplicates	1/10 samples	±1 percent of range		
			Auto calibration	1/10 samples	±1 percent of range		
Temperature	Hydrolab, Quanta Water Quality Meter or equivalent	Factory calibrated	Not required	Not required	±0.1°C	Field Coordinator or designee	7123
Depth to Water	Water Level Measurement Device (Electric)	Factory calibrated	Not required	Not required	±0.01 Ft.	Field Coordinator or designee	7721

[1]Refer to the Project Sampling SOP References table (Worksheet #21).

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

Reference Number[b]	Title, Revision Date, and/or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
DV-MS-0005	Extended List Polynuclear Aromatic Hydrocarbons by GC/MS Selective Ion Monitoring for CSLP with an attachment for Analysis by GC/MS Full Scan, Rev.7, 2/29/2008	Definitive	Organics (PAHs – ppt level)	GC/MS - SIM	TA-Denver,CO	Y, project specific modifications of EPA Method 8270 to improve sensitivity
DV-MS-0011	GC/MS Analysis Based on Methods 8270C and 625, Rev.5, 3/20/2008	Definitive	Organics (SVOCs – ppb level)	GC/MS – full scan	TA-Denver,CO	N
DV-OP-0008	Extraction of Aqueous Samples by Continuous Liquid Liquid Extraction (CLLE) by Method SW846 3520C and Methods 625 and 607, Rev.3, 3/13/2008	Definitive	Organics (SVOCs)	CLLE	TA-Denver,CO	Y, modifications described in DV-MS-0005 Appx.II

[a] All SOPs are contained in Appendix B.

[b] It is expected that the procedures outlined in these SOPs will be followed. Procedural modifications to individual SOPs may be warranted depending upon an individual sample matrix, interferences encountered, or limitations imposed by the procedure. Deviations from individual SOPs will be documented in the laboratory records. Substantive modification to any SOP will be approved in advance by the AECOM PM and Project QA Officer and communicated to the MPCA Project Leader and to the USEPA Remedial Project Manager. The ultimate procedure employed will be documented in the report summarizing the results of the sampling event or field activity.

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[a]
GC/MS (SVOC-full scan)	Decafluorotriphenylphosphine (DFTPP) tune; Initial and Continuing Calibration as required in SOP	Verify tune every 12 hours; Initial calibration after instrument set up, after major instrument changes and when continuing calibration criteria are not met.	Initial Calibration %RSD ≤30% for CCCs; ICAL %RSD ≤15% or linear curve r ≥ 0.995, or quadratic curve r^2 ≥0.990 ; SPCC minimum avg. RF. ≥0.05 CCV %D ≤20% for CCCs, <35% others; SPCC minimum avg. RF. ≥0.05	Inspect system, correct problem, rerun calibration and affected samples	Analyst	DV-MS-0011
GC/MS (SVOC-SIM)	Autotune w/FC43; Initial and Continuing Calibration as required in SOP	Verify tune every 12 hours; Initial calibration after instrument set up, after major maintenance, and/or instrument changes have occurred	Initial Calibration %RSD ≤30%[b] CCV %D ≤35%. [b]	Inspect system, correct problem, rerun calibration and affected samples	Analyst	DV-MS-0005

[a] Refer to the Analytical SOP References table (Worksheet #23). All laboratory analytical SOPs are contained in Appendix C.

[b] Exceptions to this requirement are allowed for 7H-dibenzo(c,g)carbazole, 3-methylcholanthrene, and the dibenzopyrene isomers due to poor response.

QAPP Worksheet #25 (UFP-QAPP Manual Section 3.2.2) Analytical Instrument and Equipment Maintenance, Testing, and Inspection Tables

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[a]
GC/MS (SVOC-SIM and full scan)	Change septa, clean injectors, change or trim columns, install new liners	Detector signals and chromatogram review	Instrument performance and sensitivity	Daily or as needed	See SOP	See SOP	Analyst or Section Supervisor	DV-MS-0011 DV-MS-0005
GC/MS (SVOC-SIM and full scan)	Clean sources and quadrupole rods; maintain vacuum pumps	Tuning	Instrument performance and sensitivity	Service vacuum pumps twice per year; other maintenance as needed	See SOP	See SOP	Analyst or Section Supervisor	DV-MS-0011 DV-MS-0005

[a] Refer to the Analytical SOP References table (Worksheet #23). All laboratory analytical SOPs are contained in Appendix C.

Inspection and Acceptance Requirements for Supplies/Sample Containers

This section documents the activities that will be performed to ensure that all sampling supplies and containers are free of contaminants of concern. For PAH, 1-liter amber glass bottles will be used (Table B-5). Caps will be fitted with pre-cleaned Teflon liners. Six bottles are required for each low-level PAH sample collected and two bottles for each PAH ppb and Extended Analysis sample collected. An independent commercial firm shall provide pre-cleaned bottles to TA for use on this project.

In the event the laboratory is required to prepare bottles for sampling, the bottles will be prepared as follows:

1. Wash bottles with hot detergent water.
2. Rinse thoroughly with tap water followed by three or more rinses with organic-free water.
3. Rinse with Burdick & Jackson quality redistilled acetone, followed by equivalent quality methylene chloride.
4. Allow to air dry in a contaminant-free area.

QAPP Worksheet #25 (UFP-QAPP Manual Section 3.2.2) Analytical Instrument and Equipment Maintenance, Testing, and Inspection Tables

5. Caps and liners must be washed and rinsed also. Bottles should be stored and shipped with the Teflon-lined caps securely fastened.
6. Vendor certified clean sample bottles can be used in place of above cleaning procedures.

TA provides adequate sampling supplies (coolers, containers, packaging supplies, etc.) that are stored by CSLP at Water Treatment Plan Number 1. Supplies are ordered as needed, by the Field Sampling Team members. TA is responsible for ensuring the cleanliness of the sampling supplies, and the Field Sampling Team is responsible for using only clean containers and supplies.

The TA Denver procurement policy is rigidly controlled to ensure the quality of supplies and services received. Calibration standards specifically are to be received with a certification report, and are traceable to the National Institute of Standards and Technology (NIST) or an equivalent source where available. If unavailable, standards are tested for accuracy prior to use. Information regarding procurement of supplies, including calibration standards, is in the laboratory Quality Assurance Manual (QAM), Section 5.0 titled Procurement of Supplies and Services.

Special procedures are performed to ensure that supplies used in the field and laboratory are available when needed and are free of contaminants. Laboratory supplies, such as reagents, are evaluated for purity and cleanliness by the specifications outlined in Test America SOP ST-001, “Testing of Solvents and Acids”. A forecast of the sample load is completely weekly by the TA to insure adequate quantities of supplies are available. Orders of supplies are made on a weekly basis and extra supplies are kept in stock in case of unanticipated sample loads.

A forecast of the field sampling supplies is made prior to the spring and fall sampling events. The supplies are ordered and checked before the sampling begins. The qualities of supplies are evaluated on a weekly basis throughout the sampling events. Sampling supplies used in the field include the following:

QAPP Worksheet #25 (UFP-QAPP Manual Section 3.2.2) Analytical Instrument and Equipment Maintenance, Testing, and Inspection Tables

Item	Cleanliness Check	Person Responsible for Checking Items and Corrective Action	Vendor[1]
Bailers	Equipment Blank Sample	William Gregg, AECOM	MVTL, Inc. Minneapolis, MN (Autoclaves Stainless Steel Bailers)
Sample Jars	I-Chem analytical analysis	Gail DeRuzzo, TA	I-Chem
Nitrile Gloves	Visual	William Gregg, AECOM	AirGas Safety Bristol, PA

[1] Alternate vendors may be used if necessary

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

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): AECOM Field Team (see Worksheet 21 for a list of the sample collection methods)
Sample Packaging (Personnel/Organization): AECOM Field Team
Coordination of Shipment (Personnel/Organization): AECOM and CSLP Field Teams
Type of Shipment/Carrier: UPS or FedEx for overnight delivery.
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Assigned laboratory personnel (see Worksheet 30 for laboratory providing analytical services)
Sample Custody and Storage (Personnel/Organization): Assigned laboratory personnel (see Worksheet 30 for laboratory providing analytical services)
Sample Preparation (Personnel/Organization): Assigned laboratory personnel (see Worksheet 30 for laboratory providing analytical services)
Sample Determinative Analysis (Personnel/Organization): Assigned laboratory personnel (see Worksheet 30 for laboratory providing analytical services)
SAMPLE ARCHIVING
Samples will not be stored in the field but will be shipped to the designated laboratory the same day as collection or no later than the day after collection. If circumstances require that the samples be stored in the field, they will be maintained under the method-specified conditions (e.g., kept at 4±2° C).
Sample Extract/Digestate Storage (No. of days from extraction/digestion): Sample extraction and digestion holding times are summarized in Worksheet 19.
SAMPLE DISPOSAL
Personnel/Organization: Assigned laboratory personnel (see Worksheet 30 for laboratory providing analytical services).
Number of Days from Analysis: Laboratory is required to give AECOM 30 days notice prior to intent to discard any project samples.

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

Sample Handling and Custody

The St. Louis Park Groundwater Study is a cooperative effort between CSLP and AECOM, whose responsibilities include sample retrieval, and TA, whose responsibilities include sample analysis. Proper sample handling and analysis is essential to the success of the study, therefore a formal sample custody procedure has been developed to insure the integrity of all samples. Sample labels shall be completed for each sample using waterproof ink. The information recorded on the sample label includes:

Sample Number - Unique coded sample identification number as described below.

Date and Time – The date of collection and the 4-digit number indicating the military time of collection.

Sampler - Initials of person collecting the sample.

Remarks - Any pertinent observations or further sample description.

The sample number includes three parts (source code, sampling point code, and date code) in the following sequence:

YYYYY-ZZZZZZ

Where:

YYYYY = Sampling Point Code (ex: SLP6 = Monitoring Well SLP6)

ZZZZZZ = Date Code (ex: 053099 = May 30, 1999)

Those samples which will be taken in accordance with this QAPP for quality control purposes will be identified by appending to the sampling point codes the following:

Field blank = FB

Field duplicate = D

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

Matrix spike = MS

Matrix spike duplicate = MSD

As an example, a field blank sample taken from sampling point SLP11 on September 21, 2008, would be identified as follows:

SLP11FB-092108

During the sampling event, one sample will be taken per sampling point unless it is duplicated. Duplicate samples will be collected as specified in Worksheet# 20. Those samples collected for matrix spike analysis will be selected at the time of sampling and labeled in the field.

After collection, identification, and preservation, the sample will be maintained under chain-of-custody procedures discussed below.

Packaging and shipment of samples shall be in accordance with SOP 7510 (Appendix B). The samples will be iced or refrigerated at 4°C from the time of collection until extraction. PAHs Nare known to be light sensitive; therefore, samples will be stored in amber bottles and kept away from prolonged exposure to light. All samples for GC/MS analysis will be extracted within seven days of collection. The analysis will be completed within 40 days following extraction.

Samples will be protected from breakage and shipped in coolers at a temperature of 4°C ± 2°C. An overnight carrier will be selected to insure delivery at the laboratory within 24 to 36 hours after collection. Samples received at the laboratory will be checked for leakage and a notation made regarding sample temperature at time of receipt. All samples should be stored in an organic-free refrigerator at 4°C ± 2°C.

Worksheet# 19 outlines the required sample volumes, container types, number of containers, preservation procedures, and holding time for each analytical parameter.

Sample custody procedures ensure the timely, correct, and complete analysis of each samples 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

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

- Is in his/her possession and has been placed in a secured location
- Is in a designated secure area

Sample custody documentation provides a written record of sample collection and analysis. The sample custody procedures require the specific identification of samples associated with an exact location and the recording of pertinent information associated with the sample, including time of collection and any preservation techniques, and a COC record which serves as physical evidence of sample custody. Custody procedures will be similar to the procedures outlined in 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 for three areas: sample collection, laboratory analysis, and final evidence files, which are described in Worksheet 27.

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 in Worksheet 27 are designed to ensure that the samples arrive at the laboratory with the COC intact. Specific preservation procedures required for each analytical method are described in Worksheet 19.

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): The field sample custody procedures including sample packing, shipment, and delivery requirements, are discussed in Worksheets 17 and 26. Sample management information is also provided in SOPs 7510, 1007 and 7130.

Laboratory Sample Custody Procedures (receipt of samples, archiving, disposal): The laboratory has 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, document sample condition upon receipt at the laboratory and will sign and date all appropriate receiving documents. Additional information on laboratory sample receiving procedures is provided in the text below this summary table.

Sample Identification Procedures: Each sample will be assigned a unique sample identification number using the rulesas discussed above. This identification nomenclature will consist of a unique alphanumeric code that identifies the sample location and date of collection.

Chain-of-Custody Procedures: A chain-of-custody will accompany all samples from the time of sampling through all custody transfers. Samples of the COC form are provided in SOP 7510 and 1007; the COC procedures are summarized below and in SOP 7510 and SOP 1007 provided in Appendix B.

Chain of Custody Procedure

The COC form serves as an official communication to the laboratory detailing the specific analyses required for each sample. The COC record is prepared by the field sample custodian and accompanies samples from the time of sampling through all transfers of custody. The COC will be retained by the laboratory which analyzes and archives the samples. Three copies of the COC are created; one copy is retained in the field and two copies are sent to the laboratory.

Transfer of Custody and Shipment

Sample custody must be maintained form the time of sampling through shipment and receipt at the laboratory. The procedures for custody transfer are outlined in SOP 7510 and SOP 1007(included in Appendix B).

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

Sample Packaging and Shipping Requirements

Sample custody must be maintained through shipment of samples to the contracted laboratory. All samples will be packaged and shipped at the end of each day unless other arrangements have been made with the laboratory. Samples will be shipped using the procedures outlined in SOP 7510 (Appendix B).

Laboratory Custody Procedures

The laboratory will have an SOP that details the procedures used to document sample receipt and custody within the laboratory. The following procedures must be addressed in the laboratory custody SOP:

- Each laboratory must have a designated sample custodian who accepts custody of the samples at the time of delivery to the laboratory and verifies that the information on the sample labels matches the information on the COC. The sample custodian must sign and date all appropriate receiving documents and note any discrepancies in sample documentation as well as the condition of the samples at the time of receipt.
- 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 as outlined in the laboratory QAM.
- To ensure traceability of samples during the analytical process the laboratory will assign a sample ID number based on procedures outlined in the laboratory QAM or laboratory SOP.
- The following procedures, at a minimum, must be documented by the laboratory:
  - Sample extraction /preparation
  - Sample analysis
  - Data reduction
  - Data reporting
- Laboratory personnel are responsible for sample custody until the samples are returned to the sample custodian.

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

- When sample analysis and QC procedures are completed any remaining sample must be stored in accordance with contractual terms. A minimum of 30 days notice must be provided before disposal of any sample. Data sheets, custody documents and all other laboratory records must be retained in accordance with contractual agreements.

Final Evidence Files

Laboratory records including COCs and other sample receiving records, sample preparation and analysis records, and the final data package become part of the laboratory final evidence file and must be retained as required by the contractual agreement. An original copy of the data package and associated electronic deliverable must be provided to CSLP in accordance with the contractual agreement and will be retained. Field records will be retained by AECOM for CSLP.

Final evidence files, as defined in Worksheet 29, will be maintained in a secure area under the custody of the AECOM PM for the duration of the project according to the procedures outlined in the appropriate AECOM SOPs and in accordance with the requirements of the CD-RAP. CSLP will maintain these files in a secure, limited access area, under the custody of the Project Manager. TA maintains all GC/MS raw data files on tapes or other magnetic media for an indefinite period. All files are archived by CSLP and are kept for a period of 30 years. This data will be available to USEPA and MPCA upon request

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

Matrix	Groundwater
Analytical Group	PAHs and SVOCs
Concentration Level	Low
Sampling SOP	SOP 7130
Analytical Method/ SOP Reference	DV-MS-0011 and DV-MS-0005
Sampler's Name	AECOM Field Staff
Field Sampling Organization	AECOM
Analytical Organization	TA -Denver
Number of Sample Locations	See SAP

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 and Equipment Rinsate Blank	Method Blank - 1/Batch (20 samples); Equipment Rinsate Blank: 1 per 10 samples from non-dedicated equipment	No Target Compounds>RL;	If sufficient sample is available, reanalyze samples. Qualify data as needed. Report results if sample results >20X blank result or sample results ND.	Analyst/Section Supervisor	Accuracy/Bias-Contamination	No Target Compounds>RL; no common lab contaminants >5xQL.
Surrogates	Every sample	See Laboratory % RCLs (Appendix A)	Check calculations and instrument performance; recalculate, reanalyze	Analyst/Section Supervisor	Accuracy/Bias	See Laboratory % RCLs (Appendix A)
LCS[a]	1/Batch (20 samples) 2/Batch if no MS/MSD in batch	See Laboratory % RCLs (Appendix A)	If sufficient sample is available, reanalyze samples. Qualify data as needed.	Analyst/Section Supervisor	Accuracy/Bias	See Laboratory % RCLs (Appendix A)

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

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
MS/MSD[a]	1/Batch (20 samples)	See Laboratory % RCLs/ RPD Control Limits (Appendix A)	If sufficient sample is available, reanalyze samples; otherwise flag data. Discuss in narrative.	Analyst/Section Supervisor	Accuracy/Bias-Precision	See Laboratory % Recovery/ RPD Control Limits (Appendix A)
Field Duplicate	1/10 field samples	RPD ≤ 30% if both samples are > 5x RL	Evaluate during data validation. Qualify data.	AECOM Data Validators	Precision	RPD ≤ 30% if both samples are >5x RL
Performance Evaluation Sample	Biennial	Supplier Certified Limits	Provide feedback to lab/lab reviews data	AECOM Chemists/ Laboratory Staff	Accuracy/Bias	Supplier Certified Limits

[a] A full analyte list spike solution is required. Control limits are specified only for the subset of compounds in Appx.A, Table A-1

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

Sample Collection Documents and Records	On-site Analysis Documents and Records	Off-site Analysis Documents and Records	Data Assessment Documents and Records	Other
Field notes, field data sheets, field logbooks	Field notes, field data sheets, field logbooks	Custody records and copies of airbills	Reports of field sampling audits	Progress reports
Custody records and airbills	Field instrument calibration records	Analytical data packages and EDDs	Reports of laboratory audits	Annual Report - Prepared and submitted to MPCA and USEPA.
Communication logs, records or copies of pertinent e-mails	Field measurement data	Communication logs	Validation reports	
QAPP, SAP, and HASP	QAPP, SAP, and HASP	Laboratory notebooks and bench sheets documenting sample preparation and analysis	QA reports to management	
Correction action reports and results	Correction action reports and results	Instrument maintenance and calibration records, standard preparation and traceability records	Correction action reports and results	
Documentation of field modifications	Documentation of field modifications	Laboratory SOPs and documentation of method modifications	Internal laboratory assessments, including internal audits, third-party audit reports, and PE results	
		Corrective action logs and documentation of corrective action results		

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

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

Project Document Control System

Project documents are controlled by CSLP and AECOM's PM who will maintain and manage hardcopies and electronic copies of all project related documents according to the CD-RAP. Electronic copies of all information relating to this project are maintained on the project network files which are backed up at least once per day; access to these files is limited to authorized project personnel. All project data and information must be documented in a standard format which is usable by all project personnel.

Data Recording

Data generated during this project will be captured electronically or entered by hand into bound field or laboratory logbooks or preprinted forms. Computer generated laboratory data will be managed using the laboratory information management system (LIMS); the LIMS used by the laboratory is described in their QA documentation (QAM)

Data Quality Assurance Procedures

CSLP and AECOM will monitor the progress of sample collection to verify that samples are collected as planned. The progress of sample collection and processing will be monitored through the documentation of samples collected and shipped each day. The participating laboratories must maintain a formal QA Plan defined by a Quality Assurance Manual to which they adhere and which addresses all data generating aspects of daily operations. A policy of continuous improvement will allow all data generation processes to be reviewed and modified as needed to meet project objectives. Periodic audits of field and laboratory operations will ensure that data collection, documentation and QC procedures are being followed. The frequency and nature of these audits are defined in Worksheet # 33.

Laboratory Data Transmittal

Laboratory data are managed by the LIMS beginning with the sample receiving process. Laboratories are required to provide validated data reports (sample results, QC summary information, and supporting raw data) including EDDs within the turnaround times specified in Worksheet #30. EDDs will be provided in an Earthsoft EQuIS® four-file format (modified by AECOM), using reference file tables provided by AECOM. All EDDs will be checked

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

prior to transmittal to AECOM using current versions of Earthsoft's Electronic Data Processor (EDP). Ms. Uriell of TA is responsible for transmitting the EDD to Mr. Gregg. The EDDs are downloaded into the project database by Mr.Herberich. In addition, paper copies of the laboratory analytical report are sent via mail to Mr. Gregg and the CSLP. Mr. Gregg and the data validation/assessment team spot check ten percent of the EDD against the paper copy of the laboratory report.

Data Conversion and Reduction

Data transformation is conversion of individual data point values into related values using conversion formulas. The data transformations completed on the project data include calculating the sum of the following compounds:

1. Carcinogenic PAH compounds;
2. Benzo(a)pyrene and dibenz(a,h)anthracene; and
3. The remaining non-carcinogenic PAH compounds (Other PAH).

The list of carcinogenic PAHs included in the calculation number 1 can be found in Appendix A, Table A-3. The list of non-carcinogenic "Other PAH" compounds included in calculation number 3 can be found in Appendix A, Table A-4. The results of the sums are compared to the historical data and standards outlined in the CD-RAP and summarized in the Annual Report. The standards are listed in Worksheet #10.

Data Storage and Retrieval

Completed forms, logbooks, photographs, data packages, and electronic files will be transmitted regularly to the Project Manager. The laboratory will maintain copies of all documents it generates as well as backup files of all electronic data relating to the analysis of samples. Raw data and electronic files of all field samples, QC analyses and blanks must be archived from the date of generation and maintained by each laboratory in accordance with the terms of the contract between CSLP and the laboratory. The final evidence (or data) files will be maintained for 30 years as specified in the CD-RAP. Evidence files will consist of all data necessary to completely reconstruct the analysis, and will consist of (at a minimum): all field documents, logs, project reports, raw data, continuing calibration checks, detection limits, chain-of-custody documentation, quality control data for blanks and matrix spikes, results forms, nonconformance documentation and a file custodian. In addition, the analytical report, which contains a brief discussion of the method and a more detailed narrative of any analytical issues, is included in the package. CSLP will maintain these files in a secure, limited access area, under the custody of the Project Manager. TA maintains all GC/MS raw data files on tapes or other magnetic media for an indefinite period. All files

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

are archived by CSLP and are kept for a period of 30 years. This data will be available upon request. A summary of project documents and responsible parties is provided below:

Item	Issued By	Issued To	Archived By
Electronic Laboratory Data	TA-Denver	AECOM/ CSLP	CSLP
Sampling Plan	CSLP	EPA/MPCA	CSLP
Annual Report	CSLP	EPA/MPCA	CSLP
Annual Progress Report	CSLP	EPA/MPCA	CSLP
Correspondence/Corrective Actions	All Parties	All Parties	All Parties
Field Notebooks	Field Coordinator	Sampling Team	Field Coordinator
Bill of Lauding	Shipping Company	Sampling Team	Sampling Team
Field Equipment Calibration Forms	Field Coordinator	Sampling Team	Field Coordinator
Sample Logs	Field Coordinator	Sampling Team	Field Coordinator
Chain-of-Custody Forms	Lab Sample Custodian	Field Coordinator	Lab Sample Custodian
Sample Labels	Field Coordinator	Sampling Team	Lab Sample Custodian

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

Matrix	Analytical Group	Concentration Level	Sample Locations/ ID Number	Analytical SOP	Data Package Turnaround Time[a]	Laboratory/ Organization	Backup Laboratory/ Organization)
Water	PAHs, SVOCs	Low	All	DV-MS-0005 DV-MS-0011	30 days	TA - Denver 4955 Yarrow Street Denver, CO 80002 303.736.0100	None designated

[a] Turnaround time is in calendar days from receipt of the last sample in the data package sample delivery group. In accordance with Section 3.2 of the RAP, TA has agreed to a 30-working day turnaround. CSLP, however, makes no enforceable commitment under the RAP except for a maximum of seven days from time of sample collection for extraction of organics and 40 days following extraction for analysis of organics. For non-organic analyses, CSLP makes no enforceable commitment under the RAP except to meet the recommended maximum analytical holding times.

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	Person(s) Responsible for Responding to Assessment Findings	Person(s) Responsible for Identifying and Implementing Corrective Actions (CA)	Person(s) Responsible for Monitoring Effectiveness of CA
Field Performance and System Audit	Quarterly	Internal	AECOM	Bill Gregg/PM/AECOM	Bill Gregg/ PM/AECOM	Bill Gregg/PM/AECOM	Bill Gregg/ PM/AECOM
Laboratory Performance and System Audit	Biennial	External	AECOM	Bill Gregg/PM/AECOM Lori Herberich/ QA Officer/AECOM	Mike Schmitt/ QA Manager/TA Denver	Karen Kuoppala/ QA Manager/TA Denver	Bill Gregg/ PM/AECOM
Data Package Technical Systems Audit	Quarterly	External	AECOM	Bill Gregg/PM/AECOM Jim Herberich/ Data Manager/AECOM	Lisa Uriell/ PM/TA-Denver	Lisa Uriell/ PM/TA-Denver	Bill Gregg/ PM/AECOM
Data Validation Technical Systems Audit	Quarterly	External	AECOM	Lori Herberich/ QA Officer/AECOM	Lisa Uriell/ PM/TA-Denver	Lori Herberich/ QA Officer/AECOM Karen Kuoppala/ QA Manager/TA Denver	Lori Herberich/ QA Officer/AECOM
Performance Evaluation Assessment	Biennial	External	AECOM	Bill Gregg/PM/AECOM Lori Herberich/ QA Officer/AECOM	Karen Kuoppala/ QA Manager/TA Denver	Lisa Uriell/ PM/TA-Denver	Bill Gregg/ PM/AECOM
Overall Usability Assessment	Quarterly	Internal	AECOM	Bill Gregg/PM/AECOM Lori Herberich/ QA Officer/AECOM	Bill Gregg/PM/AECOM Lisa Uriell/PM/TA-Denver	Bill Gregg/PM/AECOM Lori Herberich/ QA Officer/AECOM	Bill Gregg/ PM/AECOM

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	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response	Timeframe for Response
Field System Audit	Written audit report	Project Manager	Verbal summary of major findings within 24 hours; written report within one week.	Memo with possible reaudit	Project QA Manager, Project Manager	One week
Internal Laboratory Audits	Written audit report	Laboratory Manager	Major deficiencies within 24 hours; written report as required by laboratory QAMl	Memo or as required by laboratory QA Manual	Laboratory Manager, Laboratory PM AECOM Project QA Officer and AECOM PM (if project DQOs are affected)	As required by laboratory QAM
External Laboratory Audits by AECOM	Written audit report	Laboratory Manager	Major deficiencies communicated orally at exit meeting written report within 1 month	Letter with possible reaudit	AECOM Project QA Officer, and AAECOM PM	One month

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	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response	Timeframe for Response
PE samples	Written PE results evaluation report	Laboratory Manager	Deficiencies (results outside acceptance range) identified within one week of receiving laboratory results	Letter with request for laboratory investigation into deficiencies and corrective action, if necessary, before project field samples are analyzed. Corrective action may include investigation and preparation by the laboratory of a corrective action report, analysis of a new PE sample, or if AECOM deems appropriate, the analyses may be moved to another lab.	AECOM Project QA Officer, and Project Manager	One week

Non-Conformance/QC Reporting

A non-conformance is defined as an identified or suspected deficiency in, or deviation from, procedures described in an approved document (e.g., improper sampling procedures, improper instrument calibration, errors in calculations or errors in computer algorithms); an item where the quality of the end product itself or subsequent activities conducted using the document or item would be affected by the deficiency; or an activity that is not conducted in accordance with established plans or procedures. Any project staff member that discovers or suspects a non-conformance is responsible for initiating a non-conformance report to the Project QA Officer. The Project QA Officer will evaluate each non-conformance report and provide a response describing the actions to be taken and assigning responsibility for the corrective action. The AECOM PM will verify that the nonconforming item or procedure is not used until the corrective action has been performed and found to produce acceptable results. If the non-conformance involves instrumentation or equipment, the device must be tagged to indicate it is defective and not to be used.

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

A copy of each non-conformance report will be added to the project file. Original non-conformance reports will be maintained by the Project QA Officer.

Assessment

Assessment activities will measure the effectiveness of the project implementation and associated QA/QC activities. Audits are used as a means of monitoring the performance of field and laboratory activities and are conducted by the Project QA Officer or another member of the QA staff. Audits will include systems audits which are more qualitative in nature and will be made at appropriate intervals to ensure that all aspects of the QA program are operative. Performance audits are quantitative audits which are conducted to assess the accuracy of measurement systems; this would include the use of performance evaluation samples.

Systems audits will be conducted for field and laboratory operations to assess implementation of QA/QC requirements and determine if the systems under review are capable of meeting project DQOs. Any minor deficiencies noted during an audit will be corrected as soon as possible according to an agreed upon schedule. If a major deficiency is noted during an audit a stop work order will be issued until the deficiency can be corrected and the effectiveness of the corrective action measured and documented. A stop work order may be issued by the Project QA Officer who will notify the AECOM PM. The conditions which lead to a stop work order must be documented in sufficient detail to clearly define the problem and identify possible corrective measures. All communications among project staff which address evaluation of the problem and appropriate solutions must be attached to the stop work order. The Project QA Officer, and AECOM PM must agree in writing to resume work after review of the data supporting correction of the deficiency. The Project QA Manager will maintain a corrective action log which lists deficiencies that were noted, the individual(s) responsible for follow-up, documentation of the effectiveness of the corrective actions taken, and implementation of procedures to prevent recurrence of the problem.

A written report will be prepared for all audits regardless of the outcome and submitted to the Project Manager. Any modifications to the existing program, corrective actions required, or the need for additional audits will be documented.

In addition to participation in any audits conducted by AECOM QA personnel, participating laboratories are required to take part in regularly scheduled performance evaluations (PE) and audits required by state and federal agencies as part of ongoing certification or participation in specific contracts and to provide copies of the results of these performance evaluations and audits to the Project QA Officer. These PE and audit results will be reviewed as part of the biennial laboratory technical systems audit. Any change in laboratory ownership, management, or certification status must be immediately reported to the Project QA Officer. If any laboratory analysis is found to be out of control, the laboratory must immediately implement corrective action and notify the Project QA Officer. The laboratory PM will be responsible for documenting the effectiveness of the corrective action measures before continuing analysis of project samples.

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

Type of Report	Frequency	Projected Delivery Date(s)[a]	Person(s) Responsible for Report Preparation	Report Recipient(s)
Laboratory Performance and System Audit Report	Biennial	4th Qtr 2007	Lori Herberich/ QA Officer/AECOM and Bill Gregg/PM/AECOM	Lisa Uriell/PM/TA and Scott Anderson/ Superintendent of Utilities/SLP
Field Performance and System Audit Report	Quarterly	1 month after sampling	Bill Gregg/ Field Team Coordinator/AECOM	Bill Gregg/PM/AECOM
Data Package Technical Systems Audit Report	Quarterly	1 month following receipt of package	Jim Herberich/ Data Manager/AECOM	Bill Gregg/PM/AECOM

[a] Dates will vary depending on the sampling and audit schedule created by the Field Coordinator

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

Verification Input	Description	Internal/ External	Responsible for Verification)
Field data	The field data produced will be reviewed to ensure that the records are complete, accurate, and legible and to verify that the sampling procedures are in accordance with the protocols specified in the Work Plan.	Internal	AECOM Field Team Leader
Chain-of-Custody	The COC will be reviewed initially in the field for complete and correct information.	Internal	AECOM On-Site Coordinator or designee
	Upon receipt at the lab the COC will be compared to sample containers and any discrepancies will be resolved.	External	Laboratory Sample Custodian
	During validation the COC will be verified against laboratory receipt and reporting information.	External	AECOM Data Validator
Laboratory Data Packages and EDD	Laboratory data (hard copy and EDDs) will be verified by the laboratory performing the work for completeness and technical accuracy prior to release.	Internal	Laboratory
	Laboratory data will be assessed using the validation procedures described in Worksheets 35 and 36	External	AECOM Data Validator
Audit Reports	Audit reports will be reviewed to confirm that specified corrective actions have been taken, the corrective action has been effective and all documentation of corrective action is attached to the audit report.	Internal	AECOM Project QA Officer
Assessment actions and reports	QA/QC process will be reviewed for agreement with QAPP, SAP, and requirements of the CD-RAP	Internal	AECOM Project Manager

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

Step IIa/IIb	Validation Input	Description	Responsible for Validation
IIa	Field SOPs, field records	Verify conformance to approved sampling and field measurement procedures; ensure that activities met performance criteria; and verify that deviations from procedures or criteria were documented.	Field Coordinator AECOM
IIa	Analytical data deliverables, contractual documents	Verify the required deliverables, analyte lists, method holding times, analytical procedures, laboratory qualifiers, measurement criteria, project quantitation limits, and analyses of PE samples conform to specifications. Verify that deviations from procedures or criteria were documented.	Project QA Officer /AECOM
IIa	Custody records, analytical data reports	Review traceability from sample collection through reporting.	Project QA Officer /AECOM
IIa	Laboratory EDDs, analytical data reports, database output	Verify EDDs against hard-copy analytical reports.	PM or Data Management Task Manager/AECOM
IIa	Data validation reports, database output	Verify that entry of qualifiers was correct and complete.	Project QA Officer /AECOM
IIb	Analytical data reports	Verify that reported analytes, holding times, analytical procedures, measurement critieria, and project quantitation limits conform to the QAPP. Verify that deviations from procedures or criteria were documented.	Project QA Officer /AECOM
IIb	Analytical data reports, validation guidance	One hundred percent of the data will be validated (see details below)	Project QA Officer /AECOM
IIb	QAPP, analytical data reports, validation guidance	Verify that the qualifiers applied during validation were in conformance with the QAPP and specified validation guidance.	Project QA Officer /AECOM
IIb	Analytical data reports	Verify that PE samples were analyzed at the frequency specified in the QAPP and met the acceptance criteria.	Project QA Officer /AECOM

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

Step IIa/IIb	Validation Input	Description	Responsible for Validation
IIb	QAPP, data validation reports	Verify that data validation was performed in accordance with the QAPP specifications and that all required peer reviews were conducted. If validation actions deviated from the QAPP specifications and/or regional validation guidance based on professional judgment, verify that rationale was documented.	Project QA Officer /AECOM

Data Validation

The laboratory data will be validated and verified as described in Worksheet #36. In summary, a subset of data received will be subjected to a full data validation. The remainder of the data will receive a limited data assessment. Data will be qualified and the results of the assessment will be summarized in a validation/assessment memo.

Limited validation will be based on information provided by the laboratory on their QC forms, and will include no raw data review. At a minimum, limited validation will include the following data elements:

- Agreement of analyses conducted with chain-of-custody (COC) requests
- Completeness of deliverable
- Holding times/sample preservation
- Laboratory blanks/field blanks
- Surrogate spike recoveries
- Matrix spike (MS)/matrix spike duplicate (MSD) results
- Laboratory control sample (LCS) results
- Field duplicate results
- Sample quantitation/detection limit results (including conformance of hardcopy and EDD)

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

Full validation will include all of the data elements of the limited validation, plus the following:

- Gas chromatography/mass spectrometry (GC/MS) tuning
- Initial and continuing calibrations
- Internal standard performance
- Laboratory control sample (LCS) results
- Mass spectral qualitative identifications (full scan data only)
- Verification of selected sample calculations.

If significant issues (e.g., those affecting achievement of the DQOs) are noted during full validation, the limited validation will be expanded to include this issue. Systematic or random errors that would not be detected during a review of the summary forms might include, for example, misidentification or quantitation of compounds, transcription errors, or calculation errors. In addition, limited validation will provide review of key laboratory QC elements, which would highlight potential underlying lab issues which may require further investigation (i.e., full validation effort). If a high frequency of measurement performance issues are found, the issue will be investigated and an additional validation effort may be implemented. AECOM plans to maintain communication/notification systems with the laboratory during the analytical process to circumvent significant QC issues. If QC issues do arise, investigations and corrective actions will be documented and implemented in a timely fashion to optimize the amount of un-qualified data.

Qualifiers will be applied to the data based on the criteria in the QAPP, Region 5 validation guidance, or professional judgement.

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

Step IIa/IIb	Matrix	Analytical Group	Concentration Level	Validation Criteria*	Data Validator (title and organizational affiliation)
IIa	Water	PAHs and SVOCs	Low	"Region 5, Standard Operating Procedure for Validation of CLP Organic Data", (April 1991, Revised February 1997).	Project QA Officer/AECOM (or designate)

* These guidelines will be modified to reflect any differences in analytical methodology and to incorporate the project-specific acceptance criteria defined this QAPP. Data assessment/validation will include two types, limited and full. Full validation will be completed on 1 package from 2nd and 3rd Quarter sampling events. All other data packages will undergo a limited assessment as described in Worksheet #35 of the QAPP.

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

Summarize the usability assessment process and all procedures, including interim steps and any statistics, equations, and computer algorithms that will be used:
AECOM's data validation staff will validate all laboratory data in accordance with the protocols described in Worksheet 36. The AECOM PM, in conjunction with the project team, will determine whether the analytical data meet the requirements for use in making decisions related to further actions at the site. The results of laboratory measurements will be compared to the performance criteria described in Worksheet # 12, #15, and #28 of this QAPP.

Describe the evaluative procedures used to assess overall measurement error associated with the project:
During the data validation process the validator will use information confirming sample identification; sample preparation; analysis within holding time; instrument calibration data; and results of QC samples designed to assess blank contamination, analytical precision, and accuracy to identify any limitations in data use and, if known, data bias. The validator will apply qualifiers as needed to reflect any limitations on the use of specific data points and prepare a report detailing the information reviewed, data limitations, and overall usability. Patterns of data use limitations or anomalies which become apparent during the validation process or as the users will be reviewed with the AECOM PM and the appropriate laboratory. Data that do not meet the quality acceptance limits of Worksheet #28, or quality levels of Worksheet #15, or analytical performance criteria specified in Worksheet #12 will be clearly identified in the database so data users are aware of any limitations associated with data usability. Details of the problems identified during data validation and the bias in the data will be provided in the associated validation memorandum.

Identify the personnel responsible for performing the usability assessment:
Data validation will be performed by AECOM's data validation staff under the supervision of the AECOM PM. The usability assessment will be performed jointly by the AECOM project team and CSLP and will include input by field personnel, QA staff, and project management.

Describe the documentation that will be generated during usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies:
The documentation generated during data validation will include a comprehensive memorandum that describes the information reviewed, the results of this review and provides a recommendation on overall data usability and limitations on specific data points. The memorandum and associated validation worksheets provide information on the samples included in the review and the date they were collected; the condition of samples when received at the laboratory and any discrepancies noted during the receiving process; verification of sample preparation and analysis within the method specified holding time; instrument calibration information; review of associated QC analyses including blanks, laboratory control samples, matrix spikes, and field and/or laboratory duplicates; verification of selected reported values from raw data. As a result of this review standard qualifiers are entered into the database so that data users can readily identify any limitations associated with a specific data point.

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

Assessment of data usability will be performed by AECOM's data validation staff using current USEPA Region 5 data validation guidance. The results of the Data Usability Assessment will be summarized in the final project report. The following items will be assessed and conclusions drawn based on their results:

Holding Time: All sample data will be checked to verify that both sample preparation and analysis were performed within the method required holding time.

Calibration: Data associated with instrument calibration and verification of calibration will be reviewed to confirm that all data were generated using properly calibrated instrumentation.

Accuracy/Bias Contamination: Results for all field blanks, trip blanks, laboratory method blanks, and instrument calibration blanks will be checked against performance criteria specified in Worksheet # 28; results for analytes that exceed criteria will be identified and the impact on field sample data will be assessed. Data will be summarized by type of blank.

Accuracy/Bias Overall: Reported values of laboratory control samples, performance samples, and matrix spikes will be evaluated against the spiked or certified concentration and the percent recovery will be calculated and compared to the criteria specified in Worksheet #28. The percent recovery information will be used to assess the bias associated with the analysis. Recovery for matrix spikes in conjunction with the recovery reported for performance samples and laboratory control samples will provide information on the impact of the sample matrix on specific analyses.

Precision: Results of the RPD will be calculated for each analyte in laboratory and field duplicates. These RPDs will be checked against measurement performance criteria presented on Worksheet #28; RPDs exceeding the stated criteria will be identified. Additionally the combined RPD of each analyte will be averaged across duplicate pairs whose original and duplicate values are both greater than the RL. This information will be used to draw conclusions about the precision of the analyses and, for field duplicates, the precision of sampling and analysis. Any limitations on the use of the data will also be described.

Sensitivity: Reporting limits will be checked against the criteria presented on Worksheet #15 and QLs presented on Worksheet #15. Limitations on the use of the data and conclusions about the sensitivity of the analysis will be reported.

Representativeness: A review of field records will be used to confirm that sample collection and handling was performed in a manner that conformed to the designated SOP. Similarly laboratory preparation procedures will be reviewed during validation to ensure that a representative sample was selected

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

for analysis. Any deviations or modifications to field or laboratory procedures which might impact the representativeness of the sample will be discussed in the project final report.

Comparability: The sampling and analytical procedures which will be used in this program have been selected to ensure that the resulting data will be comparable to historical data from the site and meet all requirements of the CD-RAP. Any modifications or deviations from stated procedures which might impact data comparability will be addressed in the project final report. Data inconsistencies are potentially short-term problems that are addressed by AECOM and CSLP jointly. During the previous years of CD-RAP monitoring, when any municipal well has contained PAH above advisory levels then the specific course of action to resample that well, in accordance with the CD-RAP was followed. The data have been successfully used for previous 13 years to identify "breakthrough" at the carbon treatment plants, and to plan the replacement of the carbon in accordance with the CD-RAP. Other than the resampling prescribed by the CD-RAP, data inconsistencies are not the basis for any field corrective actions. The long-term nature of the groundwater containment remediation strategy allows any data inconsistencies to be put into the context of a large data set that defines water quality. The laboratory analytical method has evolved, and has been refined, over the years to avoid data inconsistencies that were apparent during the earlier years of this program. CSLP and AECOM will continue to evaluate the laboratory analytical procedures in an effort to understand any data inconsistencies and the potential need for corrective actions.

Completeness: Completeness for the analytical program will be calculated as the number of data points that are accepted as usable based on the validation process divided by the total number of data points for each analysis. Completeness will be reported for each analytical category and an overall value will be reported. As shown in Worksheet #12, the analytical completeness goal is ≥95%. Completeness for the field program will be calculated as the number of samples successfully collected compared to the total number proposed in the SAP. The completeness goal for the field sampling program is ≥95%.

Each of the PQOs presented on Worksheet #15 will be reviewed to determine if the stated objective was met. The major impacts observed from data validation, data quality indicators (DQI) and measurement performance criteria assessments will be used to assess the overall data quality and whether PQOs were achieved. The final report will summarize the information used to reconcile each objective and overall conclusions regarding data quality.

Overall Assessment of Environmental Data:

Data assessment will involve data evaluation and usability to determine if the data collected are of the appropriate quality, quantity, and representativeness to support the CD-RAP. This evaluation will be performed by the AECOM PM in concert with other users of the data. The QC results associated with each analytical parameter for each matrix type will be compared to the objectives presented in this QAPP. Data generated in association with QC results meeting these objectives and/or the data validation criteria will be considered usable. Data that does not meet the objectives and/or the

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

data validation criteria may still be usable. This assessment may require various statistical procedures to establish outliers, correlations between data sets, adequate sampling location coverage of the site, etc, in order to assess the effect of qualification or rejection of data. The effect of the qualification of data or loss of data deemed unacceptable for use, for whatever reason, will be discussed and decisions made on corrective action for potential data gaps.

Appendix A

Laboratory Control Limits and Project Defined Analyte Groups

Table A-1

Data Quality Objectives for Analytical QC Measurements in Aqueous Samples

Parameter	Blanks[1]	LCS %R	MS/MSD %R	MS/MSD RPD
PAH (ppt)				
Naphthalene	<PQL	30-150	30-150	50
Florene	<PQL	30-132	30-132	50
Chrysene	<PQL	30-132	30-132	50
IH-Indene	<PQL	30-150	30-150	50
Quinoline	<PQL	30-150	30-150	50
Benzo(e)pyrene	<PQL	30-150	30-150	50
2-methylnaphthalene	<PQL	30-150	30-150	50
PAH (ppb)				
Acenaphthene	<PQL	60-101	60-101	30
Acenaphthylene	<PQL	60-100	60-100	30
Anthracene	<PQL	63-103	63-103	30
Benzo(a)anthracene	<PQL	61-104	61-104	30
Benzo(b)fluoranthene	<PQL	59-105	59-105	30
Benzo(k)fluoranthene	<PQL	60-105	60-105	30
Benzo(g,h,I)perylene	<PQL	55-112	55-112	30
Benzo(a)pyrene	<PQL	58-100	58-100	30
Chrysene	<PQL	59-102	59-102	30
Fluoranthene	<PQL	62-104	62-104	30
Fluorene	<PQL	62-104	62-104	30
Indeno(1,2,3-cd)pyrene	<PQL	56-112	56-112	30
Naphthalene	<PQL	58-98	58-98	30

[1] Applies to both laboratory and field blanks. Note a full analyte list spike is required for the LCS/LCSD and MS/MSD although control limits are established for only the compounds listed in this table.

Table A-2

Data Quality Objectives for Surrogate Measurements in Aqueous Samples

Surrogate	Acceptance Criteria Percent Recovery	
	PPT	PPB
Naphthalene-d8	30 - 118	37-107
Fluorene-d10	41 - 162	36-127
Chrysene-d12	30 - 118	30-160

Table A-3

List of Carcinogenic PAH Compounds

Carcinogenic PAH[1]
benzo(a)anthracene
benzo(b)fluoranthene
benzo(j)fluoranthene
benzo(ghi)perylene
benzo(a)pyrene[2]
chrysene
dibenz(a,h)anthracene[2]
indeno(1,2,3-c,d)pyrene
quinoline

1 The total maximum levels of carcinogenic PAH established in the CD-RAP are:
- Advisory Level - 15 ng/l
- Drinking Water Criterion - 28 ng/l

2 The total maximum levels of the sum of benzo(a)pyrene and dibenz(a,h)anthracene are:
Advisory Level - 3.0 ng/l (or the lowest concentration that can be quantified, whichever is greater)
- Drinking Water Criterion - 5.6 ng/l

Table A-4

Non-Carcinogen PAH Compounds Included in "Other PAH" Calculation

CAS Number	Compound
271-89-6	2,3-Benzofuran
496-11-7	2,3-Dihydroindene
95-13-6	1H-Indene
91-20-3	Naphthalene
4565-32-6	Benzo(B)Thiophene
120-72-9	1H-Indole
91-57-6	2-Methylnaphthalene
90-12-0	1-Methylnaphthalene
92-52-4	Biphenyl
208-96-8	Acenaphthylene
83-32-9	Acenaphthene
132-64-9	Dibenzofuran
86-73-7	Fluorene
132-65-0	Dibenzothiophene
85-01-8	Phenanthrene
120-12-7	Anthracene
260-94-6	Acridine
86-74-8	Carbazole
206-44-0	Fluoranthene
129-00-0	Pyrene
207-08-9	Benzo(K)Fluoranthene
192-97-2	Benzo(E)Pyrene
198-55-0	Perylene

Appendix B

Field Standard Operating Procedures

ENSR | AECOM

Standard Operating Procedure

Chain-of-Custody Procedures

Procedure Number: 1007

Revision No.: 2

Revision Date: February 2008

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Annual review of this SOP has been performed
and the SOP still reflects current practice.

Initials: _____ Date: _____
Initials: _____ Date: _____

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1.0 Scope and applicability

1.1 Chain of custody (COC) is defined as the unbroken trail of accountability that ensures the physical security of samples, data, and records (EPA Glossary of Quality-Related Terms). This standard operating procedure (SOP) describes COC procedures applicable to environmental samples collected by ENSR during field sampling and analysis programs. Custody procedures within the laboratories analyzing the samples are not addressed.

1.2 Samples are physical evidence. The objective of COC procedures is to provide sufficient evidence of sample integrity to satisfy data defensibility requirements in legal or regulatory situations.

1.3 The National Enforcement Investigations Center (NEIC) of the U.S. Environmental Protection Agency (EPA) defines custody of evidence in the following manner:

- It is in your actual possession;
- it is in your view, after being in your physical possession;
- it was in your possession and then you locked or sealed it up to prevent tampering; or
- it is in a secure area.

1.4 This SOP is to be utilized to conduct the work identified in the title of this SOP. In the event the Project Manager or Project Team determines that the protocols and procedures listed in this SOP are not applicable to the project, there is the option to either adapt this SOP or to develop a site-specific SOP to more closely match the requirements of the project. Refer to SOP 1011, Preparation and Control of Standard Operating Procedures, for SOP modification and Project Operating Procedure (POP) development procedures.

2.0 Health and safety considerations

2.1 The health and safety considerations for the work associated with this SOP, including both potential physical and chemical hazards, will be addressed in the site-specific Health and Safety Plan (HASP). In the absence of a site-specific HASP, work will be conducted according to the ENSR Health and Safety Policy and Procedures Manual and/or direction from the Regional Health and Safety Manager.

3.0 Interferences

The following may impact the legal or regulatory defensibility of the data:

- The samples are not accompanied by a COC form,
- The information recorded on the COC form is incomplete, inaccurate, or differs from the information recorded on the sample containers,

- The documentation of person-to-person transfer of sample custody is incomplete, or contains unexplained gaps in time,
- COC seals or tape were not applied, were not applied correctly, or were lost or removed, for sample coolers/packages being transported by a party other than the sample custodian.

4.0 Equipment and materials

4.1 The following materials are relevant to this procedure:

- COC Form (Figure 1)
- Sample labels
- COC tape or seal (Figure 2)
- Indelible pen or Sharpie™
- Clear plastic sealing tape

4.2 Materials identified in related SOPs may also be needed.

5.0 Procedures

5.1 Pre-sample collection activities

5.1.1 Some measurement methods require preparation of sample collection media or special treatment of sample containers prior to sample collection. In these cases, COC procedures should be initiated with the media preparation or container treatment. This requires that sample identification numbers or media/container identification numbers be assigned. These numbers should be entered on the COC form, leaving room for the subsequent recording of the associated sample numbers. In this variation, the custodian responsible for media preparation or container treatment has the responsibilities outlined in Section 5.2, and the sampler or field sample custodian has the responsibilities stated in Section 5.3 when he or she receives the prepared media or treated containers. There are a number of acceptable approaches to this variation, and the detailed procedures should be defined in the project-specific QAPP.

5.2 Sample collection phase

5.2.1 As few people as possible should handle the samples. For certain programs, it is helpful if a single person is designated as the sample custodian (the person responsible for the care and custody of the samples until they are transferred to the laboratory for analysis).

5.2.2 While in the field, sampling personnel should be able to testify that tampering of the samples could not occur without their knowledge. Examples of actions taken may include sealing the sample containers with COC tape or locking the samples in a secure area.

5.2.3 If samples are to be shipped by commercial overnight carrier, the field sampler or sample custodian completes a COC form (Figure 1) for each cooler/package of samples and places the original of completed form inside the associated cooler/package before the package is sealed (a copy is retained and kept in the field record files). Each completed COC form should accurately list the sample identification numbers of the samples with which it is packaged, and should contain the identification number of the COC tape on the cooler/package. Representatives of commercial carriers are not required to sign the COC form. Refer to ENSR SOP 7510 – Packaging and Shipment of Environmental Samples for specific packaging procedures.

5.2.4 If samples are hand carried to a laboratory, the person hand carrying the samples is the sample custodian. If the carrier is a different person than the one who filled out the COC form and packaged the samples, then that person transfers custody to the carrier by signing and dating each form in the "Relinquished By" section. The carrier then signs and dates each form in the adjacent "Received By" section. When the carrier transfers the samples to the laboratory, he or she signs and dates each form in the next "Relinquished By" section, and the laboratory sample custodian signs and dates each form in the adjacent "Received By" section.

5.2.5 If samples are transmitted to the laboratory by courier, the procedures described in either Section 5.2.3 or 5.2.4 are followed, depending on whether the courier is a commercial courier or laboratory representative, and whether the cooler has been secured by COC seals prior to pick up by a laboratory courier.

5.3 Sample labeling

5.3.1 Labeling of samples occurs at the time of sample collection.

5.3.2 Waterproof, adhesive labels are preferred. Labels should be applied to the container, not the lid whenever possible. Additional interior labels may be required for certain biological samples.

5.3.3 Sample tags may be required for certain projects requiring a strict level of legal or regulatory data defensibility. If tags are utilized, their use will be addressed in the project-specific work plan or QAPP.

5.3.4 Labels should be completed in waterproof, indelible ink. Covering the label with clear plastic tape is recommended to protect the legibility of the label and to prevent the label from detaching from the sample container.

5.3.5 The following information should be recorded on the sample label:

- Project identification (project name and number/client/site)
- Field sample identification code (exactly as it appears on the COC form)
- Sampler's initials
- Date and time of sample collection
- Analyses requested
- Preservation

5.4 Documentation of sample history

5.4.1 Sample history includes, but is not limited to, preparation of sample containers or collection media (for example, wipes), collection, handling (such as subsampling or compositing), storage, shipment, analytical preparation and analysis, reporting, and disposal.

5.4.2 Refer to SOP 7515, Recording of Field Data, for specific guidance on documentation of field activities, field measurements, and sample collection.

5.5 Documentation of custody

5.5.1 It is recommended that a COC form (Figure 1 or equivalent) be initiated upon sample collection. If this is not feasible for a particular project, the COC form may be initiated at the time of sample packaging. If this is the case, the sample collection records will serve as the initial custody document and will document the collection of the sample (sample location and identification, date and time of collection, sampler, and parameters to be analyzed, including containers and preservatives).

5.5.2 The following information is recorded on the COC form:

- Project identification (ENSR project number, client, site name and location).
- Page number (for example, 1 of 2, 2 of 2).
- Field sample identification code. This code should be unique to the sampling event and to the program. This code should agree exactly with the field sample identification code recorded on the bottle label.
- Sampling point location (optional if recorded elsewhere in field records).
- Date and time of sample collection.
- Sample matrix (soil, water, air, etc.).
- Preservative.
- Analysis requested.
- Number of containers.
- Type of sample (grab or composite). Identifying if aqueous samples have been filtered in the field is recommended.
- Signature(s) of sampling personnel and signatures of all personnel handling, receiving, and relinquishing the samples.
- Date(s) and time(s) of each sample transfer.
- Sampler remarks. These comments may serve to alert the laboratory to highly contaminated samples or identify quality control (QC) sample requirements.
- Airbill number (if shipped by overnight commercial carrier).
- Laboratory name and address.
- COC tape numbers.

5.5.3 The COC is filled out completely and legibly in indelible ink. There should be no unexplained blank spaces. Blank lines should be lined out and initialed and dated.

5.5.4 Data will not obliterated. Corrections are made, if necessary, by drawing a single line through and initialing and dating the error. The correct information is then recorded with indelible ink.

5.5.5 Information on the COC should agree exactly with that recorded on the sample containers. Discrepancies may result in the samples being incorrectly logged into the laboratory or delays in initiating sample analysis.

5.6 Sample receipt and inspection

5.6.1 Upon sample receipt, the coolers or packages are inspected for general condition and the condition of the COC tape. The coolers or boxes are then opened and each sample is inspected for damage.

5.6.2 Sample containers are removed from packing material and sample label information is verified against the COC form.

5.6.3 The condition upon receipt, including any discrepancies or problems, is documented and the COC form is completed by signing and recording the date and time of receipt.

5.6.4 Receipt and inspection of samples by subcontractor analytical laboratories will adhere to written procedures established by the laboratory.

6.0 Quality assurance / quality control

6.1 The records generated in this procedure are subject to review by the sampling team leader, project manager, or designee.

6.2 The records generated in this procedure will become a part of the evidence reviewed in the data validation process (see ENSR SOP 1009, Data Validation).

7.0 Data and records management

7.1 The records generated in this procedure are part of the permanent record supporting the associated measurements and may include, as applicable, the COC forms, sample tags, carrier waybills, and field and laboratory records of sample history (collection, handling, storage, analysis, etc.).

7.2 Unanticipated changes to the procedures or materials described in this SOP (deviations) should be appropriately documented in the project records.

7.3 Records associated with the activities described in this SOP should be maintained according to the document management policy for the project.

8.0 Personnel qualifications and training

8.1 Qualifications and training

8.1.1 The individual executing these procedures should have read, and be familiar with, the requirements of this SOP.

8.1.2 No specialized skills are necessary in order to implement these procedures; however, an understanding of the concept of custody is useful.

8.2 Responsibilities

8.2.1 The project manager is responsible for providing the project team with the materials, resources and guidance necessary to properly execute the procedures described in this SOP.

8.2.2 The individual performing the work is responsible for implementing the procedures as described in this SOP and any project-specific work plans.

8.2.3 For certain sampling programs, the project manager, sampling team leader, or designee may assign an individual to serve as sample custodian. This individual is responsible for supervising the implementation of COC procedures in accordance with this SOP and any project-specific work plans or QAPP.

9.0 References

American Society for Testing and Materials (ASTM). 2004. Standard Guide for Sample Chain-of-Custody Procedures. D 4840-99 (Reapproved 2004).

ENSR SOP 1009 – Data Validation.

ENSR SOP 1011 – Preparation and Control of Standard Operating Procedures.

ENSR SOP 7510 – Packaging and Shipment of Environmental Samples.

ENSR SOP 7515 – Recording of Field Data.

United States Environmental Protection Agency. 2001. Guidance for Preparing Standard Operating Procedures (SOPs). EPA QA/G-6. EPA/240/B-01/004. USEPA Office of Environmental Information, Washington, DC. March 2001.

10.0 Revision history

Revision	Date	Changes
0	NA	NA
1	4th Quarter, 1994	Reformatted and expanded from SOP 010-010-601
2	February 2008	Complete rewrite and format update

Figure 1 Example Chain-of-Custody Form

ENSR | AECOM

CHAIN OF CUSTODY RECORD

Page ____ of ____

Client/Project Name:	Project Location:	Analysis Requested
Project Number:	Field Logbook No.:	
Sampler (Print Name)/(Affiliation):	Chain of Custody Tape Nos.:	
Signature:	Send Results/Report to:	TAT:

Container Type
P – Plastic
A – Amber Glass
G – Clear Glass
V – VOA Vial
O – Other
E – Encore

Preservation
1 – HCl, 4°
2 – H2SO4, 4°
3 – HNO3, 4°
4 – NaOH, 4°
5 – NaOH/ZnAc, 4°
6 – Na2S2O3, 4°
7 – 4°

Matrix Codes:
DW – Drinking Water
WW – Wastewater
GW – Groundwater
SW – Surface Water
ST – Storm Water
W – Water
S – Soil
SL – Sludge
SD – Sediment
SO – Solid
A – Air
L – Liquid
P – Product

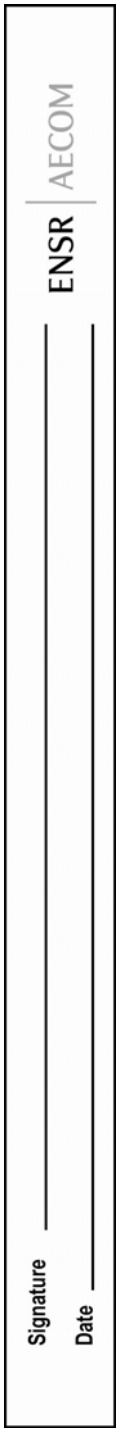
Field Sample No./Identification	Date	Time	COMP	GRAB	Sample Container (Size/Mat'l)	Matrix	Preserv.	Field Filtered											Lab I.D.	Remarks

Relinquished by: (Print Name)/(Affiliation) Signature:	Date: Time:	Received by: (Print Name)/(Affiliation) Signature:	Date: Time:	Analytical Laboratory (Destination):
Relinquished by: (Print Name)/(Affiliation) Signature:	Date: Time:	Received by: (Print Name)/(Affiliation) Signature:	Date: Time:	
Relinquished by: (Print Name)/(Affiliation) Signature:	Date: Time:	Received by: (Print Name)/(Affiliation) Signature:	Date: Time:	Sample Shipped Via: UPS FedEx Courier Other / Temp blank Yes No

Q:\GRAPHICS\FORMS\Chain of Custody (COC)\Chain-of-Custody_AECOM_06.doc

Serial No._____

Figure 2 Example Chain-of-Custody Tape



SOP NUMBER: 7121

Field and Laboratory Measurement of pH

Date:	July 1998
Revision Number:	0
Author:	Lori Fuller
Discipline:	Water

1.0 INTRODUCTION

1.1 Purpose and Applicability

This Standard Operating Procedure (SOP) provides basic instructions for routine calibration and operation of a variety of pH meters, including the Hydrolab, Hydac Multimeter Probe, Orion SA 230, YSI Model 3500, and Horiba U-10. Although these meters may measure additional parameters (e.g., temperature, specific conductivity, etc.), this SOP addresses pH measurement only (other capabilities are outlined in the appropriate SOP and manufacturer's individual instrument manuals). This SOP is designed specifically for the measurement of pH in accordance with EPA Method 150.1 and Standard Method 4500-H B which address electrometric pH measurements of drinking, surface, and saline waters, domestic and industrial wastes, and acid rain.

1.2 Quality Assurance Planning Considerations

The end use of the data will determine the quality assurance requirements that are necessary to produce data of acceptable quality. These quality assurance requirements will be defined in the site-specific workplan and/or Quality Assurance Project Plan (QAPP), hereafter referred to as the project plan, or laboratory Quality Assurance Manual (QAM) and may include duplicate or replicate measurements or confirmatory analyses.

1.3 Health and Safety Considerations

The health and safety considerations for the laboratory or site, including both potential physical and chemical hazards, will be addressed in the site-specific Health and Safety Plan (HASP) or the laboratory QAM. In the absence of a site-specific HASP, work will be conducted according to the ENSR Health and Safety Policy and Procedures Manual and/or direction from the Regional Health and Safety Manager.

2.0 RESPONSIBILITIES

2.1 The analyst is responsible for verifying that the pH meter is in proper operating condition prior to use and for implementing the calibration and measurement procedures in accordance with this SOP and the project plan.

2.2 The project manager is responsible for ensuring that project-specific requirements are communicated to the project team and for providing the materials, resources, and guidance necessary to perform the measurements in accordance with this SOP and the project plan.

3.0 REQUIRED MATERIALS

The following materials are necessary for this procedure:

- pH meter
- pH meter manufacturer's instruction manual
- Deionized water
- Clean glass or plastic beakers or cups
- 4.0, 7.0, and 10.0 buffer solutions
- Magnetic stirrer and Teflon-coated stirring bar
- Lint-free tissues
- Mild detergent and/or 10% hydrochloric acid (for use if samples contain oily material or particulate matter)
- National Institute of Standards and Technology (NIST)-traceable thermometer
- Calibration sheets
- Laboratory or field data sheets or logbooks

4.0 METHOD

4.1 Sample Handling, Preservation, and General Measurement Procedures

4.1.1 To achieve accurate pH measurements, samples should be analyzed in the field (preferably within 15 minutes), or as soon as possible after collection. Sample should be collected in plastic or glass containers.

4.1.2 After measuring a sample containing oily material or particulate matter, the electrode must be cleaned by carefully wiping with a lint-free cloth, or washing gently in a mild detergent, followed by a deionized water rinse. If this does not suffice, an additional rinse with 10% hydrochloric acid (followed by deionized water) may be needed.

4.1.3 As temperature can affect the pH measurements obtained, both the pH and the temperature of the sample must be recorded.

4.1.4 Calibration must include a minimum of two points that bracket the expected pH of the samples to be measured. An example of a calibration sheet is presented in Figure 1.

4.1.5 Primary standard buffer salts available from NIST can be purchased and are necessary for situations where extreme accuracy is required. Secondary standard buffers may be purchased as a solution from commercial vendors and are recommended for routine use. Buffers should not be used after their expiration dates as provided by the manufacturer. If the manufacturer does not supply an expiration date or if the buffers are prepared from pH powder pillows, etc., an expiration date of one year from purchase or preparation should be used. All standards must be labeled with manufacturer, lot number, and expiration date.

4.1.6 When using the meter in the laboratory, always place the buffer/sample beaker on the magnetic stirrer, and make sure the stirring bar is rotating during measurements. Rinse the stirring bar as well as the beaker between buffers/samples. CAUTION: The magnetic stirring plate can generate heat when used for an extended period of time, and lead to increased temperature of the buffers/samples.

EXCEPTION: Do not use the magnetic stirrer for acid rain samples. It is crucial not to induce dissolved gases into the sample to be absorbed or desorbed, as this will alter the pH. Stir the sample gently for a few seconds after introducing the electrode, then allow the electrode to equilibrate prior to recording temperature and pH readings.

4.1.7 When the meter is being used in the field, move the probe in a way that creates sufficient sample movement across the sensor; this insures homogeneity of the sample and suspension of solids. If sufficient movement has occurred, the readings will not drift (<0.1 pH units). Rinse the electrode with deionized water between samples and wipe gently with a lint-free tissue.

4.1.8 When measuring the pH of hot liquids, wait for the liquid to cool to 160°F or below.

4.1.9 Fluctuating readings may indicate more frequent instrument calibrations are necessary.

4.1.10 A "low sodium error" electrode may be used for samples with a pH greater than 10, to reduce sodium error.

4.2 Calibration and Measurement Procedures

4.2.1 The pH meter must be calibrated daily before any analyses are performed. The meter should be recalibrated every 12 hours or at the frequency specified in the project plan.

4.2.2 Connect the electrode to the meter. Choose either 7.0 and 10.0 (high range) or 4.0 and 7.0 (low range) buffers, whichever will bracket the expected sample range. Place the buffer in a clean beaker. If the pH is being measured in a laboratory, place the beaker on the magnetic stirrer and place the stirring bar in the beaker. Measure and record the temperatures of the buffers using a calibrated thermometer or automatic temperature compensation (ATC).

4.2.3 Place the electrode into the 10.0 buffer or into the 7.0 buffer. (The calibration procedure described here begins with the buffer of the highest pH selected. The instrument manufacturer's calibration instructions may suggest starting with the buffer of the lowest pH selected).

4.2.4 Adjust the instrument calibration according to the manufacturer's instructions. Discard the buffer and rinse the beaker and stirring bar thoroughly with deionized water.

4.2.5 Refill the beaker with the 7.0 buffer or the 4.0 buffer. Rinse the electrode, gently wipe it with a lint-free tissue, and place it in the selected buffer solution. If the pH is being measured in a laboratory, place the beaker on the magnetic stirrer and place the stirring bar in the beaker. Continue adjusting the instrument calibration according to the manufacturer's instructions. Record the electrode slope (if provided by the instrument) on the calibration sheet (an acceptable slope is between 92 and 102 percent). Measure and record the temperature of the buffer using a calibrated thermometer or ATC. Discard the buffer and rinse the beaker and stirring bar thoroughly with deionized water.

4.2.6 An additional check may be performed, if required by the project plan, by placing the electrode into an additional buffer solution. This buffer should be from a different source than the buffers used for the initial calibration. This buffer should read within +0.2 pH units of the buffer's true pH value.

4.2.7 Verify the calibration every 15 samples and after the last sample of the day with a buffer solution prepared from a different source than that used for

initial calibration. Recalibrate the instrument if the check value varies more than 0.2 pH units from the true value.

4.2.8 The electrode will be rinsed with deionized water and wiped gently with a lint-free tissue between sample analysis.

4.2.9 Recalibrate the instrument if the buffers do not bracket the pH of the samples.

4.2.10 The meter must be recalibrated following any maintenance activities and prior to the next use.

4.3 Troubleshooting Information

If there are any performance problems with the pH meter which result in inability to achieve the acceptance criteria presented in Section 5.0, consult the appropriate section of the meter instruction manual for the checkout and self-test procedures. If the problem persists, consult the manufacturer's customer service department immediately for further instructions.

4.4 Maintenance

4.4.1 Instrument maintenance should be performed according to the procedures and frequencies required by the manufacturer.

4.4.2 The electrode must be stored and maintained according to the manufacturer's instructions.

4.4.3 If an instrument with ATC is being used, the device should be checked on a quarterly basis for accuracy with an NIST thermometer.

5.0 QUALITY CONTROL

5.1 Duplicate measurements of a single sample will be performed at the frequency specified in the project plan. In the absence of project-specific criteria, duplicate measurements should agree within +0.1 pH units.

5.2 The temperature readout of the meter will be checked annually against an NIST-traceable thermometer. If the difference is greater than 0.2°C, the instrument manufacturer will be consulted for instructions. Temperature measurements will be compensated for any difference with the reference thermometer.

5.3 Some regulatory agencies may require the analysis of USEPA Water Supply (WS) or Water Pollution (WP) performance evaluation (PE) samples. These PE samples will be analyzed as required.

6.0 DOCUMENTATION

6.1 All pH meter calibration, temperature check, and maintenance information will be recorded on a daily calibration sheet (Figure 1) or equivalent. pH data may be recorded on the appropriate laboratory or field data sheets or logbooks.

6.2 Calibration documentation must be maintained in a thorough and consistent manner. At a minimum, the following information must be recorded:

- Date and time of calibration
- Signature or initials of person performing the measurement
- Instrument identification number/model
- Expiration dates and batch numbers for all buffer solutions
- Reading for pH 7.0 buffer before and after meter adjustment
- Reading for pH 4.0 or 10.0 buffer before and after meter adjustment
- Readings for all continuing calibration checks
- Temperature of buffers (corrected for any difference with reference thermometer), including units
- Slope reading (if provided by instrument)
- Comments

6.3 Documentation for recorded data must include a minimum of the following:

- Date and time of analysis
- Signature or initials of person performing the measurement
- Instrument identification number/model
- Sample identification/station location
- Temperature (corrected for any difference with reference thermometer) and pH of sample (including units and duplicate measurements)
- Comments

7.0 TRAINING/QUALIFICATIONS

To properly perform pH measurements, the analyst must be familiar with the calibration and measurement techniques stated in this SOP. The analyst must also be experienced in the operation of the meter.

Certain state certification programs require that pH measurements in the field be taken by, or in the presence of, personnel that are qualified under the certification program.

8.0 REFERENCES

APHA-AWWA-WPCF. Standard Methods for the Examination of Water and Wastewater, 17th Edition. 1989.

USEPA. Methods for the Chemical Analysis of Water and Wastes (EPA 600/4-79-020). Revised 1983.

Figure 1 Sample of Instrument Calibration Sheet

Water Quality Instrument Calibration										
Parameter	Instrument		Standard		Standard Value @25°C	Ambient Temp °C/°F	Initial Measured Value	Adjusted Measured Value	Initials Date/Time	Remarks
	Manuf/Model	Serial No.	Manuf/Lot No.	Exp. Date						

All measured values must be corrected for temperature unless the instrument is operated in the ATC mode. DO measurements must also be corrected for barometric pressure.

Temp conversion: °C = 5/9(°F-32)

SOP NUMBER: 7123

Field and Laboratory Measurement of Temperature

Date:	July 1998
Revision Number:	0
Author:	Lori Fuller
Discipline:	Water

1.0 INTRODUCTION

1.1 Purpose and Applicability

This Standard Operating Procedure (SOP) provides basic instructions for routine measurement of temperature using any high quality mercury-filled thermometer or thermistor with analog or digital read-out device such as the Hydrolab, Hydac Multimeter Probe, Seabird 911 CTD, and Horiba U-10. Multimeter instruments used for temperature measurement may measure additional parameters (e.g., conductivity, pH, etc.). This SOP addresses temperature measurement only (other capabilities are outlined in the appropriate SOP). This SOP is designed specifically for the measurement of temperature in accordance with EPA Method 170.1 and Standard Method 2550 B which address thermometric temperature measurement of drinking, surface, and saline waters, and domestic and industrial wastes.

1.2 Quality Assurance Planning Considerations

The end use of the data will determine the quality assurance requirements that are necessary to produce data of acceptable quality. These quality assurance requirements will be defined in the site-specific workplan and/or Quality Assurance Project Plan (QAPP), hereafter referred to as the project plan, or laboratory Quality Assurance Manual (QAM) and may include duplicate or replicate measurements or confirmatory measurements.

1.3 Health and Safety Considerations

The health and safety considerations for the laboratory or site, including both potential physical and chemical hazards, will be addressed in the site specific Health and Safety Plan (HASP) or the laboratory QAM. In the absence of a site-specific HASP, work will be conducted according to the ENSR Health and Safety Policy and Procedures Manual and/or direction from the Regional Health and Safety Manager.

2.0 RESPONSIBILITIES

2.1 The analyst is responsible for verifying that the temperature measuring device is in proper operating condition prior to use and for implementing the calibration and measurement procedures in accordance with this SOP and the project plan.

2.2 The project manager is responsible for ensuring that project-specific requirements are communicated to the project team and for providing the materials, resources, and guidance necessary to perform the measurements in accordance with this SOP and the project plan.

3.0 REQUIRED MATERIALS

The following materials are necessary for this procedure:

- Thermometer or thermistor with analog or digital read-out device
- Manufacturer's instruction manual for the instrument
- National Institute of Standards and Technology (NIST)-traceable thermometer
- Laboratory or field data sheets or logbooks
- Clear glass or plastic containers

4.0 METHOD

4.1 Sample Handling, Preservation, and General Measurement Procedures

To achieve accurate temperature measurements, samples should be analyzed immediately upon collection (preferably within 15 minutes). Samples should be collected in glass or plastic containers.

4.2 Calibration and Measurement Procedures

4.2.1 ENSR-owned temperature measuring devices will, at a minimum, be checked annually as described in Section 5.0. The device will be checked against an NIST-traceable thermometer and the necessary compensation made for the difference in temperature between the two. Rental equipment will be checked by the manufacturer and documentation provided to ENSR. Certain oceanographic instruments such as the Seabird 911 CTD and the Coastal Microqual will be calibrated every 6 months in the manufacturer's laboratory using ITS90 standards. ITS90 is the International Temperature Scale developed by the Joint Panel of Oceanographic Tables and Standards. Post-cruise calibration records received from the manufacturer may be used to post-calibrate field data.

4.2.2 Immerse the thermometer or temperature measuring device into the sample.

4.2.3 Swirl and take a reading when the value stabilizes.

4.2.4 Record the temperature reading to the nearest 0.5° for a thermometer or 0.1° for digital meter-type instruments. For instruments other than the oceanographic instruments, compensate for any difference with the NIST-traceable thermometer.

4.2.5 Temperature measuring devices designed for *in situ* field measurement will be deployed in accordance with the manufacturer's instruction manual. For water-column profiling operations the sensor readings will be recorded manually in a designated field logbook or continuously through the use of a computer. An internal data-logger will be used for recording sensor measurements during moored deployment of a sensor. The frequency of data recording will be specified in the project plan. The location, date, and time of sensor deployment, along with depth (of measurement or mooring) will be recorded in conjunction with the temperature sensor data. Additional documentation requirements are listed in Section 6.2.

4.2.6 Temperature data may be post-calibrated using any of a variety of calibration data including, but not limited to, field calibration points, manufacturer calibration data, and analytical results from samples collected during field deployment of the sensors. The decision criteria for post calibration, and the technique used, will be specified in the project plan, and will be consistent with the manufacturer's recommendations.

4.3 Troubleshooting Information

If there are any performance problems with the meter-type temperature measuring device, consult the appropriate section of the meter instruction manual for the checkout and self-test procedures. If the problem persists, consult the manufacturer's customer service department immediately for further instructions. If performance problems exist with a thermometer, replace the thermometer.

4.4 Maintenance

Instrument maintenance for meter-type temperature measuring devices should be performed according to the procedures and frequencies required by the manufacturer.

5.0 QUALITY CONTROL

5.1 With the exception of the oceanographic instruments, the temperature measuring devices will, at a minimum, be checked against an NIST-traceable thermometer at the frequency stated in section 4.2.1. This verification procedure will be performed as follows:

- Immerse the thermometer or temperature sensor and the NIST-traceable thermometer into a sample.
- Allow the readings to stabilize.
- Record the readings and document the difference.
- Label the thermometer or temperature sensor with the correction value/adjustment and the date the accuracy check was performed.
- Compensate for the difference when sample measurements are taken.

5.2 Duplicate measurements of a single sample will be performed at the frequency stated in the project plan. In the absence of project-specific criteria, duplicate measurements should agree within $\pm 0.5^{o}$C or approximately $\pm 1.0^{o}$F.

6.0 DOCUMENTATION

6.1 Records for checking the accuracy of the thermometer or temperature measuring device (where applicable) will include:

- Date
- Thermometer or meter-type temperature measuring device checked
- Reference thermometer number
- Readings for reference thermometer and thermometer being checked
- Adjustment made for difference in readings
- Initials of analyst

6.2 Documentation for recorded data must include a minimum of the following:

- Date and time of analysis
- Signature or initials of person performing the measurement
- Thermometer ID number or instrument identification number/model
- Sample identification/station location
- Temperature of sample (including units and duplicate measurements) compensated for any difference with the reference thermometer if applicable
- Comments

7.0 TRAINING/QUALIFICATIONS

To properly perform temperature measurements, the analyst must be familiar with the calibration and measurement techniques stated in this SOP. The analyst must also be experienced in the operation of the meter.

Certain state certification programs require that temperature measurements in the field be taken by, or in the presence of, personnel that are qualified under the certification program.

8.0 REFERENCES

APHA-AWWA-WPCF. Standard Methods for the Examination of Water and Wastewater, 17th Edition. 1989.

USEPA. Methods for the Chemical Analysis of Water and Wastes (EPA 600/4-79-020). Revised 1983.

ENSR

SOP NUMBER: 7124

Field and Laboratory Measurement of Specific Conductance

Date:	July 1998
Revision Number:	0
Author:	Lori Fuller
Discipline:	Water

1.0 INTRODUCTION

1.1 Purpose and Applicability

This Standard Operating Procedure (SOP) provides basic instructions for routine calibration and operation of a variety of specific conductance meters, including the Hydrolab, Hydac Multimeter Probe, YSI Model 3500, Coastal Microqual, Horiba U-10 and Seabird 911 CTD. Although these meters may measure additional parameters (e.g., temperature, pH, etc.), this SOP addresses specific conductance measurement only (other capabilities are outlined in the appropriate SOP and manufacturer's individual instrument manuals). This SOP is designed specifically for the measurement of specific conductance in accordance with EPA Method 120.1 and Standard Method 2510 B which addresses specific conductance measurements of drinking, surface, and saline waters, domestic and industrial wastes, and acid rain.

1.2 Quality Assurance Planning Considerations

The end use of the data will determine the quality assurance requirements that are necessary to produce data of acceptable quality. These quality assurance requirements will be defined in the site-specific workplan and/or Quality Assurance Project Plan (QAPP), hereafter referred to as the project plan, or laboratory Quality Assurance Manual (QAM) and may include duplicate or replicate measurements or confirmatory analyses.

1.3 Health and Safety Considerations

The health and safety considerations for the laboratory or site, including both potential physical and chemical hazards, will be addressed in the site-specific Health and Safety Plan (HASP) or the laboratory QAM. In the absence of a site-specific HASP, work will be conducted according to the ENSR Health and Safety Policy and Procedures Manual and/or direction from the Regional Health and Safety Manager.

2.0 RESPONSIBILITIES

2.1 The analyst is responsible for verifying that the specific conductance meter is in proper operating condition prior to use and for implementing the calibration and measurement procedures in accordance with this SOP and the project plan.

2.2 The project manager is responsible for ensuring that project-specific requirements are communicated to the project team and for providing the materials, resources, and guidance necessary to perform the measurements in accordance with this SOP and the project plan.

3.0 REQUIRED MATERIALS

The following materials are necessary for this procedure:

- Specific conductance meter
- Specific conductance meter manufacturer's instruction manual
- Deionized water
- Clean glass beakers or plastic cups
- Potassium chloride (KCl) solution, 0.01M, for determination of cell constant (0.5M KCl for saline water measurements)
- KCl standard at concentration that approximates sample concentrations
- Lint-free tissues
- National Institute of Standards and Technology (NIST)-traceable thermometer
- Calibration sheets
- Laboratory or field data sheets or logbooks

4.0 4.0 METHOD

4.1 Sample Handling, Preservation, and General Measurement Procedures

4.1.1 Specific conductance measurements should be taken soon after sample collection since temperature changes, precipitation reactions, and absorption of carbon from the air can affect the specific conductance. If specific conductance measurements cannot be taken immediately (within 24 hours), samples should be filtered through a 0.45 μ filter, stored at 4°C and analyzed within 28 days.

4.1.2 Report results as specific conductance, μmhos/cm at 25°C.

4.1.3 As temperature can affect the specific conductance measurements obtained, record both the specific conductance and the temperature of the sample.

4.1.4 Secondary standards may be purchased as a solution from commercial vendors. These standards should not be used after their expiration dates as provided by the manufacturer. If the manufacturer does not supply an expiration date or if the standards are prepared from various salts (e.g., KCl), an expiration date of one year from purchase or preparation should be used. All standards must be labeled with manufacturer, lot number, and expiration date.

4.2 Calibration and Measurement Procedures

4.2.1 The specific conductance meter must be calibrated daily (or the calibration checked) before any analyses are performed. However, certain oceanographic instruments such as the Seabird 911 CTD and the Coastal Microqual, which, because of their sensitivity, are calibrated only by the manufacturer (at their specified frequency).

4.2.2 Set up the instrument according to the manufacturer's instructions.

4.2.3 Rinse the probe with deionized water and dry with a lint-free tissue.

4.2.4 Repeat the above procedure for the beakers or cups.

4.2.5 Pour a sufficient amount of the KCl standard (preferably at a concentration that approximates the sample concentrations) into the beaker or cup to cover the probe.

4.2.6 Immerse the probe in the standard.

4.2.7 Record the stabilized specific conductance reading of the standard and the temperature. Adjust the instrument reading (according to the manufacturer's instructions) to display the correct value of the standard. If the meter cannot be adjusted to display the correct value of the standard, the standard should read within 5% of the true value. If the meter reading is between 5% and 15% of the true value, calculate the cell constant using the formula below and correct all subsequent meter readings.

$$\textbf{Cell Constant} = \frac{\textbf{0.01M or 0.5M KCl Standard Conductance}}{\textbf{Conductance Meter Reading}}$$

If the meter reading exceeds the reference standard by greater than 15%, replace the instrument. If the meter does not have automatic temperature compensation (ATC), correct all measurements to 25°C by adding 2% of the reading per degree if the temperature is below 25°C and by subtracting 2% of the reading per degree if the temperature is above 25°C.

4.2.8 An additional check may be performed, if required by the project plan, by placing the probe into an additional KCl standard. This standard should be from a different source than the standard used for the initial calibration. This standard should read within 5% of the true value. Clean and rinse probe and cup as in Sections 4.2.3 and 4.2.4.

4.2.9 Verify the calibration every 15 samples and at the end of the day. Recalibrate or replace the instrument if the check value is not within 15% of the true value.

4.2.10 The probe will be rinsed with deionized water and wiped gently with a lint-free tissue between sample analyses.

4.2.11 The meter must be recalibrated following any maintenance activities and prior to the next use.

4.2.12 Conductivity meters designed for *in situ* field measurement will be deployed in accordance with the manufacturer's instruction manual. For water-column profiling operations the sensor readings will be recorded manually in a designated field logbook or continuously through the use of a computer. An internal data-logger will be used for recording sensor measurements during moored deployment of a sensor. The frequency of data recording will be specified in the project plan. The location, date, and time of sensor deployment, along with depth (of measurement or mooring) will be recorded in conjunction with the DO sensor data. Additional documentation requirements are listed in Section 6.0.

4.2.13 Conductivity data may be post calibrated using any of a variety of calibration data including, but not limited to field calibration points, manufacturer calibration data, and analytical results from samples collected during field deployment of the sensors. The decision criteria for post-calibration, and the technique used will be specified in the project plan, and will be consistent with the manufacturer's recommendations.

4.3 Troubleshooting Information

If there are any performance problems with the specific conductance meter which result in inability to achieve the acceptance criteria presented in Section 5.0, consult the appropriate section of the meter instruction manual for the checkout and self-test procedures. If the problem persists, consult the manufacturer's customer service department immediately for further instructions.

4.4 Maintenance

4.4.1 Instrument maintenance should be performed according to the procedures and frequencies required by the manufacturer.

4.4.2 The probe must be stored and maintained according to the manufacturer's instructions.

4.4.3 If an instrument with ATC is being used, the meter should be checked annually for accuracy with an NIST thermometer.

5.0 QUALITY CONTROL

5.1 The meter must be calibrated daily before use and recalibrated every 12 hours, and will not be used for sample determinations of specific conductance unless the initial check standard value is within 5% of the true value.

5.2 Oceanographic instruments will be calibrated every 6 months in the manufacturer's laboratory using the functional relationship between salinity and conductance, temperature and pressure as defined by the Practical Salinity Scale of 1978 (PSS-78). Post-cruise calibration records received from the manufacturer will be used to post calibrate field data.

5.3 Duplicate measurements of a single sample may be performed at the frequency specified in the project plan. In the absence of project-specific criteria, duplicate measurements should agree within 10%.

5.4 The temperature readout of the meter will be checked against an NIST-traceable thermometer at least quarterly. If the difference is greater than 0.2°C, the instrument manufacturer will be consulted for instructions. Temperature measurements will be compensated for any difference with the reference thermometer.

5.5 Some agencies may require the analysis of USEPA Water Pollution (WP) performance evaluation (PE) samples. These PE samples will be analyzed as required.

6.0 DOCUMENTATION

6.1 All specific conductance meter calibration, temperature check, and maintenance information will be recorded on the daily calibration sheet. (An example is presented as Figure 1). Specific conductivity data may be recorded on the appropriate laboratory or field data sheets or logbooks.

6.2 Calibration documentation must be maintained in a thorough and consistent manner. At a minimum, the following information must be recorded:

- Date and time of calibration
- Signature or initials of person performing the measurement
- Instrument identification number/model
- Expiration dates and batch numbers for all standards
- Reading for standard before and after meter adjustment
- Readings for all continuing calibration checks
- Temperature of standards (corrected for any difference with reference thermometer)
- Cell constant value
- Comments

6.3 Documentation for recorded data must include a minimum of the following:

- Date and time of analysis
- Signature or initials of person performing the measurement
- Instrument identification number/model
- Sample identification/station location
- Temperature (corrected for any difference with reference thermometer) and conductance of sample (including units and duplicate measurements) Note: show all calculations for converting instrument reading to μmhos/cm if the instrument provides readings in any other units. Useful conversions are: 1 mS/m = 10 μmho/cm or 1 μmho/cm = 0.1 mS/m.
- Comments

7.0 TRAINING/QUALIFICATIONS

To properly perform specific conductance measurements, the analyst must be familiar with the calibration and measurement techniques stated in this SOP. The analyst must also be experienced in the operation of the meter.

Certain state certification programs require that specific conductance measurements be taken in the field by, or in the presence of, personnel that are qualified under the certification program.

8.0 REFERENCES

APHA-AWWA-WPCF. Standard Methods for the Examination of Water and Wastewater, 17th Edition. 1989.

USEPA. Methods for the Chemical Analysis of Water and Wastes, (EPA 600/4-79-020). Revised 1983.

Figure 1 Example of Instrument Calibration Sheet

Water Quality Instrument Calibration

Parameter	Instrument		Standard		Standard Value @25°C	Ambient Temp °C/°F	Initial Measured Value	Adjusted Measured Value	Initials Date/Time	Remarks
	Manuf/Model	Serial No.	Manuf/Lot No.	Exp. Date						

All measured values must be corrected for temperature unless the instrument is operated in the ATC mode. DO measurements must also be corrected for barometric pressure.

Temp conversion: °C = 5/9(°F-32)

SOP NUMBER: 7130

Groundwater Sample Collection from Monitoring Wells

Date:	November, 1999
Revision Number:	2
Author:	Charles Martin
Discipline:	Geosciences

1.0 PURPOSE AND APPLICABILITY

1.1 Purpose and Applicability

This standard operating procedure (SOP) is concerned with the collection of valid and representative samples of groundwater from monitoring wells. The scope of this document is limited to field operations and protocols applicable during groundwater sample collection.

This SOP is written in a broad-based manner and considers the application of a variety of sampling equipment in the collection of representative groundwater samples. Respective state and/or federal agency regulations may require specific types of equipment to be used when applying this SOP to a particular project. The project manager should review the applicable regulatory requirements, if any, prior to the start of the field sampling program. Deviations from this SOP to accommodate regulatory requirements should be reviewed in advance of the field program and documented in the project work plan.

1.2 Quality Assurance Planning Considerations

Sampling personnel should follow specific quality assurance guidelines as outlined in the site-specific QAPP. Proper quality assurance requirements should be provided which will allow for collection of representative samples from representative sampling points. Quality assurance requirements typically suggest the collection of a sufficient quantity of quality control (QC) samples such as field duplicate, equipment and/or field blanks and matrix spike/matrix spike duplicate (MS/MSD) samples. These requirements should be outlined in the QAPP. Additional information regarding quality assurance sample collection relevant to groundwater sampling is contained in Section 5.0 of this SOP.

1.3 Health and Safety Considerations

Groundwater sampling may involve chemical hazards associated with the materials being sampled. Adequate health and safety measures must be taken to protect project sampling personnel from potential chemical exposures or other hazards.

These measures must be addressed in the project Health and Safety Plan (HASP). This plan must be approved by the project Health and Safety Officer before work commences, must be distributed to all personnel performing sampling, and must be adhered to as field activities are performed.

2.0 RESPONSIBILITIES

2.1 Project Manager

The project manager is responsible for ensuring that project-specific requirements are communicated to the project team and for providing the materials, resources, and guidance necessary to perform the measurements in accordance with this SOP and the project-specific work plan.

2.2 Sampling Technician

It is the responsibility of the sampling technician to be familiar with the sampling procedures outlined within this SOP and with specific sampling, quality assurance, and health and safety requirements outlined within project-specific work plans (Sampling Plan, HASP, QAPP). The sampling technician is responsible for collection of groundwater samples and for proper documentation of sampling activities as samples are being collected.

3.0 REQUIRED MATERIALS

Groundwater sampling objectives may vary significantly between projects. Project objectives should be defined within the project-specific work plans. The list of required materials below identifies the types of equipment which may be used for a range of groundwater sampling applications. From this list, a project-specific equipment list should be selected based upon project objectives and other factors such as the depth to groundwater, well construction, required purge volumes, and analytical parameters, among others. The various types of sampling equipment which may be used include:

<u>Well Purging Equipment</u>

- Bailers
- Bladder pumps
- Submersible pumps
- Peristaltic pumps
- Centrifugal Pumps
- Waterra™ pumps

Field Instruments

- Individual or multi-parameter meter(s) to measure temperature, pH, specific conductance, dissolved oxygen (DO) oxidation reduction potential (ORP), and/or turbidity
- Water level measuring device
- Interface probe or product detection paste

Sampling Equipment

- Reusable or disposable bailers
- Peristaltic pump
- Bladder pump

Sample Preparation Equipment

- Filtration equipment
- Intermediate containers
- Sample kit (i.e., bottles, labels, preservatives, custody records, cooler)

General Equipment

- Project-specific sampling plans (SAP, QAPP, HASP)
- Sample collection records
- Field notebook/pen
- Waterproof marker pens
- Deionized water dispenser bottler
- Sample cup
- Buckets
- Coolers, or sample shuttles
- Instrument calibration solutions
- Power source (generator of 12V marine battery)
- Equipment decontamination supplies (refer to SOP 7600)
- Health and safety supplies
- First-Aid kit
- Tool box

Expendable Materials

- Deionized water supply

- Disposable bailer string (nylon or polypropylene)
- 0.45 micron filters
- Paper towels
- Plastic sheeting
- Ice/blue ice for sample preservation
- Disposable latex powder-free glove liners
- Disposable nitrile gloves
- Plastic trash bags
- Ziplock® bags

This equipment list was developed to aid in field organization and should be used in preparation for each sampling event. Depending on the site-specific sampling plan, additional material and equipment may be necessary and should be determined before the scheduled sampling event. Similarly, not all of the items shown in this list may be necessary for any one sampling event.

Additional SOPs are also available which provide procedures for different aspects of groundwater sampling. These SOPs include:

- ENSR SOP 7121, Field and Laboratory Measurement of pH
- ENSR SOP 7122, Field and Laboratory Measurement of Dissolved Oxygen
- ENSR SOP 7123, Field and Laboratory Measurement of Temperature
- ENSR SOP 7124, Field and Laboratory Measurement of Specific Conductance
- ENSR SOP 7125, Field and Laboratory Measurement of Turbidity
- ENSR SOP 7131, Field Filtration of Water Samples for Inorganics
- ENSR SOP 7510, Packaging and Shipment of Samples
- ENSR SOP 7600, Decontamination of Equipment

4.0 METHOD

4.1 Instrument Calibration

Field instruments will be calibrated according to the requirements of the project-specific plan and water quality SOPs (see Section 3.0).

4.2 Sampling Preparation

Before opening the well, a clean working surface shall be set up around the well head using a plastic sheet with slit cut in the middle. Prior to opening the well, the required health and safety gear (as specified in the HASP) shall be donned. This, at a minimum, usually means wearing gloves to limit the potential for exposure to contaminants as well as reduce the potential for handling-induced contamination of sampling equipment.

4.3 Well Security and Condition

At each monitoring well location, observe the conditions of the well and surrounding area. The following information shall be noted on the Groundwater Sample Collection Record (Attachment 1 or 2) or in the field notebook:

- Condition of the wells identification marker
- Condition of the well lock and associated locking cap
- Integrity of the well - protective outer casing, obstructions or kinks in the well casing, presence of water in the annular space, and the top of the interior casing
- Condition of the general area surrounding the well

4.4 Measuring Point Determination

Before collecting a water level measurement, check for an existing measuring point (notch, or other visible mark) established either at the time of well installation or by the latest survey. Generally, the measuring point is referenced from the top of the well casing (TOC), not the protective casing. If no measuring point exists, a measuring point should be established, clearly marked, and identified on the Groundwater Sample Collection Record or the field logbook. The same measuring point should be used for subsequent sampling events.

4.5 Free Product Determination

Wells that may potentially contain free product should be assessed for product with an interface probe or product detection paste. Interface probes generally operate on the same principle as a water level tape although they are designed to register water and product levels usually with different audible tones. Product paste generally is used in combination with some type of measuring tape which is lowered into the well with a coating of paste applied to it. Wells containing free product are generally not used for groundwater sampling, since the concentration of contaminants present in the free product can adversely effect the quality of the water sample, lending to a non-representative water sample.

4.6 Water Level Measurement

To obtain a water level measurement, lower the probe of a water level measuring device into the well until the audible sound of the unit is detected or the light on an electronic sounder illuminates. At this time the precise measurement should be determined (to nearest 0.01 feet) by repeatedly raising and lowering the tape to converge on the exact measurement. Obtain the reading of the TOC measuring point. The water level measurement should be entered on the Groundwater Sample Collection Record or in the field records.

The measurement device shall be decontaminated immediately after use with a non-phosphatic detergent and rinsed with distilled water. Generally, only that portion of the tape which enters the water table should be cleaned. It is important that the measuring tape is never placed directly on the ground surface or allowed to become kinked. Measuring devices, including interface probes, which come into contact with free product will likely require more thorough decontamination (see SOP 7600).

4.7 Purge Volume Calculation

Wells designated for sampling require purging to remove stagnant water in the well. A single casing volume of groundwater will be calculated after measuring the length of the water column and checking the well casing diameter. The Groundwater Sample Collection Record provides information used to compute the casing volume, which includes: a diagram, a numerical conversion table, and the standard calculation. The volume of standing water in the well (ie., one purge volume) should be entered on the Groundwater Sample Collection Record.

4.8 Well Purging Methods and Procedures

4.8.1 Objectives

Prior to sample collection, purging must be performed for all groundwater monitoring wells to remove stagnant water from within the casing and gravel pack and to ensure that a representative groundwater sample is obtained.

There are three general types of non-dedicated equipment used for well purging and include: bailers, surface pumps and down-well pumps. The purge method and equipment selected should be specified in the project-specific work plans.

NOTE: This SOP only describes the most common equipment and methods used for purging. Other purging equipment, as well as dedicated equipment,

can be used provided that the method employed does not have an adverse affect on the overall quality of the groundwater.

Regardless of the purge method, purge water temperature, pH, and specific conductance will be monitored at predetermined purge volumes and recorded on the Groundwater Sample Collection Record. Additional water quality parameters may be required by the project-specific sampling plan. In general, purging will be considered complete following the withdrawal of at least 3 to 5 well volumes of groundwater and when all field parameters have stabilized to within 10% of their preceding measurements.

Purging a well to dryness may occur under some low-yield conditions. When the well recovers, a cascading effect may occur within the screened zone which can volatilize some organic compounds. This may be considered inappropriate by regulatory agencies when volatile organic compounds (VOC) are the target analyte of interest. Purging a well to dryness, then sampling after it has recovered may be acceptable for other target analytes, however. Under low yield conditions, low-flow sampling pumps such as bladder pumps may be required for VOC sample collection.

4.8.2 Bailing

General

Bailing is often the most convenient method for well purging especially if only a small volume of purge water is required during the purge routine. Bailers are constructed using a variety of materials including PVC, polyethylene, stainless steel, and Teflon®. Teflon® bailers are generally most "inert" and are available in reusable and disposable form. Disposable polyethylene bailers are relatively inert and inexpensive. Reusable stainless steel and PVC bailers must be decontaminated between uses. Most commercially available bailers are constructed to fit into a 2-inch diameter well, although other bailer diameters are available.

Waterra™ foot valves are essentially bailer check valves which manually thread onto the bottom of standard pump tubing (polyethylene, teflon). The foot valves are commercially available in a variety of diameters in stainless steel, Teflon®, and high-density plastic (Delrin). The foot valves operate by manually or mechanically raising and lowering the valve assembly within the water column which raises the water level within the discharge tube. Flow rates usually in the vicinity of 1 gallon per minute can be achieved with these devices.

Measurements of the pumping rate, temperature, pH, and specific conductance (and/or other parameters as required) should be made after each purge volume is removed and documented on the Groundwater Sample Collection Record or in the field logbook. Samples may be collected after the required purge volume has been withdrawn and the field parameters have stabilized to within 10% of their preceding measurement. Project-specific sampling objectives may require that the sample be collected with a bailer.

Bailing presents two potential problems with well purging. First, increased suspended solids may be present in samples as a result of the turbulence caused by raising and lowering the bailer through the water column. High solids concentrations may affect sample representativeness. Second, bailing may be less feasible for deep wells or wells which require a large volume of water to be removed during purging because of the time involved with continuous insertion and removal/emptying of the bailer.

Bailing Procedure

Obtain a clean bailer and a spool of clean polypropylene or nylon bailer cord. Uncover the top end of the bailer and tie a bowline knot, or equivalent, through the bailer loop. Test the knot and the bailer itself to ensure that all knots and parts are secure prior to inserting the bailer into the well.

Remove the protective wrapping from the bailer, and lower the bailer to the bottom of the monitoring well and cut the cord at a proper length. Bailer rope should never touch the ground surface at any time during the purge routine. Tie a hand loop at the end of the bailer cord.

Raise the bailer by grasping a section of cord using each hand alternatively in a "rocking" action. This method requires that the sampler's hands be kept approximately 2-3 feet apart and that the bailer rope is alternately looped onto or off each hand as the bailer is raised and lowered.

Grab the bailer with one hand as it emerges from the well. Pour the bailed groundwater from the bailer into a graduated bucket to measure the purged water volume. Repeat this procedure until one complete purge volume of water is removed from the well.

At the end of one complete well purge volume, place a small of purged water into a sample cup. Measure temperature, pH and specific conductance (and for other assigned parameters) and record the results on the Groundwater Sample Collection Record or in the field logbook. Samples may be collected

after the required purge volume has been withdrawn and the specific field parameters have stabilized to within 10% of their preceding measurement.

4.8.3 Surface Pumps

General

Well purging using pumps located at the ground surface can be performed with peristaltic or centrifugal pumps if the water level in the well is within approximately 20 feet of the top of the well.

Peristaltic pumps provide a low rate of flow typically in the range of 0.02-0.2 gallons/minute (75-750 ml/min). For this reason, peristaltic pumps are not particularly effective for well purging. Peristaltic pumps are suitable for purging situations where disturbance of the water column must be kept minimal for particularly sensitive analyses.

Centrifugal pumps are designed to provide a high rate of pumping, in the range of 5 to 40 gallons/minute (gpm), depending on pump capacity. Discharge rates can also be regulated somewhat, provided the pump has an adjustable throttle. These pumps also require polyethylene or teflon-lined polyethylene tubing as suction line. The pump may also require priming to initiate flow.

Peristaltic Pump Procedure

Attach a new suction and discharge line to the peristaltic pump. Silicon tubing must be used through the pump head and must meet the pump head specifications. A second type of tubing may be attached to the silicon tubing for use as the suction and discharge lines. The secondary tubing material, usually consisting of polyethylene or teflon-lined polyethylene, should be compatible with the target analytes. The suction line must be long enough to extend to the static groundwater surface and reach further should drawdown occur during pumping.

Measure the length of the suction line and lower it down the monitoring well until the end is in the upper foot or more of the water column. Start the pump and direct the discharge into a graduated bucket. Adjust the pumping rate with the speed control knob so that a smooth flowing discharge is attained.

Measure the pumping rate in gallons per minute by recording the time required to fill a calibrated bucket. The pumping shall be monitored to assure

continuous discharge. If drawdown causes the discharge to stop, the suction line will be lowered very slowly further down into the well until pumping restarts.

Measurements of temperature, pH and specific conductance (and/or other assigned parameters) should be made after each well purge volume and documented on the Groundwater Sample Collection Record or in the field logbook. Samples may be collected after the required purge volume has been removed and the specific field parameters have stabilized to within 10% of their preceding measurement. Project-specific sampling objectives may require that the sample be collected with a bailer.

Centrifugal Pump Procedure

Attach a new suction and discharge line to the centrifugal pump. Start the pump and record the stabilized rate of discharge. As with other well purging systems, measurement of temperature, pH, and specific conductance (or other parameters as required) will be made after each well purge volume has been removed. These measurements shall be recorded on the Groundwater Sample Collection Record or in the field logbook. Samples may be collected after the required purge volume has been removed and the field parameters have stabilized to within 10% of their preceding measurement. Project-specific sampling objectives may require that the sample be collected with a bailer.

4.8.4 Down-Well Pumps

General

Groundwater withdrawal using non-dedicated down-well pumps may be performed with a submersible pump or a bladder pump.

Electric submersible pumps provide an effective means for well purging and in some cases sample collection. Submersible pumps are particularly useful for situations where the depth to water table is greater than 20 feet and where the depth or diameter of the well requires that a large purge volume be removed before sample collection.

Commonly available submersible pumps include the Johnson-Keck pump model SP-82, the Grunfos Ready-Flow 2 pump, and disposable marine galley pumps, all of which are suited for operation in 2-inch or larger internal diameter wells.

Recently, the use of bladder pumps (positive gas-displacement pumps) has been promoted by the EPA for use in well purging and sampling primarily because the pumps can be operated at low flow rates (less than 1 liter per minute). Bladder pumps generally reduce the potential turbidity of the sample and theoretically reduce the potential for loss of VOC constituents, ultimately providing a more representative groundwater sample. Use of bladder pumps may require additional time for purging and sampling because of the low flow rate. Please note, however, that when using bladder pumps, it may not be necessary to purge an entire well volume of water prior to each check of the water quality parameters. Well purging is accomplished at such a low rate that, theoretically, the influent flow into the pump represents groundwater flow through the well screen, thereby eliminating the requirement for purging several entire well volumes of water before sample collection.

Bladder pumps usually consist of a stainless steel pump housing with an internal teflon or polyethylene bladder. Discharge tubing is generally made from teflon, polyethylene, or teflon-lined polyethylene. The pump is operated by lowering it into the water column within the well screen, then pulsing air into the bladder with an air compressor and pump controller unit. Pumps and controllers are often not interchangeable between manufacturers, therefore, it is usually necessary to have both items provided by the same manufacturer. Pump bladders are generally field-serviceable and replaceable.

A check of well condition may be required prior to inserting any down-well pump if the well has not been sampled for some time or if groundwater quality conditions are not known. The well condition check should include a check of casing plumbness as a bent well casing could cause a pump to get stuck. Casing plumbness can be checked by lowering a clean cylindrical tube with the approximate pump dimensions into the well. If the well casing is not plumb then an alternative purging method should be used.

The well inspection should also include a check of air quality or headspace conditions within the well for potentially explosive gasses and a check for free product which could foul the pump. Well casing headspace conditions can be monitored with a photoionization detector (PID) and/or an explosimeter for the presence of potentially explosive gasses. If potentially hazardous conditions exist, then an alternative purging method should be used. In general, it is rare for explosive conditions to be present.

The presence of free product should be determined before inserting the submersible pump into the well because free product may contaminate the pump's internal mechanisms making it extremely difficult to decontaminate.

An interface probe should be used to check for free product. Refer to Section 4.5 of this SOP for additional information on free product determination.

Electric Submersible Pump Procedure

Once the above well conditions have been assessed, and assuming its safe to proceed, slowly lower the submersible pump with attached discharge line into the monitoring well taking notice of any roughness or restriction within the well riser pipe. The pump should be placed in the uppermost section of the static water column of the monitoring well. The power cord should be attached to the discharge line with an inert material (i.e., zip-ties) to prevent the power cord from getting stuck between the pump, discharge line, and the well casing. Secure the discharge line and power cord to the well casing, using tape or a clamp, taking care not to crimp or cut either the discharge line or power cord.

Connect the power cord to the power source (i.e., rechargeable battery pack, auto battery, or generator) and turn the pump on. Voltage and amperage meter readings on the pump controller (if provided) should be monitored closely during purging. The operations manual for the specific pump used should be reviewed regarding changes in voltage/amperage and the potential impacts on pump integrity. Pumping should be discontinued if warning conditions occur and/or if the well is pumped to where drawdown falls below the pump's intake level.

If drawdown continues to the extent that the well is pumped dry, the pump should be shut off and the well allowed to recharge. This on/off cycle may be necessary in order to purge the well properly.

Measurements of the pumping rate, temperature, pH, and specific conductance (and/or other required parameters) should be made after each purge volume is removed and documented on the Groundwater Sample Collection Record or in the field logbook. Samples may be collected after the required purge volume has been withdrawn and the field parameters have stabilized to within 10% of their preceding measurement. Project-specific sampling objectives may require that the sample be collected with a bailer.

Bladder Pump Procedure

To operate the bladder pump system, the pump and discharge line should be lowered into the well close to the bottom of the well screen, then secured to

the well casing with a clamp. The air compressor should then be turned on to activate pumping. The pump controller is used to vary the discharge rate to the required flow.

Measurements of the pumping rate, temperature, pH, and specific conductance (and/or other required parameters) should be made at periodic intervals while water is removed and documented on the Groundwater Sample Collection Record or in the field logbook. Samples may be collected after the required field parameters have stabilized to within 10% of their preceding measurement. Generally, because of the low flow rate, samples are usually obtained from the bladder pump discharge line.

4.9 Sample Collection Methods and Procedures

4.9.1 Objectives

Groundwater samples can be collected using similar methods employed for purging, provided these methods do not adversely affect the quality of the groundwater. These methods include bailing, surface pumping and down-well pumping.

In most cases during sampling, groundwater will be transferred to the appropriate containers directly for the discharge source. During transfer, discharge tubing and other equipment shall not contact the inside of the sample containers. In addition, a clean pair of nitrile or latex gloves will be worn during sample collection and handling.

As a general rule of thumb, samples should be collected in order of decreasing volatilization of the target parameters. The preferred order of sample collection is as follows: volatile organic compounds, extractable organic compounds (e.g., semivolatile organic compounds, PCBs, pesticides), metals, and general water chemistry (ions and turbidity).

4.9.2 Bailers

The methods and procedures described in Section 4.9.2 also apply to collecting groundwater samples with a bailer. If a bailer was used to purge the well, the same bailer may be used for sampling. If other well purging equipment was used, a decontaminated or new disposable bailer should be used for sampling.

When volatile organic compounds are the target sampling parameter, a bottom discharge tip should be used during sample transfer. A discharge tip restricts the outflow of the sample from the bailer and diminishes the potential for volatilization. Reusable bailers may require a special screw-on tip fitted with a bottom discharge top. Disposable bottom discharge tips are usually supplied with disposable bailers.

Bailer cord shall be discarded after sampling is completed. Disposable bailers should only be used in one well. Reusable bailers should be appropriately decontaminated between uses.

4.9.3 Surface Pumps

The methods and procedures described in Section 4.9.3 for peristaltic and centrifugal pumps also apply to groundwater sample collection.

Peristaltic Pumps

Peristaltic pumps equipped with the appropriate type tubing will be used to collect groundwater from wells in which the water resides at a depth less than 20 feet. Sample bottles shall be filled directly from the pump's discharge line and care shall be taken to keep the discharge tube from contacting the sample container.

Groundwater samples requiring filtration prior to placement in sample containers can be placed in intermediate containers for subsequent filtration, or may be filtered directly with in-line disposable 0.45-micron filters, as described in SOP 7131.

After sampling is complete, all used tubing and filters shall be disposed of appropriately.

Centrifugal Pumps

Centrifugal pumps are generally not recommended for use in sample collection, especially when volatile organic compounds are the target analyte of interest. Samples for other analytes, however, may be obtained with use of an in-line sample trap. It is suggested that if samples cannot be obtained before going through the pump, that samples be obtained by using a bailer once purging is complete and pumping has ceased. Collecting samples from the pump discharge is not recommended.

After sampling is complete, all suction line tubing should be disposed of properly.

4.9.4 Down-Well Pumps

Electric Submersible Pump

Using the pump methods described in Section 4.9.4, groundwater samples can be collected directly from the pump discharge line, provided the discharge line is composed of inert material. Sample bottles will be filled directly from the discharge line of the pump. This method is generally not recommended for collection of volatile organic samples.

After sampling is complete, the pump, discharge line and power cord shall be decontaminated according to the procedures contained in SOP 7600 and/or disposed of as required by the project-specific work plan.

Bladder Pumps

Groundwater samples, including those collected for VOC analysis, may be collected directly from the pump discharge tubing under active pumping conditions. Sample bottles will be filled directly from the discharge line of the pump.

After sampling is complete, the pump, discharge line and power cord shall be decontaminated according to the procedures outlined in SOP 7600 and/or disposed of as required by the project-specific work plan.

4.10 Sample Filtration

The filtration of groundwater samples will be performed in accordance with SOP 7131. Groundwater samples collected for total dissolved metals analyses will be filtered prior to being placed in sample containers and properly preserved. Groundwater filtration will be performed using a peristaltic pump and a 0.45-micron in-line water filter. Disposable filters are commonly available in 0.45-micron size. Low-capacity or high-capacity cartridges are available and may be selectively used based on sample turbidity.

The filtration of groundwater samples shall be performed either directly from the pump discharge line or from laboratory-supplied intermediate containers. In either case, well purging shall be performed first. Fresh groundwater shall then be filtered directly into sample containers.

4.11 Sample Handling

All samples collected should be packaged and handled according to SOP 7510 and the project-specific sampling plan. Preservatives should be used where analytical methods require preservation. The QAPP will indicate the type of sample preservation necessary.

5.0 QUALITY CONTROL

5.1 Field Blank/Equipment Blank Sample Collection

Field blank samples serve as a quality assurance check of equipment and field conditions at the time of sampling. Field blank samples are usually prepared by transferring analyte-free water into a clean set of sample containers, then analyzing it as a sample. Sometimes, the analyte-free water is transferred over or through the sampling device before it is placed into the sample containers. This type of field blank sample is known as an equipment blank. The QAPP contains specific information regarding the type and number of field blanks or equipment blanks required for collection.

5.2 Field Duplicate Sample Collection

Field duplicate samples are collected for the purpose of providing two sets of results for comparison. These samples are used to assess precision. Duplicate samples are usually prepared by splitting the sample into two sets of sample containers, then analyzing each set as a separate sample. The QAPP contains specific information regarding the type and number of duplicate samples for collection.

5.3 MS/MSD Sample Collection

MS/MSDs provide information about the effect of the sample matrix on digestion and measurement methodology. For samples submitted for MS/MSD analysis, triple sample volume is generally required (contact the analytical laboratory for information specific to the project analytical parameters). The QAPP contains specific information regarding the frequency of MS/MSD samples.

6.0 DOCUMENTATION

Specific information regarding sample collection should be documented in several areas: the sample chain-of-custody record, sample collection record, field notebook, and sample

labels, tags. Additional information regarding each form of documentation is presented in the following paragraphs:

6.1 Sample Chain-of-Custody Record

This ENSR standard form requires input of specific information regarding each collected sample for laboratory analytical purposes. The information requested includes site name and location, project number, field notebook reference, collection date and type of analysis requested. Each sample submitted for analysis is also listed individually using its field identification number, number and type of container, and requested analyses (see SOP 7510).

6.2 Groundwater Sample Collection Record

This form (Attachment 1 or 2) requires input of specific information regarding the collection of each individual sample including sample identification, water quality parameters, collection method, and containers/preservation requirements.

6.3 Field Logbook

This logbook should be dedicated to the project and should be used by field personnel to maintain a general log of activities throughout the sampling program. This logbook should be used in support of, and in combination with, the sample collection record. Documentation within the logbook should be thorough and sufficiently detailed to present a concise, descriptive history of the sample collection process.

6.4 Sample Labels/Tags

Sample labels shall be completed at the time each sample is collected and attached to each sample container. Labels will include the information listed below.

- Client or project name/project number
- Sample number
- Sample designation
- Analysis type
- Preservative
- Sample collection date
- Sample collection time
- Sampler's name

The project-specific work plan may also require the use of sample tags which generally contain the same information as the sample labels. Sample tags, if used, should be tied to each sample bottle with wire ties.

7.0 TRAINING/QUALIFICATIONS

Groundwater sample collection is a relatively involved procedure requiring formal training and a variety of equipment. It is recommended that initial sampling attempts be supervised by more experienced personnel. Sampling technicians should be health and safety certified as specified by OSHA (29 CFR 1910.120(e)(3)(i)) to work on sites where hazardous waste materials are considered to be present.

8.0 REFERENCES

EPA, Handbook for Sampling and Sample Preservation of Water and Wastewater, EPA-600/4-82-029, September 1982.

EPA, RCRA Groundwater Monitoring Technical Enforcement Guidance, November 1992.

Geotrans, Inc., RCRA Permit Writer's Manual, Groundwater Protection, prepared for the U.S. EPA, Contract No. 68-01-6464, October 1983.

Code of Federal Regulations, Chapter 40 (Section 261.4(d)).

Attachment 1 Ground Water Sample Collection Record
Attachment 2 Low Flow Ground Water Sample Collection Record

Attachment 1
Groundwater Sample Collection Record

ENSR

Well/Piezo ID:

Ground Water Sample Collection Record

Client: _____ Date: _____
Project No: _____ Time: Start _____ am/pm
Site Location: _____ Finish _____ am/pm
Weather Conds: _____ Collector(s) _____

WATER LEVEL DATA: (measured from Top of Casing) Well ☐ Piezometer ☐

a. Total Well Length _____ c. Casing Material _____ e. Length of Water Column _____

b. Water Table Depth _____ d. Casing Diameter _____ f. Calculated Well Volume (see back) ____

WELL PURGING DATA

a. Purge Method _____

b. Acceptance Criteria defined (from workplan)
- Minimum Required Purge Volume (@ _____ well volumes) _____
- Maximum Allowable Turbidity _____ NTUs
- Stabilization of parameters _____ %

c. Field Testing Equipment Used: Make Model Serial Number

_____

_____

d. Field Testing Equipment Calibration Documentation Found in Field Notebook # _____ Page #_____

Time	Volume Removed (gal)	T° (C/F)	pH	Spec. Cond (umhos)	Turbidity (NTUs)	DO	Color	Odor	Other

e. Acceptance criteria pass/fail

	Yes	No	N/A
Has required volume been removed	☐	☐	☐
Has required turbidity been reached	☐	☐	☐
Have parameters stabilized	☐	☐	☐

If no or N/A - Explain below.

_____

_____

SAMPLE COLLECTION: Method: _____

Sample ID	Container Type	No. of Containers	Preservation	Analysis	Time

Comments _____

_____

Signature _____ Date _____

Attachment 2
Low Flow Ground Water Sample Collection Record

ENSR

Well ID:

Low Flow Ground Water Sample Collection Record

Client: _____ Date: _____ Time: Start _____ am/pm
Project No: _____ Finish _____ am/pm
Site Location: _____
Weather Conds: _____ Collector(s): _____

1. WATER LEVEL DATA: (measured from Top of Casing)

a. Total Well Length _____ c. Length of Water Column _____ (a-b)

Casing Diameter/Material _____

b. Water Table Depth _____ d. Calculated System Volume (see back) _____

2. WELL PURGE DATA

a. Purge Method: _____

b. Acceptance Criteria defined (see workplan)

- Temperature	3%	-D.O.	10%
- pH	± 1.0 unit	- ORP	± 10mV
- Sp. Cond.	3%	- Drawdown	< 0.3'

c. Field Testing Equipment used: Make _____ Model _____ Serial Number _____

Time (24hr)	Volume Removed (Liters)	Temp. (°C)	pH	Spec. Cond. (µS/cm)	DO (mg/L)	ORP (mV)	Turbidity (NTU)	Flow Rate (ml/min)	Drawdown (feet)	Color/Odor

(continued on back)

d. Acceptance criteria pass/fail

	Yes	No	N/A
Has required volume been removed	☐	☐	☐
Has required turbidity been reached	☐	☐	☐
Have parameters stabilized	☐	☐	☐

If no or N/A - Explain below.

_____

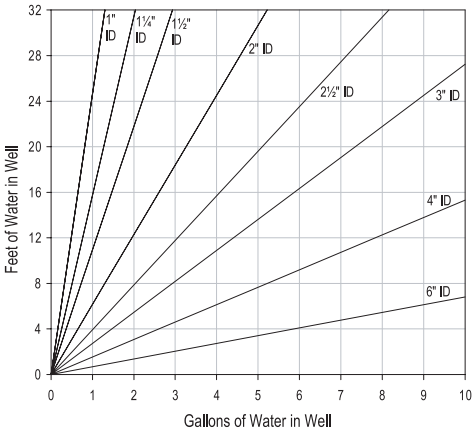
3. SAMPLE COLLECTION: Method: _____

Sample ID	Container Type	No. of Containers	Preservation	Analysis Req.	Time

Comments _____

Signature _____ Date _____

Purge Volume Calculation



Volume / Linear Ft. of Pipe		
ID (in)	Gallon	Liter
0.25	0.0025	0.0097
0.375	0.0057	0.0217
0.5	0.0102	0.0386
0.75	0.0229	0.0869
1	0.0408	0.1544
1.25	0.0637	0.2413
1.5	0.0918	0.3475
2	0.1632	0.6178
2.5	0.2550	0.9653
3	0.3672	1.3900
4	0.6528	2.4711
6	1.4688	5.5600

(continued from front)

Time (24 hr)	Volume Removed (Liters)	Temp (°C)	pH	Spec. Cond. (µS/cm)	DO (mg/L)	ORP (mV)	Turbidity (NTU)	Flow Rate (ml/min)	Drawdown (ft)	Color/Odor

ENSR

SOP NUMBER: 7510

Packaging and Shipment of Environmental Samples

Date:	4th Qtr. 1999
Revision Number:	4
Author:	Charles Martin
Discipline:	Geosciences

1.0 PURPOSE AND APPLICABILITY

1.1 Purpose and Applicability

This Standard Operating Procedure (SOP) describes the procedures associated with the packaging and shipment of environmental samples. Two general categories of samples exist: environmental samples consisting of water and soil submitted for routine environmental testing, and waste material samples which include non-hazardous solid wastes and/or hazardous wastes as defined by 40 CFR Part 261 submitted for environmental testing or bench/pilot-scale treatability testing. Packaging and shipping procedures will differ for the two sample categories.

This SOP is applicable to packaging and shipment of environmental samples submitted for routine environmental testing. Environmental samples are not considered a hazardous waste by definition; therefore, more stringent Department of Transportation (DOT) regulations regarding sample transportation do not apply. Environmental samples do, however, require fairly stringent packaging and shipping measures to ensure sample integrity as well as safety for those individuals handling and transporting the samples.

This SOP is designed to provide a high degree of certainty that environmental samples will arrive at their destination intact. This SOP assumes that samples will often require shipping overnight by a commercial carrier service, therefore, the procedures are more stringent than may be necessary if a laboratory courier is used or if samples are transported directly to their destination by a sampling team member. Should the latter occur, the procedures may be modified to reflect a lesser degree of packaging requirements.

Respective state or federal agency (regional offices) protocols may require or recommend specific types of equipment for use in sample packaging or a specific method of shipment that may vary from the indicated procedures. Deviations from this SOP to accommodate other regulatory requirements should be reviewed in advance of the field program, should be explained in the project work plan, and must be documented in the field project notebook when they occur.

1.2 General Principles

Sample packaging and shipment generally involves the placement of individual sample containers into a cooler or other similar shipping container and placement of packing materials and coolant in such a manner as to isolate the samples, maintain the required temperature, and to limit the potential for damage to sample containers when the cooler is transported.

1.3 Quality Assurance Planning Considerations

Sampling personnel should follow specific quality assurance guidelines as outlined in the site-specific work plan or Quality Assurance Project Plan (QAPP). Proper quality assurance requirements should be provided which will specify sample packaging and shipment requirements if variations to the indicated procedures are necessary on a particular project.

1.4 Health and Safety Considerations

Sampling personnel should be aware that packaging and shipment of samples involves potential physical hazards primarily associated with handling of occasional broken sample containers and lifting of heavy objects. Adequate health and safety measures must be taken to protect sampling personnel from these potential hazards. The project Health and Safety Plan (HASP) generally addresses physical and other potential hazards. This plan must be approved by the project Health and Safety Officer before work commences, must be distributed to all personnel performing sampling, and must be adhered to as field activities are performed. In the absence of a HASP, work will be conducted according to the ENSR Health and Safety Policy and Procedures Manual and/or direction from the Regional Health and Safety Manager.

2.0 RESPONSIBILITIES

2.1 Sampling Technician

It is the responsibility of the sampling technician to be familiar with the procedures outlined within this SOP and with specific sampling, quality assurance, and health and safety requirements outlined within the project-specific plans. The sampling technician is responsible for proper packaging and shipment of environmental samples and for proper documentation of sampling activities for the duration of the sampling program.

2.2 Sampling Coordinator

Large sampling programs may require additional support personnel such as a sampling coordinator. The sampling coordinator is responsible for providing management support such as maintaining an orderly sampling process, providing instructions to sampling technicians regarding sampling locations, and fulfilling sample documentation requirements, thereby allowing sampling technicians to collect samples in an efficient manner.

2.3 Project Manager

The project manager is responsible for ensuring that project-specific requirements are communicated to the project team and for providing the materials, resources, and guidance necessary to perform the activities in accordance with the project plan and this SOP. The project manager is also responsible for ensuring that proper arrangements have been made with the designated analytical laboratory. These arrangements include, but are not necessarily limited to, subcontractor agreements, analytical scheduling, and bottle/cooler orders. The project manager may delegate some of these responsibilities to other project staff.

3.0 REQUIRED MATERIALS

- Sample coolers
- Sample containers
- Shipping labels
- Chain-of-custody records, custody seals
- Bubble wrap
- Vermiculite (granular), or styrofoam pellets
- "Blue Ice" refreezable ice packs, or ice cubes
- Transparent tape, or rubber bands
- Fiber tape
- Duct tape
- Zipper-lock plastic bags

- Trash bags
- Health and Safety supplies
- Equipment decontamination materials
- Field project notebook/pen

4.0 METHOD

4.1 General Information

4.1.1 Regulatory Information

The extent and nature of sample containerization will be governed by the type of sample, and the most reasonable projection of the sample's hazardous nature and constituents. The EPA regulations (40 CFR Section 261.4(d)) specify that samples of solid waste, water, soil or air, collected for the sole purpose of testing, are exempt from regulation under the Resource Conservation and Recovery Act (RCRA) when any of the following conditions are applicable:

- Samples are being transported to a laboratory for analysis;
- Samples are being transported to the collector from the laboratory after analysis;
- Samples are being stored (1) by the collector prior to shipment for analyses, (2) by the analytical laboratory prior to analyses, (3) by the analytical laboratory after testing but prior to return of sample to the collector or pending the conclusion of a court case.

4.1.2 Sample Information:

The following information must accompany each shipment of samples on a chain-of-custody form (Figure 1) where each sample has an individual entry:

- Sample collector's name, mailing address and telephone number,
- Analytical laboratory's name, mailing address and telephone number,
- A unique identification of each sample,

- Sample description (matrix),
- Number and type of sample containers,
- Container size,
- Preservative,
- Type and method of analysis requested, and
- Date and time that the samples were collected and prepared for shipping,
- Special handling instructions, including notation of suspected high concentration samples.

4.1.3 Laboratory Notifications:

Prior to sample collection, the Project Manager, or designated alternative must notify the laboratory manager of the number, type and approximate collection and shipment dates for the samples. If the number, type or date of sample shipment changes due to program changes which may occur in the field, the Project Manager or alternate must notify the laboratory of the changes. Additional notification from the field is often necessary when shipments are scheduled for weekend delivery.

4.2 General Site Preparation

4.2.1 Small Projects

Small projects of one or two days duration may require packaging and shipment of samples using the field vehicle as the sample preparation area. If sample coolers will be sent via third party commercial carrier service, adequate sample packaging materials should be sent to the project location in advance of sampling or purchased from stores located near the site.

4.2.2 Large Projects

Multi-day or week sampling programs usually require rental of an office trailer or use of existing office/storage facilities for storage of equipment as well as for sample preparation. If possible, a designated area should be selected for storage of unused sample containers/coolers and another area for sample handling, packaging, and shipment. Handling of environmental samples should preferably be conducted in a clean area and away from unused

sample containers to minimize the potential for cross contamination. Large quantities of packaging materials may require advance special ordering. Shipping forms/labels may be preprinted to facilitate shipping.

4.2.3 Cooler Inspection and Decontamination

Laboratories will often re-use coolers. Every cooler received at a project location should be inspected for condition and cleanliness. Any coolers that have cracked interior or exterior linings/panels or hinges should be discarded as their insulating properties are now compromised. Any coolers missing one or both handles should also be discarded if replacement handles (i.e., knotted rope handles) can not be fashioned in the field. Replacement coolers may be purchased in the field if necessary.

The interior and exterior of each cooler should be inspected for cleanliness before using it. Excess strapping tape and old shipping labels should be removed. If the cooler interior exhibits visible contamination or odors it should be decontaminated in accordance with ENSR SOP-7600 (Decontamination of Equipment) prior to use. Drain plugs should be sealed on the inside with duct tape.

4.2.4 Other Considerations

VOC Samples - Sample containers used for VOC analysis may be grouped into a single cooler, with separate chain-of-custody record, to limit the number of trip blanks required for transportation and analysis. Individual VOC samples may also be placed into Zipper-lock bags to further protect the samples.

Contaminated Samples - Sample containers with presumed high contaminant concentrations should be isolated within their own cooler with each sample container placed into a Zipper-lock bag.

4.3 Sample Packaging Method

Sample packaging should be conducted in the following manner:

4.3.1 Place plastic bubble wrap matting over the base of each cooler or shipping container as needed. A 2- to 3-inch thickness layer of vermiculite may be used as a substitute base material.

4.3.2 Insert a clean trash bag into the cooler to serve as a liner.

4.3.3 Check that each sample container is sealed, labelled legibly, and is externally clean. Re-label and/or wipe bottles clean if necessary. Clear tape should be placed over the labels to protect them. Wrap each sample bottle individually with bubble wrap secured with tape or rubber bands. Place bottles into the cooler in an upright single layer with approximately one inch of space between each bottle. Do not stack bottles or place them in the cooler lying on their side. If plastic and glass sample containers are used, alternate the placement of each type of container within the cooler so that glass bottles are not placed side by side.

4.3.4 Insert cooler temperature blanks if required.

4.3.5 Place additional vermiculite, bubble wrap, and/or styrofoam pellet packing material throughout the voids between sample containers within each cooler to a level which meets the approximate top of the sample containers. Packing material may require tamping by hand to reduce the potential for settling.

4.3.6 Place cubed ice or cold packs in heavy duty Zip-lock type plastic bags, close the bags, and distribute the packages in a layer over the top of the samples. Cubed ice should be double-bagged to prevent leakage. Loose ice should never be used. Cold packs should be used only if the samples are chilled before being placed in the cooler.

4.3.7 Add additional bubble wrap/styrofoam pellets or other packing materials to fill the balance of the cooler or container.

4.3.8 Obtain two pieces of chain of custody tape as shown in Figure 2 and enter the custody tape numbers in the appropriate place on the chain-of-custody form. Sign and date the chain-of-custody tape.

4.3.9 Complete the chain-of-custody form. If shipping the samples involves use of a third party commercial carrier service, sign the chain-of-custody record thereby relinquishing custody of the samples. Shippers should not be asked to sign chain of custody records. If a laboratory courier is used, or if samples are transported to the laboratory, the receiving party should accept custody and sign the chain-of-custody records. Remove the last copy from the form and retain it with other field notes. Place the original (with remaining copies) in a Zipper-lock type plastic bag and tape the bag to the inside lid of the cooler or shipping container.

4.3.10 Close the top or lid of the cooler or shipping container.

4.3.11 Place the chain of custody tape at two different locations (i.e., one tape on each side) on the cooler or container lid and overlap with transparent packaging tape.

4.3.12 Packaging tape should be placed entirely around the sample shipment containers. A minimum of two full wraps of packaging tape will be placed at least two places on the cooler.

4.3.13 Repeat the above steps for each cooler or shipping container.

4.4 Sample Shipping Method

Packaged sample coolers should be shipped using one of the following options:

4.4.1 Hand Delivery

When a project member is transporting samples by automobile to the laboratory, the cooler should only be sealed with tape. In these cases, chain-of-custody will be maintained by the person transporting the sample and chain-of-custody tape need not be used. Chain-of-custody records should be relinquished upon delivery and a copy of the record retained in the project file.

4.4.2 Laboratory Courier

Laboratory couriers are usually employees of the analytical laboratory receiving the samples. As such, they will accept custody of the samples and must be asked to sign the chain-of-custody records. Chain-of-custody records do not need to be sealed in the cooler although it is recommended that the coolers be sealed with tape. All other packaging requirements generally apply unless otherwise specified in the QAPP.

If the laboratory courier is not authorized to accept custody of the samples, or if the requirements of the project plan preclude transfer to the laboratory courier, samples will be handled as described below in Section 4.4.3.

4.4.3 Third Party Courier

If overnight shipment is required, a third party package delivery service should be used. Transport the cooler to the package delivery service office

or arrange for package pick-up at the site. Fill out the appropriate shipping form or airbill and affix it to the cooler. Some courier services may use multi-package shipping forms where only one form needs to be filled out for all packages going to the same destination. If not, a separate shipping form should be used for each cooler. Keep the receipt for package tracking purposes should a package become lost. Please note that each cooler also requires a shipping label which indicates point of origin and destination. This will aid in recovery of a lost cooler if a shipping form gets misplaced. Never leave coolers unattended while waiting for package pick-up. Airbills or waybills will be maintained as part of the custody documentation.

4.5 Sample Receipt

Upon receipt of the samples, the analytical laboratory will open the cooler or shipping container and will sign "received by laboratory" on each chain-of-custody form. The laboratory will verify that the chain-of-custody tape has not been broken previously and that the tape number corresponds with the number on the chain-of-custody record. The laboratory will note the condition of the samples upon receipt and will identify any discrepancies between the contents of the cooler and chain-of-custody. The analytical laboratory will then forward the back copy of the chain-of-custody record to the project manager to indicate that sample transmittal is complete.

5.0 QUALITY CONTROL

The potential for samples to break during transport increases greatly if individual containers are not snugly packed into the cooler. Completed coolers may be lightly shake-tested to check for any loose bottles. The cooler should be repacked if loose bottles are detected.

Environmental samples are generally shipped so that the samples are maintained at a temperature of approximately 4°C. Temperature blanks may be required for some projects as a quality assurance check on shipping temperature conditions. These blanks usually are supplied by the laboratory and consist of a 40-ml vial or plastic bottle filled with tap water. Temperature blanks should be placed near the center of the cooler.

6.0 DOCUMENTATION

Documentation supporting sample packaging and shipment generally consists of chain-of-custody records and shipping records. In addition, a description of sample packaging procedures will be written in the field project notebook. All documentation will be retained in the project files following project completion.

7.0 TRAINING/QUALIFICATIONS

Sample packaging and shipment is a relatively simple procedure requiring minimal training and a minimal amount of equipment. It is, however, recommended that initial attempts be supervised by more experienced personnel. Sampling technicians should be health and safety certified as specified by OSHA (29 CFR 1910.120(e)(3)(i)) to work on sites where hazardous waste materials are considered to be present.

Figure 1. Chain of Custody Form

0

CHAIN OF CUSTODY RECORD

3DJH _____ RI _____

&OLHQW3URMHFW1DPH	3URMHFW/RFDWLRQ	$QDO\VLV5HTXHVWHG
3URMHFW1XPEHU	)LHOG/RJERRN1R	
6DPSOHU3ULQW1DPH$IILOLDWLRQ	&KDLQRI&XVWRG\7DSH1R	
6LJQDWXUH	6HQG5HVXOWV5HSRUWWR	

)LHOG6DPSOH1R ,GHQWLILFDWLRQ	'DWH	7LPH	*UDE	&RPS	6DPSOH&RQWDLQHU 6L]H0DWO	6DPSOH7\SH /LTXLG6OXGJH(WF	3UHVHUYDWLYH	)LHOG)LOWHUHG							/DE,'	5HPDUNV

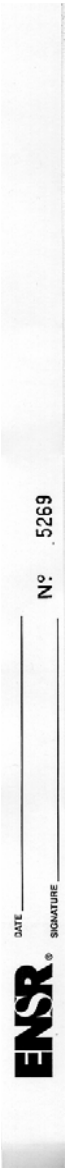
5HOLQTXLVKHGE\3ULQW1DPH	'DWH	5HFHLYHGE\3ULQW1DPH	'DWH
6LJQDWXUH	7LPH	6LJQDWXUH	7LPH
5HOLQTXLVKHGE\3ULQW1DPH	'DWH	5HFHLYHGE\3ULQW1DPH	'DWH
6LJQDWXUH	7LPH	6LJQDWXUH	7LPH
5HOLQTXLVKHGE\3ULQW1DPH	'DWH	5HFHLYHGE\3ULQW1DPH	'DWH
6LJQDWXUH	7LPH	6LJQDWXUH	7LPH

$QDO\WLFDO/DERUDWRU\'HVWLQDWLRQ

(165
:/D3RUWH$YH
)RUW&ROOLQV&2

6HULDO1R

Figure 2. Chain of Custody Tape



SOP NUMBER: 7600

Decontamination of Field Equipment

Date:	4th Qtr. 1994
Revision Number:	4
Author:	Charles Martin
Discipline:	Geosciences

1.0 PURPOSE AND APPLICABILITY

1.1 Purpose and Applicability

This SOP describes the methods to be used for the decontamination of field equipment used in the collection of environmental samples. The list of field equipment may include a variety of items used in the collection of soil and/or water samples, such as split-spoon samplers, trowels, scoops, spoons, bailers and pumps. Heavy equipment such as drill rigs and backhoes also require decontamination, usually in a specially constructed temporary decontamination area.

Decontamination is performed as a quality assurance measure and a safety precaution. Improperly decontaminated sampling equipment can lead to misinterpretation of environmental data due to interference caused by cross-contamination. Decontamination protects field personnel from potential exposure to hazardous materials. Decontamination also protects the community by preventing transportation of contaminants from a site.

This SOP emphasizes decontamination procedures to be used for decontamination of reusable field equipment. Occasionally, dedicated field equipment such as well construction materials (well screen and riser pipe) or disposable field equipment (bailers or other general sampling implements) may also require decontamination prior to use. The project-specific work plan should indicate the specific decontamination requirements for a particular project.

Respective state or federal agency (regional offices) regulations may require specific types of equipment or procedures for use in decontamination of field equipment. The project manager should review the applicable regulatory requirements, if any, prior to the start of the field investigation program.

1.2 General Principles

Decontamination is accomplished by manually scrubbing, washing, or spraying equipment with detergent solutions, tap water, distilled/deionized water, steam and/or high pressure water, or solvents. The decontamination method and agents

are generally determined on a project-specific basis and must be stated in the Quality Assurance Project Plan (QAPP).

Generally, decontamination of equipment is accomplished at each sampling site between collection points. Waste decontamination materials such as spent liquids and solids will be collected and managed as investigation-derived waste for later disposal. All decontamination materials, including wastes, should be stored in a central location so as to maintain control over the quantity of materials used or produced throughout the investigation program.

1.3 Quality Assurance Planning Considerations

1.3.1 General Considerations

Sampling personnel should follow specific quality assurance guidelines as outlined in the site-specific QAPP. The QAPP guidelines typically require collection of equipment blank samples in order to determine the effectiveness of the decontamination procedure.

The decontamination method, solvent, frequency, location on site and the method of containment and disposal of decontamination wash solids and solutions are dependent on site logistics, site-specific chemistry, and nature of the contaminated media to be studied and the objectives of the study. Each topic must be considered and addressed during development of a decontamination strategy and should be outlined in the Quality Assurance Project Plan (QAPP).

1.3.2 Solvent Selection

There are several factors which need to be considered when deciding upon a decontamination solvent. The solvent should not be an analyte of interest. The sampling equipment must be resistant to the solvent. The solvent must be evaporative or water soluble or preferably both. The applicable regulatory agency may have specific requirements regarding decontamination solvents. The QAPP should specify the type of solvent to be used for a particular project.

The analytical objectives of the study must also be considered when deciding upon a decontamination solvent. Pesticide-grade methanol is the solvent of choice for general organic analyses. It is relatively safe and effective. Hexane, acetone, and isopropanol are sometimes used as well. A 10% nitric acid in deionized water solution is the solvent of choice for general metals

analyses. Nitric acid can be used only on Teflon, plastics and glass. If used on metal equipment, nitric acid will eventually corrode the metal and lead to the introduction of metals to the collected samples. Dilute hydrochloric acid is usually preferred over nitric acid when cleaning metal sampling equipment.

Equipment decontamination should be performed a safe distance away from the sampling area so as not to interfere with sampling activities but close enough to the sampling area to maintain an efficient working environment. If heavy equipment such as drill rigs or backhoes are to be decontaminated, then a central decontamination station should be constructed with access to a power source and water supply.

1.4 Health and Safety Considerations

Decontamination procedures may involve chemical exposure hazards associated with the type of contaminants encountered or solvents employed and may involve physical hazards associated with decontamination equipment. When decontamination is performed on equipment which has been in contact with hazardous materials or when the quality assurance objectives of the project require decontamination with chemical solvents, the measures necessary to protect personnel must be addressed in the project Health and Safety Plan (HASP). This plan must be approved by the project Health and Safety Officer before work commences, must be distributed to all personnel performing equipment decontamination, and must be adhered to as field activities are performed.

2.0 RESPONSIBILITIES

2.1 Sampling Technician

It is the responsibility of the sampling technician to be familiar with the decontamination procedures outlined within this SOP and with specific quality assurance, and health and safety requirements outlined within project-specific work plans (HASP, QAPP). The sampling technician is responsible for decontamination of field equipment and for proper documentation of decontamination activities. The sampling technician is also responsible for ensuring that decontamination procedures are followed by subcontractors when heavy equipment requires decontamination.

2.2 Field Project Manager

The field project manager is responsible for ensuring that the required decontamination procedures are followed at all times. The project manager is also responsible for ensuring that subcontractors construct and operate their decontamination facilities according to project specifications. The project manager is responsible for collection and control of IDW in accordance with project specifications.

3.0 REQUIRED MATERIALS

- Decontamination agents (per work plan requirements):
  - LIQUI-NOX, ALCONOX, or other phosphate-free biodegradable detergent,
  - Tap water,
  - Distilled/deionized water,
  - Nitric acid and/or hydrochloric acid,
  - Methanol and/or hexane, acetone, isopropanol.
- Health and Safety equipment
- Chemical-free paper towels
- Waste storage containers: drums, 5-gallon pails w/covers, plastic bags
- Cleaning containers: plastic buckets or tubs, galvanized steel pans, pump cleaning cylinder
- Cleaning brushes
- Pressure sprayers
- Squeeze bottles
- Plastic sheeting
- Aluminum foil
- Field project notebook/pen

4.0 METHODS

4.1 General Preparation

4.1.1 It should be assumed that all sampling equipment, even new items, are contaminated until the proper decontamination procedures have been performed on them or unless a certificate of analysis is available which demonstrates the items cleanliness.

Field equipment that is not frequently used should be wrapped in aluminum foil, shiny side out, and stored in a designated "clean" area. Small field equipment can also be stored in plastic bags to eliminate the potential for contamination. Field equipment should be inspected and decontaminated prior to use if the equipment appears contaminated and/or has been stored for long periods of time. Unless customized procedures are stated in the QAPP for decontamination of equipment, the standard procedures specified in this SOP shall be followed.

4.1.2 Establish the decontamination station within an area that is convenient to the sampling location. If single samples will be collected from multiple locations, then a centralized decontamination station, or a portable decontamination station should be established.

4.1.3 An investigation-derived waste (IDW) containment station should be established at this time also. The project-specific work plan should specify the requirements for IDW containment. In general, decontamination solutions are discarded as IDW between sampling locations. Solid waste is disposed of as it is generated.

4.2 Decontamination for Organic Analyses

4.2.1 This procedure applies to soil sampling and groundwater sampling equipment used in the collection of environmental samples submitted for organic constituents analysis. Examples of relevant items of equipment include split-spoons, trowels, scoops/spoons, bailers, and other small items. Submersible pump decontamination procedures are outlined in Section 4.4.

4.2.2 Decontamination is to be performed before sampling events and between sampling points.

4.2.3 After a sample has been collected, remove all gross contamination from the equipment or material by brushing and then rinsing with available tap water.

This initial step may be completed using a 5-gallon pail filled with tap water. Steam or a high-pressure water rinse may also be conducted to remove solids and/or other contamination.

4.2.4 Wash the equipment with a phosphate-free detergent and tap water solution. This solution should be kept in a 5-gallon pail with its own brush.

4.2.5 Rinse with tap water or distilled/deionized water until all detergent and other residue is washed away. This step can be performed over an empty bucket using a squeeze bottle or pressure sprayer.

4.2.6 Rinse with methanol or other appropriate solvent using a squeeze bottle or pressure sprayer. Rinsate should be collected in a waste bucket.

4.2.7 Rerinse with deionized water to remove any residual solvent. Rinsate should be collected in the solvent waste bucket.

4.2.8 Allow the equipment to air-dry in a clean area or blot with chemical-free paper towels before reuse. Wrap the equipment in tin foil and/or seal it in a plastic bag if it will not be reused for a while.

4.2.9 Dispose of soiled materials and spent solutions in the designated IDW disposal containers.

4.3 Decontamination for Inorganic (Metals) Analyses

4.3.1 This procedure applies to soil sampling equipment used primarily in the collection of environmental samples submitted for inorganic constituents analysis. Examples of relevant items of equipment include split-spoons, trowels, scoops/spoons, bailers, and other small items.

4.3.2 For plastic and glass sampling equipment, follow the steps outlined in 4.2 above, however, use a 10% nitric acid solution (acid in water) in place of the solvent rinse in Section 4.2.6.

4.3.3 For metal sampling equipment, follow the steps outlined in 4.2 above, however, use a 10% hydrochloric acid solution (acid in water) in place of the solvent rinse in Section 4.2.6.

4.4 Decontamination of Submersible Pumps

4.4.1 This procedure will be used to decontaminate submersible pumps before and between ground-water sample collection points. This procedure applies to both electric submersible and bladder pumps. This procedure also applies to discharge tubing if it will be reused between sampling points.

4.4.2 Prepare the decontamination area if pump decontamination will be conducted next to the sampling point. If decontamination will occur at another location, the pump and tubing may be removed from the well and placed into a clean trash bag for transport to the decontamination area. Pump decontamination is easier with the use of 3-foot tall pump cleaning cylinders (i.e., Nalgene cylinder) for the various cleaning solutions, although the standard bucket rinse equipment may be used.

4.4.3 Once the decontamination station is established, the pump should be removed from the well and the discharge tubing and power cord coiled by hand as the equipment is removed. If any of the equipment needs to be put down temporarily, place it on a plastic sheet (around well) or in a clean trash bag. If a disposable discharge line is used it should be removed and discarded at this time.

4.4.4 As a first step in the decontamination procedure, use a pressure sprayer with tap water to rinse the exterior of the pump, discharge line, and power cord as necessary. Collect the rinsate and handle as IDW.

4.4.5 Place the pump into a pump cleaning cylinder or bucket containing a detergent solution (detergent in tap water). Holding the tubing/power cord, pump solution through the pump system. A minimum of one gallon of detergent solution should be pumped through the system. Collect the rinsate and handle as IDW.

4.4.6 Place the pump into another cylinder/bucket containing a 10% solution of solvent (methanol, or other designated solvent) in distilled/deionized water. Pump until the detergent solution is removed. Collect the rinsate and handle as IDW.

4.4.7 Place the pump into another cylinder/bucket containing distilled/deionized water. Pump a minimum of 3 to 5 pump system volumes (pump and tubing) of water through the system. Collect the rinsate and handle as IDW.

4.4.8 Remove the pump from the cylinder/bucket and if the pump is reversible, place the pump in the reverse mode to discharge all removable water from the system. If the pump is not reversible the pump and discharge line should be drained by hand as much as possible. Collect the rinsate and handle as IDW.

4.4.9 Using a pressure sprayer with distilled/deionized water, rinse the exterior of the pump, discharge line, and power cord thoroughly, shake all excess water, then place the pump system into a clean trash bag for storage. If the pump system will not be used again right away, the pump itself should also be wrapped with aluminum foil before placing it into the bag.

4.5 Decontamination of Large Equipment

4.5.1 Consult the QAPP for instruction on the location of the decontamination station and the method of containment of the wash solutions. On large projects usually a temporary decontamination facility (decontamination pad) is required which may include a membrane-lined and bermed area large enough to drive heavy equipment (drill rig, backhoe) onto with enough space to spread other equipment and to contain overspray. Usually a small sump with pump is necessary to collect and contain rinsate. A water supply and power source is also necessary to run steam cleaning and/or pressure washing equipment.

4.5.2 Upon arrival and prior to leaving a sampling site, all heavy equipment such as drill rigs, trucks, and backhoes should be thoroughly cleaned and then the parts of the equipment which come in contact or in close proximity to sampling activity should be decontaminated. This can be accomplished in two ways, steam cleaning or high pressure water wash and manual scrubbing. Following this initial cleaning, only those parts of the equipment which come in close proximity to the sampling activities (i.e., auger stems, rods, backhoe bucket) must be decontaminated in between sampling events.

Occasionally, well construction materials such as well screen and riser pipe may require decontamination before the well materials are used. These materials may be washed in the decontamination pad, preferably on a raised surface above the pad (i.e., on sawhorses), with clean plastic draped over the work surfaces. Well materials usually do not require a multistep cleaning process as they generally arrive clean from the manufacturer. Usually, a thorough steam-cleaning of the interior/exterior of the well materials will be sufficient. The QAPP should provide specific guidance regarding decontamination of well materials.

5.0 QUALITY CONTROL

5.1 Field Blank Sample Collection

General guidelines for quality control check of field equipment decontamination usually require the collection of one field blank from the decontaminated equipment per day. The QAPP should specify the type and frequency of collection of each type of quality assurance sample.

Field blanks are generally made by pouring laboratory-supplied deionized water into, over, or through the freshly decontaminated sampling equipment and then transferring this water into a sample container. Field blanks should then be labeled as a sample and submitted to the laboratory to be analyzed for the same parameters as the associated sample. Field blank sample numbers, as well as collection method, time and location should be recorded in the field notebook.

6.0 DOCUMENTATION

Specific information regarding decontamination procedures should be documented in the project-specific field notebook. Documentation within the notebook should thoroughly describe the construction of each decontamination facility and the decontamination steps implemented in order to show compliance with the project work plan. Decontamination events should be logged when they occur with the following information documented:

- Date, time and location of each decontamination event
- Equipment decontaminated
- Method
- Solvents
- Noteable circumstances
- Identification of field blanks and decontamination rinsates
- Method of blank and rinsate collection
- Date, time and location of blank and rinsate collection
- Disposition of IDW

Repetitive decontamination of small items of equipment does not need to be logged each time the item is cleaned.

7.0 TRAINING/QUALIFICATIONS

All sampling technicians performing decontamination must be properly trained in the decontamination procedures employed, the project data quality objectives, health and safety

procedures and the project QA procedures. Specific training or orientation will be provided for each project to ensure that personnel understand the special circumstances and requirements of that project. Field personnel should be health and safety certified as specified by OSHA (29 CFR 1910.120(e)(3)(i)) to work on sites where hazardous materials may be present.

8.0 REFERENCES

Not applicable.

ENSR | AECOM

Standard Operating Procedure

Water Level Measurements

Procedure Number: 7721

Revision No.: 0

Revision Date: February 2008

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Annual review of this SOP has been performed and the SOP still reflects current practice.

Initials: _____ Date: _____
Initials: _____ Date: _____

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1.0 Scope and applicability

1.1 This Standard Operating Procedure (SOP) defines the methods to be used for measuring the depth to groundwater and total depth of groundwater monitoring wells and piezometers. Similar procedures can also be used to measure the depth to water in other structures such as catch basins or cisterns or in surface water bodies from fixed structures such as bridges, culverts, or piers.

Water level and well depth measurements collected from monitoring wells or piezometers may be used for the following purposes, among others:

- To evaluate the well condition (potential silt accumulation, height of water column, etc.);
- To establish sampling requirements, such as purge volumes and drawdown during purging;
- To calculate the horizontal hydraulic gradient and the direction of groundwater flow;
- To calculate the vertical hydraulic gradient, if well nests are used (i.e., the direction of groundwater flow in the vertical plane);
- To evaluate the effects of manmade and natural stresses on the groundwater system; and
- To calculate other important hydrogeologic characteristics (e.g., measuring drawdown during slug tests or aquifer pumping tests).

This information, when combined with other location-specific information, is important in understanding the current distribution of constituents in groundwater and their potential for migration in the future. Hydrogeologic characterization is important not only in evaluating potentially contaminated groundwater but also in evaluating non-contaminated groundwater resources.

1.2 Some wells may contain a light non-aqueous phase liquid (LNAPL) floating on the water surface. The procedures outlined in this SOP may be used to measure water levels in such wells, but the results may not be representative of the hydraulic head/potentiometric level. For measurement of LNAPL in wells, refer to ENSR SOP No. 7722.

1.3 There are other methods for measuring water depths than those described in this SOP, for example, a weighted tape with or without a sounding device ("plopper"), pressure transducers, air line pressure, strip recorders, etc. This SOP addresses the methods in most common and regular use.

1.4 This SOP is to be utilized to conduct the work identified in the title of this SOP. In the event the Project Manager of Project Team determines that the protocols and procedures listed in this SOP are not applicable to the project, there is the option to either adapt this SOP or to develop a site-specific SOP to more closely match the requirements of the project. Refer to SOP 1011, Preparation and Control of Standard Operating Procedures, for SOP modification and Project Operating Procedure (POP) development procedures.

2.0 Health and safety considerations

2.1 The health and safety considerations for the work associated with this SOP, including both potential physical and chemical hazards, will be addressed in the site specific Health and Safety Plan (HASP). In the absence of a site-specific HASP, work will be conducted according to the ENSR Health and Safety Policy and Procedures Manual and/or direction from the Regional Health and Safety Manager.

3.0 Interferences

3.1 Potential interferences could result in inaccurate readings if the sensor on the water level meter is wet or dirty, or if the cable cannot be kept vertically upright (for example, from a well that is not plumb or from a bridge in windy conditions). Care shall be taken to keep the probe clean, and to take appropriate measures to reduce these interferences when measuring water levels. The probe may also be shaken to remove water or other fluids that may adhere to the probe. If there is any concern that a particular reading may not be accurate, this shall be noted in the field log book.

3.2 If LNAPL is present in a well, the measured depth to water may not be representative of the hydraulic head/potentiometric level. If the LNAPL thickness and specific gravity are known, an accurate hydraulic head can be calculated (see ENSR SOP No. 7722).

3.3 Some water level meters (especially oil/water interface probes) may rely on optical technology for readings. In these cases, the readings may be influenced by the presence of light. While this is not an issue in wells, it may be at surface water bodies.

3.4 The measured depth to water is not always representative of the hydraulic head in the aquifer. Interferences may include barometric pressure effects, timing during tidal cycles, well construction details, confined/artesian aquifers, well efficiency, etc. Where such influences may be important, the project-specific work plan should specify any corrective measures or additional data to be collected. Interpretation and use of water level data should be performed by a trained specialist.

4.0 Equipment and materials

4.1 Electronic Water Level Meter - Electronic water level meters consist of a spool of small-diameter cable (or tape) with a weighted probe attached to the end. The cable (or tape) is marked with measurement increments in feet (ft) or meters (m) (accurate to 0.01 ft/0.01m), with the zero point being the sensor of the probe. When the probe comes in contact with the water, an electrical circuit is closed, and a light and/or buzzer within the spool will signal the contact. The cable must be of sufficient length the reach to the expected depth of the water to be measured. The probe shall be tested (using water containing dissolved ions) at the start of the field program to ensure proper operation.

An oil/water interface probe may be used to measure water depths. However, in some cases, there may be increased risk of cross-contamination using a probe that is regularly placed in separate-phase liquids. Where such risks are considered significant, project-specific requirements will specify that oil-water interface probes are not to be used in wells where no separate-phase liquids are expected.

4.2 Other Materials - Other materials that may be required:

- Health and safety supplies (as required by the HASP)
- Equipment decontamination materials, including absorbent pads if appropriate
- Plastic sheeting or bucket for resting instrument off the ground
- Water level field form (if applicable)
- Well construction records
- Approved plans (e.g., Field Sampling Plan, Quality Assurance Plan, HASP)
- Field project logbook/waterproof pen
- Appropriate hand tools and keys to access monitoring wells

5.0 Procedures

5.1 Summary of method

Measurements will involve measuring the depth to water and/or total well depth to the nearest 0.01 ft/0.01m using an electronic water level meter. The depths within wells will be measured from the top of casing (typically the inner casing) at the surveyed elevation point. This reference point should be marked so that readings are consistently taken from the same reference point. Depths to surface water may be similarly measured from a marked reference point on the fixed structure (e.g., bridge, culvert, pier, wharf) passing over or bordering the surface water body.

5.2 General preparation

5.2.1 Well records review: Well completion diagrams should be reviewed to determine well construction characteristics, including the location of the reference point and the total depth of the well. Historic static water level measurements and survey information may also be reviewed.

5.2.2 Well access: Many wells may be locked for security reasons. The necessary procedures and equipment to access the wellhead shall be identified prior to entering the site.

5.2.3 Equipment: There are many different water level meters available. Field personnel should make sure the appropriate equipment is used based on well construction details (e.g., well diameter, anticipated depth to water). The specific equipment to be used should be inspected. Field personnel should be sure the equipment is in proper working order, and the measurement increment marks are legible. The type of power supply (e.g., type of batteries) should be determined so that an appropriate back-up supply can be obtained if needed. Sometimes water level meters may be repaired by removing a length of cable near the sensor and re-splicing the cable to the sensor. If this kind of repair has taken place, the measurement markings on the cable are no longer accurate. This condition should be observed and noted, and if appropriate, a replacement water level meter may be obtained as an alternative to correcting the water level measurement for the length of the splice.

5.2.4 Calibration: Manufacturer's instructions, if any, for calibrating or maintaining the accuracy of the instrument shall be followed. If there are project-specific requirements for calibration, these shall also be implemented as outlined in project-specific plans.

5.2.5 Equipment decontamination: All down-hole equipment should be decontaminated prior to and after use and between well locations in accordance with project-specific requirements. Note that some water level probes may be made of materials that are incompatible with certain decontamination solvents.

5.2.6 Order of measurement: For some projects, there may be a specific order in which measurements are to be collected, for example, from the least to most contaminated wells. Any such requirements will be specified in the project-specific plans.

5.2.7 Opening the well: Prior to accessing the well, the wellhead should be cleared of debris and/or standing water. For example, it is common to find standing water in flush mount wellheads that, if not removed, will enter the monitoring well, potentially causing inaccurate water level measurements and/or contamination of the groundwater. Nothing from the ground surface should be allowed to enter the well. Once the wellhead is clear, open the well to obtain the measurements. In some cases, it may be necessary to allow the water level to equilibrate prior to measurement (e.g., wells with fully submerged screened intervals).

5.3 Measurement procedures

5.3.1 At each location (well, piezometer, bridge/culvert, pier/wharf, etc.), determine the location of the surveyed elevation mark. For wells, general markings may include either a notch in the riser pipe or a permanent ink mark on the riser pipe. Some projects may specify a consistent reference point for all wells, for example, the highest point on the riser or the northernmost point. For monitoring surface water levels, there may be a painted mark on an existing structure or the reference point must be known if not marked.

5.3.2 If the reference point is not marked, a point may be selected and clearly and permanently marked to be used for future measurements. If this is done, the project manager must be notified to arrange for the elevation of the new reference point to be surveyed.

5.3.3 To obtain a water level measurement, lower the probe of the water level meter down into the water in the well until the audible sound of the unit is detected or the light on an electronic sounder illuminates. In wells, the probe shall be lowered slowly into the well to avoid disruption of formation water and creation of turbulent water within the well. At this time, the precise measurement should be determined (to the nearest 0.01 ft/0.01m) by repeatedly raising and lowering the tape to converge on the exact measurement. Obtain the reading from the stadia-marked cable where it crosses the surveyed reference point. If the cable is not marked to the nearest 0.01ft/0.01m, a manual rule may be used to interpolate between marked measurements.

5.3.4 Record the water level measurement as well as the location identification number, measuring point (surveyed elevation point), date, time, and weather conditions in the field logbook and/or field form. Any problems with the condition of the well should be noted so that appropriate maintenance can be performed.

5.3.5 To measure the total depth of a well, lower the probe (turn down signal as appropriate) slowly to the bottom of the well. For deep wells or wells with a soft or silty base, the depth may be difficult to determine. It may be helpful to lower the probe until there is slack in the tape, and

gently pull up until it feels as if there is a weight at the end of the tape. Obtain the depth reading (to the nearest 0.01 ft/0.01m) from the cable where it crosses the surveyed reference point. If the cable is not marked to the nearest 0.01ft/0.01m, a manual rule may be used to interpolate between marked measurements.

5.3.6 Record the total well depth in the field logbook and/or field form.

5.3.7 The meter will be decontaminated in accordance with appropriate project-specific requirements and equipment use and care requirements. If the probe was in contact with separate-phase liquids, the potential for cross-contamination is greater, so appropriate care should be taken during decontamination, as specified in project-specific requirements. It is important to avoid placing the measuring tape and probe directly on the ground surface (to minimize potential cross-contamination) or allowing the cable to become kinked (which affects the accuracy of the measured depths).

5.4 Special conditions

5.4.1 Wells containing pumps or other equipment. It may be difficult to obtain accurate water level depths in wells where down-hole equipment is present. There may not be sufficient space within the well for the water level meter, or the meter cable may become bound up in the tubing, cables, or other equipment in the well. It is preferable to remove down-hole equipment when feasible. If removal of the equipment is not feasible and there is a reasonable chance of getting the meter caught in the well and not being able to remove it, it may be preferable to avoid collecting water level data.

5.4.2 Drinking water wells. The water level meter represents a potential source of surface contamination when introduced into drinking water wells, particularly for bacteriological contamination. If it is necessary to measure water level depths in drinking water wells using the procedures in this SOP, appropriate disinfection procedures should be performed.

6.0 Quality assurance / quality control

6.1 Field personnel will follow site-specific quality assurance guidelines. Where measured depths are not consistent with well records or previously measurements, the depths should be re-measured, verified, and documented in the field records.

6.2 Field duplicates of the depth-to-water measurements will be obtained if required by and at the frequency specified in project-specific requirements. To collect a field duplicate measurement, the water level probe will be fully withdrawn from the well, then re-lowered to obtain a second reading of the depth to water. No more than a few minutes should elapse between the two measurements. Field duplicates will not be obtained if water levels are changing rapidly, for example, during pumping tests.

6.3 Manufacturer's instructions, if any, for calibrating or maintaining the accuracy of the instrument shall be followed.

7.0 Data and records management

7.1 All field information will be recorded in the field logbook or on a field collection form by field personnel. Recording of field data will follow the guidance presented in ENSR SOP 7515, Recording of Field Data.

7.2 Unanticipated changes to the procedures or materials described in this SOP (deviations) will be appropriately documented in the project records.

7.3 Records associated with the activities described in this SOP will be maintained according to the document management policy for the project.

8.0 Personnel qualifications and training

8.1 Qualifications and training

8.1.1 The individual executing these procedures must have read, and be familiar with, the requirements of this SOP.

8.1.2 Collecting water level measurements is a relatively simple procedure requiring minimal training and a relatively small amount of equipment. It is recommended that the collection of water level measurements be initially supervised by more experienced personnel.

8.1.3 Field personnel must be health and safety trained as required by the project conditions and local/national standards.

8.2 Responsibilities

8.2.1 The project manager is responsible for providing the project team with the materials, resources and guidance necessary to properly execute the procedures described in this SOP.

8.2.2 The individual performing the work is responsible for implementing the procedures as described in this SOP and any project-specific work plans.

8.2.3 Field personnel are responsible for the proper use, maintenance, and decontamination of all equipment used for obtaining water level measurements, as well as proper documentation in the field logbook or field forms (as appropriate).

9.0 References

American Society for Testing Materials. 1993. ASTM Standard D4750, Test Method for Determining Subsurface Liquid Levels in a Borehole or Monitoring Well (Observation Well).

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ENSR SOP 1011 – Preparation and Control of Standard Operating Procedures.

ENSR SOP 7515 – Recording of Field Data.

ENSR SOP 7722 – Measurement of Non-Aqueous Phase Liquids (NAPL) in Wells.

United States Environmental Protection Agency. 2001. Guidance for Preparing Standard Operating Procedures (SOPs). EPA QA/G-6. EPA/240/B-01/004. USEPA Office of Environmental Information, Washington, DC. March 2001.

10.0 Revision history

Revision	Date	Changes
0	February 2008	NA

Appendix C

Laboratory Standard Operating Procedures

Title: Extended List Polynuclear Aromatic Hydrocarbons by GCMS Selective Ion Monitoring for CSLP with an Attachment for Analysis by GCMS Full Scan [Method No 8270C]

Approvals (Signature/Date):

William Rhoades 2/12/08 William Rhoades — Date Technical Manager	Adam W. Alban 13 Feb 08 Adam Alban — Date Health & Safety Manager / Coordinator
Karen Kuoppala 2-12-08 Karen Kuoppala — Date Quality Assurance Manager	Robert C. Hanisch 2/15/08 Robert C. Hanisch — Date Laboratory Director

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1.0 Scope and Application

This procedure is a Gas Chromatography/Mass Spectrometry (GC/MS) technique for the determination of polynuclear aromatic hydrocarbons (PAHs) at the part per trillion (ppt or ng/L) level. The list of analytes is shown in Table II. This method should be used for the analysis of water samples previously characterized by a method such as 8270C and containing organic components at less than 10,000 ng/L. Samples containing greater than 10,000 ng/L semivolatile organics should be analyzed by the Full Scan method designed to detect PAHs at part-per- billion levels. Please see Appendix I for details on the Full Scan method. The extraction procedures are defined in Appendix II.

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 Refer to Table 1 for a list of compounds and quantitation ions. Reporting limits range from 2 to 10 ppt for the PAH compounds extracted using 4 liters of water and from 10 to 50 ppb for the samples utilizing a 1 liter extraction volume.

2.0 Summary of Method

The PAH compounds are extracted from 4 liters of water using methylene chloride in a continuous liquid-liquid extractor (CLLE). A 1-liter extraction procedure for PAHs is also used. Quantitation of the isolated target analytes is performed by gas chromatography - mass spectrometry (GC/MS) in the selected ion monitoring mode (SIM). The ions used for analysis of each compound are shown in Table I.

3.0 Definitions

3.1 Refer to the Laboratory Quality Assurance Manual (QAM) for definitions of QA/QC terms used in this document.

3.2 Selected Ion Monitoring (SIM) - A mass spectrometry technique that provides lower detection level capability by monitoring fewer mass scans for longer periods of time than is done in full scan methods.

3.3 Primary Ion Area - The signal chosen for quantitation purposes.

3.4 Secondary Ion Area - The signal chosen for identification and confirmation purposes.

4.0 Interferences

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the ion current profiles. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks. The use of high-purity reagents and solvents helps to minimize interference problems.

4.2 Matrix interference may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the environment being sampled.

4.3 An interference that is unique to selected ion monitoring techniques can arise from the presence of an interfering compound, which contains the quantitation mass ion. This event results in a positive interference to the reported value for the compound of interest. An example of this situation is a commonly detected peak that nearly co-elutes with the target analyte Indole. The unknown interfering peak elutes just after Indole, but contains the quantitation ion. Interferences such as this one are controlled to some degree by acquiring data for a confirmation ion. If the ion ratios between the quantitation ion and the confirmation ion are not within the specified limits, then interferences may be present.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, and this document.

5.1 Specific Safety Concerns or Requirements

5.1.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

NOTE: Latex and vinyl gloves provide no protection against the organic solvents used in this method. Nitrile or similar gloves must be used.

5.1.2 The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and should take appropriate safety precautions.

5.1.3 The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

5.1.4 There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating.

NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.

A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Methanol	Flammable Poison	200 ppm (TWA) 260 mg/m^3 (TWA)	May be fatal or cause blindness if swallowed. Inhalation, ingestion, or skin absorption can cause significant disturbances in vision, including blindness. Prolonged and/or repeated contact may cause defatting of the skin and dermatitis. Inhalation of methanol vapor initially causes central nervous system depression with nausea, headache, vomiting, dizziness, and incoordination. Prolonged exposure leads to metabolic acidosis and sever visual effects, up to and including blindness and/or nervous system effect.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.

(1) Exposure limit refers to the OSHA regulatory exposure limit.

6.0 Equipment and Supplies

6.1 Gas Chromatograph

6.1.1 Gas chromatograph/mass spectrometer system: an analytical system complete with a temperature-programmable gas chromatograph suitable for split/splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to the source.

6.2 Mass Spectrometer

6.2.1 A mass spectrometer operating at 70 eV (nominal) electron energy in the electron impact ionization mode and tuned to maximize the sensitivity of the instrument to the compounds being analyzed. The GC capillary column is fed directly into the ion source of the mass spectrometer.

6.2.2 Autosampler: LEAP Technologies CTC A200S, HP7683 Autosampler or equivalent.

6.2.3 GC/MS Interface: Any GC-to-MS interface that gives acceptable calibration points and achieves acceptable tuning performance criteria may be used.

6.2.4 A computer system interfaced to the mass spectrometer allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer has software that allows searching any GC/MS data file for ions of a specific mass and plotting such ion abundances versus time or scan number. The computer allows acquisition at pre-selected mass windows for selected ion monitoring.

6.3 Supplies

6.3.1 Carrier gas: Ultra high-purity helium.

6.3.2 An Rtx®-5MS 30-meter fused silica capillary column, 0.25mm I.D., 0.5 df, or equivalent.

6.4 Reagents and Standards

Refer to Appendix IV for extraction reagents and standards.

6.4.1 Acetone, reagent grade.

6.4.2 Methanol, purge-and-trap grade.

6.4.3 Methylene chloride, reagent grade, tested for purity prior to use per SOP S-T-001 or S-T-001DEN.

6.4.4 Calibration Standard Stocks

6.4.4.1 The primary calibration stock contains all target analytes, including surrogate compounds, at 1.2 µg/mL in methylene chloride

6.4.4.2 The second-source calibration stock contains the conventional PAH compounds (see Table I) at 20 µg/mL in methylene chloride.

6.4.5 Surrogate Spiking Solutions

6.4.5.1 Surrogate solutions are obtained from a commercial vendor. The surrogate compounds include naphthalene-d8, fluorene-d10, and chrysene-d12.

6.4.5.2 mL of the surrogate spike solution is added to samples and QC. In the 4-liter extraction, this results in a final spike concentration of 75 ng/L. In the 1-liter extraction, this results in a final spike concentration of 300 ng/L.

6.4.6 Internal Standard Solutions

6.4.6.1 A solution containing 6 µg/mL is prepared from vendor stock solutions and contains acenapthene-d10, phenanthrene-d10, and perylene-d12.

6.4.7 Matrix Recovery Standard Spiking Solution

6.4.7.1 A solution containing the following compounds at the listed concentrations is prepared by diluting a concentrated stock solution purchased from a vendor. The concentrations of the spiking solutions are shown in the following table:

MATRIX RECOVERY STANDARD SPIKING SOLUTIONS

Compound	Spiking Solution (ng/mL)
Naphthalene	300
Fluorene	300
Chrysene	300
Indene	300

Quinoline	300
Benzo(e)pyrene	300
2-Methylnaphthalene	300

6.4.7.2 LCS spikes are prepared by adding 1.0 mL of the spike solution to reagent water (see Section 25.1). Matrix spikes are prepared by adding 1.0 mL of the spike solution to 1 or 4 liters of sample. The final spike concentration is 75 ng/L for the 4-liter extraction process or 300 ng/L in the 1-liter procedure.

6.4.8 A SIM Second Source Initial Calibration Verification Standard is prepared for the compounds listed in the table below. Due to the difficulty obtaining standards for a number of the compounds, not all analytes have a second source standard.

SECOND SOURCE VERIFICATION STANDARD SOLUTION

Compound	Spiking Solution (ng/L)
1H-Indene	600
Naphathalene	600
2-Methylnaphthalene	600
1-Methylnaphthalene	600
Acenaphthylene	600
Acenaphthene	600
Dibenzofuran	600
Fluorene	600
Phenanthrene	600
Anthracene	600
Carbazole	600
Fluoranthene	600
Pyrene	600
Benzo(a)anthracene	600
Chrysene	600
Benzo(b)fluoranthene	600
Benzo(k)fluoranthene	600
Benzo(a)pyrene	600
Indeno(1,2,3-cd)pyrene	600
Dibenz(a,h)anthracene	600
Benzo(g,h,i)perylene	600

6.4.9 A Full Scan Second Source Initial Calibration Verification Standard is prepared for the compounds listed in the table below. Due to the difficulty obtaining standards for a number of the compounds, not all analytes have a second source standard.

SECOND SOURCE VERIFICATION STANDARD SOLUTION

Compound	Spiking Solution (µg/L)
1H-Indene	100
Naphathalene	100
2-Methylnaphthalene	100
1-Methylnaphthalene	100
Acenaphthylene	100
Acenaphthene	100
Dibenzofuran	100
Fluorene	100
Phenanthrene	100
Anthracene	100
Carbazole	100
Fluoranthene	100
Pyrene	100
Benzo(a)anthracene	100
Chrysene	100
Benzo(b)fluoranthene	100
Benzo(k)fluoranthene	100
Benzo(a)pyrene	100
Indeno(1,2,3-cd)pyrene	100
Dibenz(a,h)anthracene	100
Benzo(g,h,i)perylene	100

7.0 Sample Collection, Preservation, Shipment and Storage

7.1 The samples are collected into four 1-liter or one 1-gallon amber glass containers chilled to 4 ± 2°C and shipped via overnight carrier to the laboratory.

7.2 The samples must be protected from light and refrigerated at 4 ± 2°C from the time of receipt until analysis and storage after analysis.

7.3 Water samples must be extracted within 7 days of the time of sample collection and extracts must be analyzed within 40 days from sample extraction.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
SIM Waters	Liter amber glass	4 liters	Cool 4 ± 2°C	Extraction-7 days Analytical – 40 days	SW846 Chapter 4
Full Scan Waters	Liter amber glass	1 Liter	Cool 4 ± 2°C	Extraction-7 days Analytical – 40 days	SW846 Chapter 4

8.0 Quality Control

8.1 An initial demonstration of capability (IDOC) and a method detection limit study must be performed before samples can be analyzed. Current MDLs are found in the laboratory LIMS system. See section 13 of this SOP for details concerning MDL and IDOC studies.

8.2 Any QC results that fail to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified. The QA group also receives the NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DEN-QA-0031. This is in addition to the corrective actions described in the following sections.

Additional Compounds

8.3 This method is capable of monitoring many compounds other than those listed in Table 1. Prior to reporting an additional compound by this method, a validation procedure for the compound should be performed. It should be noted in the final report if validation procedures have not been performed for an analyte.

The Quality Control measures defined for this method are summarized in the following table:

QC Type	Frequency
Internal Standards (IS)	each sample
Surrogate	each sample
Matrix Spike & Matrix Spike Duplicate (MS/MSD)	1 set per analytical batch or every 20 samples - whichever is most frequent
Laboratory Control Sample (LCS)	1 per analytical batch or every 20 samples - whichever is most frequent
Method Blank (MB)	1 per analytical batch

8.4 Method Blank (MB)

Laboratory reagent water must be treated by extracting with methylene chloride prior to use as method reagent water (see Appendix II, section 11.2).

A Method Blank is analyzed with each analytical batch not to exceed 20 samples.

Due to the low-level nature of this analysis, low-level blank contamination of target analytes is routinely encountered. Target analytes that are detected above the reporting limit in the blank must be flagged on any associated sample's report with a "B" qualifier. For the 4-liter procedure, if the method blank contains any of the carcinogenic PAHs at concentrations greater than the method detection limit (MDL), or any other target PAH compound at a concentration 5 times greater than the MDL, the method blank is considered to be out of control. Corrective action includes reanalysis of the blank extract, an investigation into laboratory sources of contamination, and qualifying the sample data that relates to the blank. For the 1-liter procedure, the method blank is considered in control if all target analytes are less than the RL. The blank contamination level should be considered the minimum level of contamination in all samples that are analyzed with the blanks.

8.5 Matrix Spike and Matrix Spike Duplicate (MS/MSD) Analyses

Samples designated for matrix spike analysis are spiked as described in Appendix IV, section 8. An MS is prepared by spiking a second aliquot of a selected field sample. An MSD is prepared by spiking a third aliquot of the selected sample.

When sufficient sample volume is available (i.e., at least 12 liters), prepare an MS and MSD for each analytical batch not to exceed 20 samples. Generally, sample volume is not sufficient for an MS and MSD, therefore the laboratory analyzes LCS/LCSD samples as requested.

The initial default control limits for the matrix spike are as follows:

Spike Component	Control Limits (% Recovery)
1H- Indene	30 – 150
Naphthalene	30 – 150
Quinoline	30 – 150
2- Methylnaphthalene	30 -150
Fluorene	30 – 132
Chrysene	30 – 132
Benzo(e)pyrene	30 – 150

The average recovery for the spike pair must be within the above criteria with one compound being allowed to fall outside of the acceptance limits.

As sufficient data are collected (ideally at least 30 data points), the matrix spike control limits will be updated to ± 3 standard deviations of the historical mean % recovery. Limits will be routinely updated annually.

If the matrix spike criteria are not met and errors or problems are discovered with sample preparation or instrumentation, then the samples will be reanalyzed. Otherwise, a nonconformance memo (NCM) will be completed, and data for the samples will be reported but qualified as being outside the acceptance criteria of the method.

Projects may have specific QC limits for spike and/or surrogate recoveries, which are communicated to the lab by the Structured Analysis Code.

8.6 Laboratory Control Sample (LCS)

A Laboratory Control Sample (LCS) is analyzed with each analytical batch not to exceed 20 samples.

The LCS results are compared to established control limits, which are the same limits that are used for the matrix spike recovery (see Section 8.5). As sufficient data are collected (ideally at least 30 data points), the control limits will be updated to ± 3 standard deviations of the historical mean % recovery. Limits will be routinely updated annually. One duplicate LCS pair may be required for specific projects.

8.7 Internal Standards

The internal standards are monitored in all field samples and QC samples. The peak area for each internal standard should be between 50% and 200% of the peak area for the continuing calibration standard.

8.8 Surrogate Compound Analysis

All samples and quality control samples are spiked with deuterated PAH surrogate compounds. The surrogate compounds are spiked into the sample prior to extraction. Therefore, evaluation of the surrogate results indicates individual sample matrix effects associated with sample preparation and analysis. Surrogate compounds include naphthalene-d_8, fluorene-d_{10}, and chrysene-d_{12}.

Corrective action is required whenever the surrogate recovery for any one or more surrogates falls outside of the established control limits. The initial default control limits for surrogate recovery are as follows:

Surrogate	Control Limits (% Recovery)
Naphthalene-d_8	30 – 108
Fluorene-d_{10}	41 – 162
Chrysene-d_{12}	30 – 118

As sufficient data are collected (ideally at least 30 data points), the control limits will be updated to ± 3 standard deviations of the historical mean % recovery. Limits will be routinely updated annually.

8.9 Corrective Action

Check calculations to ensure there are no errors;

Check instrument performance;

If the upper control limit is exceeded for only one surrogate, and the instrument calibration, surrogate standard concentration, etc. are in control, it can be concluded that

an interference specific to the surrogate was present that resulted in high recovery. This interference would not affect the quantitation of other target compounds.

If the surrogate could not be measured because the sample required a dilution, no corrective action is required. The recovery of the surrogate is recorded as not calculated (NC).

Reanalyze the extract or re-extract the sample if the steps above fail to reveal a problem. If reanalysis of the extract yields surrogate recoveries within the stated limits, then the reanalysis data will be reported. If re-extraction of the sample yields surrogate recoveries within the stated limits, then both the original and reanalysis data will be reported.

9.0 Procedure

One time procedural variations are allowed only if deemed necessary in the professional judgment of a supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM (see DEN-QA-0031 for details). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified. The QA group also receives the NCMs by e-mail for tracking and trending purposes. If contractually required, the client shall be notified. The NCM shall be filed in the project file. The nonconformance is also addressed in the case narrative.

9.1 Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

9.2 Gas Chromatography/Mass Spectrometry (GC/MS) Analysis

- Perform all aliquotting, extract dilutions, and spike additions in the trace hood using equipment dedicated to SIM analysis.
- Add extract aliquots to 2-mL vials for GC/MS analysis to allow for re-analysis, if necessary.
- Prior to analysis, transfer an aliquot of internal standard solution to the sample vial using a 25-µL or 50-µL syringe to give a final internal standard concentration of 600 ng/mL in the extract.
- Set up the GC/MS system operating conditions as summarized in the following table:

Injector Temperature	275°C
Transfer Line Temperature	290°C or 300°C
Initial Oven Temperature	40°C
Initial Hold Time	0 min.
Ramp Rate	20°C/min. to 120°C 17°C/min. to 150°C 15°C/min. to 325°C

	Hold for 8 min
Final Temperature	325°C

- Inject representative aliquots of each sample extract into the GC/MS system.
- The effluent from the GC capillary column is fed directly into the ion source of the mass spectrometer. The MS is operated in the selected ion monitoring (SIM) mode using appropriate windows to include the quantitation and confirmation masses for each PAH as shown in Table I. All compounds detected at a concentration above the MDL are checked to ensure that the confirmation ion is present at the appropriate ratio.

9.3 Sample Preparation

Please refer to Appendix II.

Please refer to the concentration SOP DV-OP-0007.

9.4 Calibration

Mass tuning is performed using the mass calibration compound FC43. Tuning serves to maximize the sensitivity of the mass spectrometer for the mass range of compounds being analyzed. An autotune program is run which maximizes the sensitivity of the instrument in the mass region of interest for the Extended List PAH analysis. After major system maintenance (e.g. source cleaning, column change, or when the analyst deems necessary), the lab runs the autotune program (atune.u). The autotune program does not allow the operator to set specific mass abundances, so there are no defined criteria. The autotune procedure maximizes sensitivity over the scan range. The instrument may require a mass axis calibration, which is performed at the analysts' discretion. Additionally, peak resolution in autotune shall be left up to the analysts' judgement as to whether or not is affecting the performance of the compounds of interest. Generally if the peak shape and response are adequate to meet the calibration criteria, it is acceptable.

NOTE: The GC/MS is not tuned to meet decafluorotriphenylphosphine (DFTPP) ion abundance criteria. This requirement is not appropriate for selected ion monitoring (SIM) methods. The analyst should tune the instrument to maximize the sensitivity for the compounds being analyzed as described above.

9.4.1 The instrument is calibrated at seven concentration levels. This corresponds to sample concentrations in the 10 to 1200 ng/L range. Any samples with target compounds exceeding the working range must be diluted so that the concentrations of all target compounds fall within the range of the calibration curve.

9.4.2 Calibration standards are made by diluting a stock standard with a concentration of 1.2 µg/mL down to the seven calibration levels in methylene chloride. Internal standard solution is added to the aliquots such that the final concentration of the internal standard compounds is 600 ng/mL. An example of the volumes used to aliquot the calibration levels

can be seen below. Please note that changes in the final volume may be made at the analyst's discretion, so long as the calibration concentrations remain unchanged.

Level	Stock amount	Solvent amount	IS amount	Final Volume
10 ng/mL	4.2 µL	495.8 µL	50 µL	500 µL
20 ng/mL	8.3 µL	491.7 µL	50 µL	500 µL
150 ng/mL	62.5 µL	437.5 µL	50 µL	500 µL
300 ng/mL	125 µL	375 µL	50 µL	500 µL
600 ng/mL	250 µL	250 µL	50 µL	500 µL
800 ng/mL	333.3µL	166.7 µL	50 µL	500 µL
1200 ng/mL	500 µL	none	50 µL	500 µL
600 ng/mL (ICV)	15 µL	485 µL	50 µL	500 µL

9.4.3 The second-source stock used to make the ICV is at a concentration of 20 µg/mL.

9.4.4 If the initial calibration response factors are less than 30 relative percent difference (RPD), sample analysis may proceed. If, for any analyte, the initial calibration response factor is greater than 30 RPD, the initial calibration curve must be repeated for that compound prior to the analysis of samples. The following compounds are excepted for this criteria due to poor response by this method: 7H-dibenzo(c,g)carbazole, 3-methylcholanthrene, and the dibenzopyrene isomers.

NOTE: Generally, it is NOT acceptable to remove points from a calibration for the purposes of meeting calibration criteria, unless the points are the highest or lowest on the curve AND the reporting limit and/or the linear range is adjusted accordingly. The only exception is that a level may be removed from the calibration if the reason can be clearly documented, for example a broken vial. A minimum of five levels must remain in the calibration for a linear regression. The documentation must be retained with the initial calibration. Alternatively, if the analyst believes that a point on the curve is inaccurate, the point may be reanalyzed and the reanalysis used for the calibration. All initial calibration points must be analyzed without any changes to instrument conditions, and all points must be analyzed within 12 hours.

9.4.5 Table II contains example retention times for target compounds.

9.4.6 Initial Calibration Verification (ICV)

Analysis of a second-source initial calibration verification standard is performed after each ICAL using the 600 ng/mL concentration level for each of the conventional PAH compounds shown in Table I. The acceptance criterion for the ICV is the same as for the CCV (+/-35%).

9.4.7 Continuing Calibration (CCV)

Every 12 hours, the mass spectrometer response for each PAH compound relative to the internal standard is determined using the 300 ng/mL calibration standard. The response factors for each compound must be compared to the initial calibration curve. If the continuing calibration response factors are within ± 35 percent of the corresponding calibration curve value, the analysis may proceed. If, for any analyte, the continuing calibration response factor is not within ± 35 percent of the corresponding calibration curve value, a five-point calibration curve must be repeated for that compound prior to the analysis of samples. The following compounds are excepted for this criteria due to poor response by this method: 7H-dibenzo(c,g)carbazole, 3-methylcholanthrene, and the dibenzopyrene isomers.

10.0 Sample Analysis

Qualitative Identification

Obtain electronic ion current profiles (EICP) for the primary m/z and the confirmatory ion. The following criteria must be met to make a qualitative identification:

The characteristic mass ions of each analyte of interest must maximize in the same or within one scan of each other.

For the qualitative identification, the relative retention time (RRT) of unknown peaks must fall within ± 0.06 RRT of the RRT for the compound in the daily calibration standard (mid-point ICAL or daily CCV).

> **NOTE:** The RRT is calculated as the retention time for the target compound divided by the retention time for the nearest internal standard. Therefore, as an example, the earliest eluting IS compound in this SOP is acenapthene-d10, with a typical RT of 8.0 minutes, and ± 0.06 RRT is equal to 0.48 minutes for the associated compounds. In practice, the retention time windows in the Target method are never set tighter than ± 0.2 minutes, which is ≤ 0.025 RRT given the RTs for the IS compounds in this method.

The relative peak areas of the primary ion compared to the confirmation or secondary ion masses in the EICPs must fall within ± 20% of the relative intensities of these masses in a reference mass spectrum. The reference mass spectrum can be obtained from a standard analyzed in the GC/MS system or from a reference library. A compound that

does not meet secondary ion confirmation criteria may still be determined to be present in a sample after close inspection of the data by the mass spectroscopist. Supportive information includes correct relative retention time and the presence of the secondary ion, but the ratio is greater than ± 20% of the primary ion which may be caused by an interference of the secondary ion.

Structural isomers that have very similar mass spectra and less than 30 seconds difference in retention time can be explicitly identified only if the resolution between authentic isomers in a standard mix is acceptable. Acceptable resolution is achieved if there is a definitive inflection between the two peaks, according to the analyst's judgement. Otherwise, structural isomers are identified as isomeric pairs.

11.0 Calculations / Data Reduction

The following formula is used to calculate the response factor for each PAH compound:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

Where:

A_s = Area of the characteristic ion for the parameter to be measured.

A_{is} = Area of the characteristic ion for the internal standard.

C_{is} = Concentration of the internal standard, ng/mL.

C_s = Concentration of the parameter to be measured, ng/mL.

Based on these response factors, the concentration of each PAH compound in the sample extract is calculated using the following formula.

$$C_e = \frac{A_s \times I_s}{A_{is} \times \overline{RF}}$$

Where:

Ce = Sample extract concentration, ng/mL.

A_s = Area of the characteristic ion for the parameter to be measured.

A_{is} = Area of the characteristic ion for the internal standard.

I_s = Amount of internal standard added to each extract, (ng/mL).

$\overline{RF}$ = The average response factor.

The actual sample concentration (C) for each compound is calculated by the following formula:

$$C = C_e \times Df \times \left(\frac{V_E}{V_s}\right)$$

Where:

C	=	Concentration of the sample, ng/mL.
Df	=	Dilution factor.
V_E	=	The final extract volume, mL.
V_s	=	The original volume of sample extracted, L.
C_e	=	The amount measured in the analytical extract, ng/mL.

A second-level technical review of the organic data is performed prior to data reporting. This review is performed by a peer or supervisor using the guidelines and checklists detailed in SOP DV-QA-0020.

12.0 Method Performance

Method Detection Limit

An initial method detection limit study must be performed on each instrument before samples can be analyzed. MDL studies are conducted annually as follows:

Prepare seven samples at three to five times the estimated MDL concentration.

Extract and analyze the MDL standards as described in Section 11.

Calculate the mean concentration found (X) in ng/L, and the standard deviation of the mean concentration in ng/L, for each analyte. Then calculate the MDL (single-tailed, 99% confidence level, as described in Policy DV-QA-005P) for each analyte.

MDL studies are repeated annually, and MDL results are stored in the laboratory LIMS system. Method detection limit studies and concentrations can be located on L:\QA\Read\MDL.

Initial Demonstration of Capability

Each laboratory must make a one-time initial demonstration of capability for each individual method. Demonstration of capability for both soils and water matrices is required (if both matrices are analyzed by the method). This requires analysis of QC check samples containing all of the standard analytes for the method. Complete details are given in SOP DEN-QA-0024.

Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid-level calibration.

Calculate the mean recovery and standard deviation of the mean recovery for each analyte of interest. Compare these results to the control limits in LIMS.

If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

Training Qualification

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

13.0 Pollution Control

Standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents needing disposal.

14.0 Waste Management

All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Corporate Safety Manual, and HS-001, "Waste Management Program."

The following waste streams are produced when this method is carried out:

- Expired Extract Vials – Waste Stream A
- Used methylene chloride – Waste Stream B
- Flammable Solvents – Waste Stream C
- Expired Chemicals/Reagents/Standards – Contact Waste Coordinator

NOTE: Radioactive and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

SW846, Test Methods For Evaluating Solid Waste, Third Edition, Update III, December 1996, Method 3520C, 8270C.

16.0 Method Modifications:

- A 4-liter low-level extraction can be used, in addition to the 1-liter extraction.
- The source method describes preparing IS standards in carbon disulfide and methylene chloride, whereas this procedure uses methylene chloride only.
- HP autotune using FC43 is performed, rather than tuning with DFTPP.
- DDT breakdown checks and tailing checks with benzidine and pentachlorophenol are not performed because the masses scanned for PAH analysis do not include the characteristic ions for these compounds.

- SPCC and CCC compounds, which are used to evaluate calibrations and calibrations in open-scan 8270C, are not part of the SIM analysis.

17.0 Attachments

Table I: Compounds and MS Quantitation Mass Ions

Table II: Example Retention Times for the Compounds Associated With the Low-Level PAH and Heterocycle Methodology

Appendix I: Extended List PAH Analysis by GC/MS Full Scan

Appendix II: Liquid/Liquid Extraction (CLLE) by Method SW-846 3520C for Extended List PAHs for City of St. Louis Park

18.0 Revision History

- Title changed to incorporate 1-liter and Full Scan level procedures.
- References to 1-liter procedure added as necessary.
- Idole interference mentioned in 4.3.
- 4-liter CLLE glassware cleaning procedure updated to reflect current practice.
- Calibration standard concentration information added.
- Surrogate and LCS/MS/MSD spike concentrations for 1-liter procedure added.
- Added section 9.6.6 to refer to project-specific QC limits.
- Sample extraction information is removed and replaced with a reference to Organic Prep SOPs.
- Appendix III added.
- Appendix I and II deleted. Appendix III changed to Appendix I. Name change to TestAmerica and format change.

Table I: Compounds and MS Quantitation Mass Ions*

Conventional Polynuclear Aromatic Hydrocarbons			
Compound	**Mass Ion**	**Confirmation Ion**	**Internal Standard Reference**
Naphthalene	128	102	1
Acenaphthylene	152	151	1
Acenaphthene	154	153	1
Fluorene	166	165	2
Phenanthrene	178	176	2
Anthracene	178	176	2
Fluoranthene	202	200	2
Pyrene	202	200	2
Benzo(a)anthracene	228	226	3
Chrysene	228	226	3
Benzofluoranthenes	252	250	3
Benzo(a)pyrene	252	250	3
Indeno(1,2,3,cd)pyrene	276	274	3
Dibenz(a,h)anthracene	278	279	3
Benzo(g,h,i)perylene	276	274	3

Internal Standards			
Compound	**Mass Ion**	**Confirmation Ion**	**Internal Standard Reference**
Acenaphthene-d_{10}	164	162	1
Phenanthrene-d_{10}	188	184	2
Perylene-d_{12}	264	132	3

* The relative peak areas of the primary ion compared to the confirmation or secondary ion masses in the EICPs must fall within ± 20% of the relative intensities of these masses in a reference mass spectrum.

Table I: Compounds and MS Quantitation Mass Ions (continued)

Surrogates			
Compound	**Mass Ion**	**Confirmation Ion**	**Internal Standard Reference**
Naphthalene-d_8	136	134	1
Fluorene-d_{10}	176	174	2
Chrysene-d_{12}	240	236	3

Heterocycles and Other PAH			
Compound	**Mass Ion**	**Confirmation Ion**	**Standard Reference**
Indene	116	115	1
Indole	117	90	1
2,3-dihydroindene	117	118	1
2,3-benzofuran	118	90	1
Quinoline	129	102	1
Benzo(b)thiophene	134	89	1
2-methylnaphthalene	142	115	1
1-methylnaphthalene	142	115	1
Biphenyl	154	153	1
Carbazole	167	166	2
Dibenzofuran	168	139	1
Acridine	179	178	2
Dibenzothiophene	184	139	2
Perylene	252	250	3
Benzo(e)pyrene	252	250	3
7,12-Dimethylbenz(a)anthracene	256	241	3
3-Methylcholanthrene	268	252	3

Table II: Example Retention Times

Compound	Absolute Retention Time (minutes)
Benzofuran	4.64
Dihydroindene	5.04
Indene	5.14
Naphthalene-d_8(Surr.)	6.56
Naphthalene	6.58
Benzo(b)thiophene	6.66
Quinoline	7.15
Indole	7.69
2-Methylnaphthalene	7.84
1-Methylnaphthalene	8.02
Biphenyl	8.69
Acenaphthylene	9.47
Acenaphthene	9.79
Dibenzofuran	10.12
Fluorene-d_{10}(Surr.)	10.67
Fluorene	10.71
Dibenzothiophene	12.17
Phenanthrene	12.38
Anthracene	12.48
Acridine	12.54
Carbazole	12.75
Fluoranthene	14.42
Pyrene	14.80
Benz(a)anthracene	16.79
Chrysene-d_{12} (Surr.)	16.81
Chrysene	16.84
Benzofluoranthenes	18.46
Benzo(e)pyrene	18.86
Benzo(a)pyrene	18.93
Perylene	19.06
Indeno(1,2,3 cd)pyrene	20.82
Dibenz(ah)anthracene	20.86
Benzo(ghi)perylene	21.36

Appendix I: Extended List PAH Analysis by GC/MS Full Scan

Summary of Method

The Full Scan analysis of the Extended List PAHs is intended for higher-level samples from sites where SIM analysis of samples is being conducted concurrently.

Modifications from the SIM analysis are as follows:

DFTPP is analyzed as the GC/MS tune check and to initiate the 12-hour tune window. The mass spectrometer must be tuned such that the regular 8270c ion abundance criteria are met for DFTPP (see SOP DV-MS-0011). Additionally, the tailing factors are calculated for Pentachlorophenol and Benzidine and a DDT breakdown check is performed.

The instrument is calibrated at six concentration levels. The calibration levels are made by diluting a stock standard with a concentration of 200 µg/mL down to the following concentrations in methylene chloride.

Level	Stock amount	Solvent amount	IS amount	Final Volume
10 µg/mL	25 µL	475 µL	50 µL	500 µL
20 µg/mL	50 µL	450 µL	50 µL	500 µL
50 µg/mL	125 µL	375 µL	50 µL	500 µL
80 µg/mL	200 µL	300 µL	50 µL	500 µL
120 µg/mL	300 µL	200 µL	50 µL	500 µL
160 µg/mL	400 µL	100 µL	50 µL	500 µL
100 µg/mL (ICV)	250 µL	250 µL	50 µL	500 µL

The second source calibration stock is also at 200 µg/mL in methylene chloride. Response factors for each compound must be ≤30% RSD. The following compounds are exempt from this criteria (as well as the criteria for CCVs) due to poor response by this method: 7H-Dibenzo(c,g)carbazole, 3-Methylcholanthrene, and the dibenzopyrene isomers.

Initial Calibration Verification

The second source verification (SSV or ICV) is run at 100 µg/mL. The acceptance criterion for the ICV is the same as for the CCV: ±35%D.

Continuing Calibration Verification

The CCV is run at 80 µg/mL. Criterion: ±35%D.

Sample extraction: See DV-OP-0008.

Internal Standard final concentration is 40 µg/mL in standards and extracts. The stock is at 400 µg/mL

Surrogate spike = 50 µg/mL

LCS/MS/MSD spike = 50 µg/mL

Spike Component	Control Limits (% Recovery)
1H- Indene	49 – 108
Naphthalene	43 – 128
Quinoline	40 – 126
2- Methylnaphthalene	47 -138
Fluorene	51 – 120
Chrysene	43 – 124
Benzo(e)pyrene	30 – 150

GC/MS instrumentation configured the same as in the SIM analysis with the following exceptions: MS is operated in the full scan mode, scanning from amu 35 – 500. GC inlet is operated in the split mode.

APPENDIX II: LIQUID/LIQUID EXTRACTION (CLLE) BY METHOD SW-846 3520C FOR EXTENDED LIST PAHS FOR CITY OF ST. LOUIS PARK

1.0 Scope and Applicaton

This SOP is applicable to the solvent extraction of organic compounds in aqueous samples using a continuous liquid/liquid extractor (CLLE). This is based on SW-846 Method 3520C.

The determinative methods used in conjunction with this procedure are described in SOP DV-MS-0005.

This procedure does not include the concentration and cleanup steps. See the SOP DV-OP-0007 for those details.

2.0 Summary of Method

A measured volume of sample, either1 liter or 4 liters, is placed in a continuous liquid/liquid extractor (CLLE). The pH is adjusted, as required, for efficient extraction of specific compounds. The organic compounds are extracted with methylene chloride for 18 to 24 hours per pH adjustment. The water phase is discarded.

3.0 Definitions

- Extraction Holding Time: The elapsed time expressed in days from the date of sample collection to the date the extraction starts. The holding time is tracked in the laboratory LIMS system, and is the primary basis of prioritizing work.

- Preparation Batch: A group of up to 20 samples that are of the same matrix and are processed together in the same extraction event using the same procedure and lots of reagents and standards.

- Quality Assurance Summary (QAS): Certain clients may require extensive specific project instructions or program QC, which are too lengthy to fit conveniently in the special instructions/client requirements field in LIMS. In those situations, laboratory Project Managers describe the special requirements in a written QAS to address these requirements. QASs are posted on a public drive for easy accessibility by all lab employees. Normally QASs are introduced to analysts in an initial project kick-off meeting to be sure that the requirements are understood.

- QC Program: The QC Program is the portion of the LIMS structured analysis code (SAC) assigned to a sample that defines the client QA program that applies to the analytical work. For example,

  01 = Standard commercial work
  9G = AFCEE program under the Version 3.1 QAPP

  As such, the QC Program defines in the LIMS database a specific set of spike compounds, spike levels, and control limits. It is necessary to check the QC Program to know which spike solution and how much spike solution to use for batch QC.

- Quality Assurance Summary (QAS): Certain clients may require extensive specific project instructions or program QC, which are too lengthy to fit conveniently in the special instructions/client requirements field in LIMS. In those situations, laboratory Project Managers describe the special requirements in a written QAS to address these requirements. QASs are posted on a public drive for easy accessibility by all lab employees. Normally QASs are introduced to analysts in an initial project kick-off meeting to be sure that the requirements are understood.

- Aliquot: A part which is a definite fraction of a whole; as aliquot samples for testing or analysis. "Aliquot" is also used as a verb meaning to take all or part of a sample for preparation, extraction, and/or analysis.

4.0 Interferences

- Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

- Visual interferences or anomalies (such as foaming, emulsions, odor, etc.) must be documented.

- The most common interference is laboratory contamination, which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, and this document.

5.1 Specific Safety Concerns

CLLE glassware is often times fragile and awkward to use. Care should be taken to prevent breakage and injury.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating.

NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the Material Safety Data Sheet (MSDS) for each of the materials listed in the table.

A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Safety Information for Primary Materials Used

Material [1]	Hazards	Exposure Limit [2]	Signs and Symptoms of Exposure
Methylene Chloride	Carcinogen Irritant	25 ppm (TWA) 125 ppm (STEL)	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting, and headache. Causes irritation, redness, and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sodium Hydroxide	Corrosive Poison	2 mg/m^3	Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract depending on severity of exposure. Symptoms may include sneezing, sore throat, runny nose. Swallowing may cause severe burns of mouth, throat, and stomach. Sever scarring of tissue and death may result. Symptoms include bleeding, vomiting, diarrhea. Damage may appear days after exposure. Skin and eye contact causes irritation or severe burns, blindness.
Methanol	Flammable Poison	200 ppm (TWA) 260 mg/m^3 (TWA)	May be fatal or cause blindness if swallowed. Inhalation, ingestion, or skin absorption can cause significant disturbances in vision, including blindness. Prolonged and/or repeated contact may cause defatting of the skin and dermatitis. Inhalation of methanol vapor initially causes central nervous system depression with nausea, headache, vomiting, dizziness, and incoordination. Prolonged exposure leads to metabolic acidosis and sever visual effects, up to and including blindness and/or nervous system effect.
Sulfuric Acid	Corrosive Carcinogenic	1 mg/m3	Inhalation: Causes irritation of nose and throat and labored breathing. Chronic exposure to mist is a cancer hazard. Skin Contact: Redness, pain, severe burns. Circulatory collapse with clammy skin, weak and rapid pulse, shallow respiration. Ingestion: Severe burns of mouth, throat, stomach, leading to death.
Acetone (used for cleaning glassware)	Flammable Irritant	1000 ppm (TWA)	Inhalation of vapors irritates respiratory tract. May cause coughing, dizziness, dullness, and headache. Can produce nervous system depression, narcosis, and unconsciousness. Irritating to skin due to defatting action. Causes redness, pain, drying, and cracking of the skin. Vapors are irritating to eyes.
Always add acid to water to prevent violent reactions. Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

NOTE: All glassware used in this procedure, is cleaned following SOP DV-OP-0004. In addition, the glassware is rinsed with methylene chloride immediately prior to use. For the 4-liter CLLEs used in the part-per-trillion level analysis of PAHs, please see Appendix II, section 11.2 and section 11.10 for instructions on how to clean the CLLEs after extraction.

- Continuous Liquid-Liquid Extractor (CLLE), 1 and 4 liter in size, equipped with ground glass joints and polytetrafluoroethylene (PTFE) stopcock. (See Figure 1.)
- Snyder Column
- 250 mL boiling flask with ground glass joint.
- Boiling Chips: Contaminant free, approximately 10/40 mesh, Teflon®, PTFE, carbide, or equivalent.
- Cooling Condensers
- Heating Mantle: Rheostat controlled.
- Balance: >1400 g capacity, accurate to ±1 g, calibration checked daily per SOP DV-QA-0014.
- pH indicator paper, wide range.
- Teflon® stir rods.
- Pipetter with disposable 1.0 mL tips, calibration checked daily per SOP DV-QA-0008.
- Aluminum Foil
- Re-circulating Chiller – Set at 10°C.

7.0 Reagents and Standards

7.1 Reagents - All materials must be reagent grade or higher quality, unless otherwise specified

- Reagent Water - TeatAmerica Denver has two ELGA water purification systems. The water coming from the ELGA system should be 17 – 18.2 Mohm-cm. The performance of the water polishing system is checked daily and recorded per SOP DV-QA-0026. Reagent water is prepared by adding approximately 1 gram of baked sodium chloride to 1 L of the organic-free de-ionized water from the ELGA water system. This is done to give the reagent water some ionic strength to more closely mimic the matrix of actual water samples. Record the lot number of the sodium chloride on the bench sheet.
- Methylene Chloride - Each lot of solvent is tested following SOP S-T-001 or S-T-001 DEN-1 before it is put into use. QA personnel post the list of approved lots at solvent storage areas.

- Methanol - Purge and Trap Grade. For use in preparing working LCS and surrogate standards

- Acetone - Reagent Grade. For use in glassware cleaning.

- Sodium Hydroxide (NaOH), 10 N - Purchased at ready-to-use concentration from commercial vendors.

- Sufuric Acid, (H2SO4), 1:1 - While stirring, slowly add 1 part concentrated reagent grade sulfuric acid (36 N) to 1 part reagent water in a container that is sitting in an ice water bath and on a stir plate.

- Sodium Chloride - Bake in a 400 °C oven for at least 4 hours

7.2 Standards - Stock standards are obtained from commercial sources.

- Verification - All standards are subject to verification using a second-source standard before they are used for sample analysis. This process is described in SOP DV-QA-0015.

- Storage Requirements - All standards must be refrigerated at 4 ±2 °C and protected from light. Standard solutions are allowed to adjust to room temperature before using.

- Expiration Date of Standards - Stock standards are monitored for signs of degradation or evaporation. The standards must be replaced annually from the date of receipt or earlier if the vendor indicates an earlier date. Dilutions from stock standards cannot have a later expiration date than the date assigned to the parent stock solutions. The standards must be replaced at least annually or sooner if comparison with check standards indicates a problem.

- Surrogate Spiking Solutions – A solution containing the following compounds at the listed concentrations is prepared by diluting in purge and trap methanol a concentrated stock solution purchased from a vendor. The concentrations of the spiking solutions are shown in the following table:

Surrogate Compound	Working Surrogate Solution for Full Scan Analysis	Working Surrogate Solution for SIM Analysis
Naphthalene-d8	50 µg/mL	0.3 µg/mL
Fluorene-d10	50 µg/mL	0.3 µg/mL
Chrysene-d12	50 µg/mL	0.3 µg/mL

1.0 mL of the surrogate spike solution is added to samples and QC. In the 4-liter SIM extraction, this results in a final surrogate concentration of 75 ng/L. In the 1-liter SIM extraction, this results in a final surrogate concentration of 300 ng/L.

- Laboratory Control Sample and Matrix Spike Solution - TestAmerica Denver QC Policy DV-QA-003P requires that the same spike solution and spike amount be used for LCSs and MS/MSDs.

  A solution containing the following compounds at the listed concentrations is prepared by diluting in purge and trap methanol a concentrated stock solution purchased from a vendor. The concentrations of the spiking solutions are shown in the following table:

Spike Compound	Working Spike Solution for Full Scan Analysis	Working Spike Solution for SIM Analysis
Acenaphthene	50 µg/mL	0.3 µg/mL
Acenaphthylene	50 µg/mL	0.3 µg/mL
Acridine	50 µg/mL	0.3 µg/mL
Anthracene	50 µg/mL	0.3 µg/mL
Benz(a)anthracene	50 µg/mL	0.3 µg/mL
2,3-Benzofuran	50 µg/mL	0.3 µg/mL
Benzo(b)fluoranthene	50 µg/mL	0.3 µg/mL
Benzo(k)fluoranthene	50 µg/mL	0.3 µg/mL
Benzo(a)pyrene	50 µg/mL	0.3 µg/mL
Benzo(e)pyrene	50 µg/mL	0.3 µg/mL
Benzo(g,h,i)perylene	50 µg/mL	0.3 µg/mL
Benzo(b)thiophene	50 µg/mL	0.3 µg/mL
Biphenyl	50 µg/mL	0.3 µg/mL
Carbazole	50 µg/mL	0.3 µg/mL
Chyrsene	50 µg/mL	0.3 µg/mL
7H-Dibenzo(c,g)carbazole	50 µg/mL	0.3 µg/mL
Dibenz(a,h)acridine	50 µg/mL	0.3 µg/mL
Dibenz(a,j)acridine	50 µg/mL	0.3 µg/mL
Dibenz(a,h)anthracene	50 µg/mL	0.3 µg/mL
Dibenzofuran	50 µg/mL	0.3 µg/mL
Dibenzothiophene	50 µg/mL	0.3 µg/mL
Dibenzo(a,e)pyrene	50 µg/mL	0.3 µg/mL
Dibenzo(a,i)pyrene	50 µg/mL	0.3 µg/mL
Dibenzo(a,h)pyrene	50 µg/mL	0.3 µg/mL
Dibenzo(a,l)pyrene	50 µg/mL	0.3 µg/mL
2,3-Dihydroindene	50 µg/mL	0.3 µg/mL
7,12-Dimethylbenz(a)anthracene	50 µg/mL	0.3 µg/mL
2,6-Dimethylnaphthalene	50 µg/mL	0.3 µg/mL
Di-n-octyl phthalate	50 µg/mL	0.3 µg/mL
Fluoranthene	50 µg/mL	0.3 µg/mL
Fluorene	50 µg/mL	0.3 µg/mL
Indene	50 µg/mL	0.3 µg/mL

Indole	50 µg/mL	0.3 µg/mL
Indeno(1,2,3-cd)pyrene	50 µg/mL	0.3 µg/mL
3-Methylcholanthrene	50 µg/mL	0.3 µg/mL
6-Methylchrysene	50 µg/mL	0.3 µg/mL
1-Methylnaphthalene	50 µg/mL	0.3 µg/mL
2-Methylnaphthalene	50 µg/mL	0.3 µg/mL
1-Methylphenanthrene	50 µg/mL	0.3 µg/mL
Naphthalene	50 µg/mL	0.3 µg/mL
Perylene	50 µg/mL	0.3 µg/mL
Phenanthrene	50 µg/mL	0.3 µg/mL
Pyrene	50 µg/mL	0.3 µg/mL
2,3,5-Trimethynaphthalene	50 µg/mL	0.3 µg/mL
Quinoline	50 µg/mL	0.3 µg/mL

1.0 mL of the surrogate spike solution is added to samples and QC. In the 4-liter SIM extraction, this results in a final surrogate concentration of 75 ng/L. In the 1-liter SIM extraction, this results in a final surrogate concentration of 300 ng/L.

8.0 Sample Collection, Preservation and Storage

Sample holding time for aqueous samples is 7 days. Aqueous samples are stored refrigerated at 4 ± 2 °C in amber glass bottles with Teflon-lined lids.

If the holding time is exceeded, a Nonconformance Memo (NCM) must be prepared and the client notified immediately.

9.0 Quality Control

The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS QC program code and special instructions to determine specific QC requirements that apply.

- The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, Quality Control Program.
- Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), AFCEE, etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs.
- Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via special instructions in the LIMS.
- Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the

following sections.

- Preparation Batch - A standard batch can contain up to 20 field samples, not including MS/MSDs. Each preparation batch must contain a method blank, an LCS, and an MS/MSD pair. Special program or project requirements can include additional requirements. Always refer to special project instructions for details before proceeding with the analysis.

- Method Blank (MB)

  At least one MB must be processed with each preparation batch. The MB is used to identify any background interferences or contamination of the analytical system that may lead to the reporting of elevated concentration levels or false positive data.

  For the Full Scan analysis and the 1-liter part-per-billion SIM analysis, MBs are prepared using 1,000 mL of reagent water spiked with the surrogates.

  For the 4-liter part-per-trillion SIM analysis, MBs are prepared using 4,000 mL of pre-extracted reagent water spiked with the surrogates

  Acceptance Criteria: The result for the method blank must be less than the reporting limit for the analyte(s) of interest or less than 5% of the analyte concentration found in the associated samples, whichever is higher.

  Corrective Action: If the MB exceeds the acceptance limit for the determinative method and/or the project, the general requirement in Policy DV-QA-003P is to re-extract and reanalyze all associated samples.

- Laboratory Control Sample (LCS)

  At least one LCS must be processed with each preparation batch. Some projects require two LCSs (LCS and LCSD) in every batch – check special project instructions. The LCS is used to monitor accuracy of results within the batch and long-term precision. It provides evidence that the method is being performed properly, independent of any sample matrix effects.

  For the Full Scan and the 1-liter part-per-billion SIM analysis, LCSs are prepared using 1,000 mL of reagent water that is spiked with the LCS standards and surrogate compounds, as discussed in Sections 7.11 and 7.12.

  For the 4-liter part-per-trillion SIM analysis, LCSs are prepared using 4,000mL of reagent water that is spiked with the LCS standards and surrogate compounds. , as described in Section 7.

  Acceptance Criteria: The recovery results for the LCS must fall within the established control limits. Control limits are set at ± 3 standard deviations around the historical mean and must be no wider than the limits specified in the reference methods or by specific projects, if applicable.

  Corrective Action: Because LCSs are completely under the laboratory's control, failing LCSs generally lead to re-extraction and reanalysis of associated samples. On-line control charts of LCS results over time are available and are useful for trouble-shooting method performance problems.

- Matrix Spike/Matrix Spike Duplicate (MS/MSD)

  A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked exactly as the MS) prepared and analyzed along with the sample and matrix spike. One MS/MSD pair must be processed for each preparation batch. Samples identified as field blanks or equipment blanks cannot be used for MS/MSD analysis. If insufficient sample volume is available for MS/MSD, an NCM must be written. Some clients may require a LCSD in this case.

  Acceptance Criteria: The recovery results for the MS and MSD must fall within the established control limits, which are set at ± 3 standard deviations around the historical mean, unless otherwise dictated by project requirements. The relative percent difference (RPD) between the MS and MSD must be less than the established RPD limit, which is set at 3 standard deviations above the historical mean.

  Corrective Action: If the method blank and LCS results are within control limits and the MS/MSD results fail to meet control limits, the general requirement is to qualify the results reported to the client using data qualifier flags and/or comments in the final report case narrative.

- Surrogate Spikes

  Each sample, method blank, LCS, and MS/MSD in the batch is spiked with surrogate standard. Surrogates are organic compounds which are similar to the target analytes in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples. Surrogates are used to monitor the accuracy of results for each sample.

  Acceptance Criteria: Surrogate spike recoveries are evaluated against percent recovery control limits, which are set at ± 3 standard deviations around the historical mean, unless otherwise dictated by project requirements.

  Corrective Action: If LCS and method blank results are within control limits, but sample surrogate recoveries are outside of control limits, the general requirement is to qualify the results reported to the client using data qualifier flags and/or comments in the final report case narrative. However, some projects require automatic re-extraction when surrogates fail. Any special requirements such as this will be documented in special project instructions and/or the QAS.

10.0 Calibration and Standardization

Not applicable.

11.0 Procedure

One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

11.1 Critical Procedural Considerations

As stated throughout this SOP, analysts must review client requirements, the LIMS SAC, and any applicable QASs before starting work. This review is also documented on the Organic Extraction Checklist (see Attachment 1).

Focus on using clean technique throughout this procedure is required. Any parts or pipettes that come into direct contact with dirty surfaces or any other CLLE than the designated one should be cleaned or disposed of before coming into contact with the sample.

11.2 Special Pre-Extraction Procedure for 4-liter part-per-trillion SIM analysis

- For each sample and QC sample, rinse a 250 mL boiling flask with methylene chloride. Add a few boiling chips and fill with about 150 mL of methylene chloride.
- For each sample and QC sample, rinse a 3-ball Snyder column with methylene chloride.
- For each sample and QC sample, rinse a 4-liter CLLE with methylene chloride. Then fill the bottom of the CLLEs with methylene chloride to a level approximately 2 cm above the opening of the solvent flow arm. (See Figure 1.)
- Set the CLLE on a stand in front of a heating mantle and under a slot hood. Attach the 250 mL boiling flask containing methylene chloride and boiling chips to the Snyder column and attach the Snyder column to the side arm of the CLLE and place a heating mantle under the flask.
- With the stopcock of the CLLE open, add reagent water to each CLLE until the methylene chloride in the bottom of the CLLE starts to spill over through the solvent flow arm and into the boiling flask. Be careful not to allow water to spill over. You may need to add additional methylene chloride to the CLLE to prevent water from entering the solvent flow arm.
- Attach the cold condenser (chilled at 10 °C). Check that the hoses are not kinked. Check the temperature and the water level of the chiller.

- Check again for boiling chips in the boiling flask. Turn on the heating mantle. Inspect joints for leaks once solvent has begun cycling.
- Extract for at least 18 hours.
- At the end of the pre-extraction, turn off the heating mantle and allow the extractor to cool. Remove the boiling flasks and discard the methylene chloride in the appropriate waste container.
- For the CLLEs to be used for the extraction of samples and MS/MSDs, remove the water and methylene chloride from the extractor body, discarding them in the appropriate waste containers. Return the CLLEs to the stands under the slot hood. For the CLLEs to be used for the extraction of method blanks and LCSs, the water and the small amount of methylene chloride are kept in the CLLE. This pre-extracted water will be used as the method blank and LCS samples.
- Rinse a fresh 250 mL boiling flask with methylene chloride. Add a few boiling chips and fill with about 150 mL of methylene chloride. Attach these boiling flasks to the Snyder columns on the side arms of the CLLEs.
- Fill the bottom of the CLLEs to be used for samples and MS/MSDs with methylene chloride to a level approximately 2 cm above the opening of the solvent flow arm. (See Figure 1.)
- Label each boiling flask with the sample ID or batch QC ID.
- Proceed to Appendix II, section 11.3.

11.2 Assemble and Clean the Glassware for the Full Scan and the 1-liter part-per-billion SIM analysis.

- For each sample and QC sample, rinse a 250 mL boiling flask with methylene chloride. Add a few boiling chips and fill with about 150 mL of methylene chloride.
- For each sample and QC sample, rinse a CLLE with methylene chloride. Then fill the bottom of the CLLEs with methylene chloride to a level approximately 2 cm above the opening of the solvent flow arm. (See Figure 1.)
- Set the CLLE on a stand in front of a heating mantle and under a slot hood. Attach the 250 mL boiling flask containing methylene chloride and boiling chips to the side arm of the CLLE and place a heating mantle under the flask.
- Label each boiling flask with the sample ID or batch QC ID.

11.3 Aliquot Samples

- Weigh the bottle containing the approximately 1-liter sample and record the gross weight to the nearest gram. For the 4-liter part-per-trillion analysis weigh 4 bottles.
- Measure the initial sample pH with wide-range pH paper and record the pH on the extraction benchsheet.
- Open the stopcock if the CLLE is equipped with one. Pour the sample into the CLLE on top of the methylene chloride. Attempt to transfer the entire sample into the CLLE. Be careful to allow only the methylene chloride in the bottom of the CLLE to spill over into the boiling flask, and not to allow water to spill over. Add additional methylene chloride or reagent water to the CLLE if needed.
- Rinse the sample containers with methylene chloride and add the solvent to the CLLE.

- Reweigh the bottle and record the sample volume on the benchsheet to the nearest milliliter, assuming a density of 1.0 g/mL.

11.4 Prepare Method Blanks

- See Section 11.2 for method blanks for the 4-liter part-per-trillion SIM analysis. For the Full Scan and 1-liter part-per-billion SIM analyses, add approximately 1000 mL of reagent water to CLLEs marked for the method blanks. Add enough reagent water to cause the methylene chloride to begin to pour over the arm and into the boiling flask.

11.5 Prepare LCS and MS/MSD QC

- See Section 11.2 for LCSs for the 4-liter part-per-trillion SIM analysis. For the Full Scan analysis and the 1-liter part-per billion SIM analysis, add approximately 1000 mL of reagent water to CLLEs marked for the LCS.
- Using a calibrated pipette, add 1.0mL of the appropriate working LCS and MS/MSD standard to the CLLE containing any LCS, LCSD, MS, and MSD samples. Record the ID of the standard used on the benchsheet. Note that the standard should be allowed to come to room temperature before spiking the samples.
- Spiking events must be witnessed by a second person, and the witnessing is documented on the Organic Extraction Checklist (see Attachment 1).

11.6 Add Surrogates to All Field and QC Samples

- Using a calibrated pipette, add 1.0 mL of the appropriate working surrogate standard to the CLLEs for each field sample and QC sample. Record the ID of the standard used on the benchsheet.

11.7 Adjust pH of Samples

- Adjust sample pH as indicated in the chart below using a minimum amount of 1:1 sulfuric acid or 10 N sodium hydroxide, as necessary. Add acid in 2-mL increments. Add base in 5-mL increments.

Method	Initial Extraction pH	Secondary Extraction pH
SIM (both 1 Liter and 4 Liter extraction)	≥ 12	NA
Full Scan	≤ 2	≥ 12

- Stir the sample using a Teflon stir-rod. Check the pH and record the adjusted pH and the lot number of the acid or base on the benchsheet.

11.8 Start the Initial Extraction

- Attach the cold condenser (chilled at 3-10 °C). Check that the hoses are not kinked. Check the temperature and the water level of the chiller.
- Turn on the heating mantle. Check again for boiling chips in the boiling flask and inspect joints for leaks once solvent has begun cycling. The methylene chloride in the round bottom flask should be boiling steadily, but no too rapidly that the

condenser cannot condense the vapor as fast as it is being generated. The Snyder column should be chattering, but should not flood.

- Record the date and time the extraction started on the benchsheet.
- Extract for 18 to 24 hours.
- At the end of the extraction, turn off the heating mantle and allow the extractor to cool. Record the date and time the extraction stopped on the benchsheet.
- Remove the boiling flask, cap with aluminum foil, and store refrigerated until concentration. For Full Scan analysis, extraction at a second pH is required, continue on to Appendix II, section 11.9. For 1-liter and 4-liter SIM analysis extraction at a second pH is not required, go to Appendix II, section 11.10.

11.9 Start the Secondary Extraction

- Attach a boiling flask with fresh methylene chloride and boiling chips to the CLLE. Label each boiling flask with the sample ID or batch QC ID. Also include on the label what fraction, acid or base.
- Remove the condenser and adjust the pH of the sample in the extractor body to the pH indicated in Appendix II, section 11.7 with the minimum amount of 10N NaOH.
- Stir the sample using a Teflon stir-rod. Check the pH and record the adjusted pH and the lot number of the acid or base on the benchsheet.
- Attach the cold condenser (chilled at 3-10 °C). Check that the hoses are not kinked. Check the temperature and the water level of the chiller.
- Turn on the heating mantle. Check again for boiling chips in the boiling flask and inspect joints for leaks once solvent has begun cycling. The methylene chloride in the round bottom flask should be boiling steadily, but no too rapidly that the condenser cannot condense the vapor as fast as it is being generated. The Snyder column should be chattering, but should not flood.
- Record the date and time the extraction started on the benchsheet.
- Extract for 18 to 24 hours.
- At the end of the extraction, turn off the heating mantle and allow the extractor to cool. Record the date and time the extraction stopped on the benchsheet.
- Remove the boiling flask, cap with aluminum foil, and store refrigerated until concentration.

11.10 Break-down the Extraction Apparatus

- Dispose of the methylene chloride remaining in the CLLE body in the methylene chloride Waste Stream B.
- Dispose of the basic aqueous sample in Waste Stream Y.
- Initial weights and volumes of samples are entered into LIMS, and the transcribed data must be verified by a second person. This verification is documented on the Organic Extraction Checklist (see Attachment 1).
- Wash the CLLEs per DV-OP-0004. After following the steps in that procedure, rinse the 4-liter CLLEs and their Snyder columns with acetone to remove any water. Then rinse with methylene chloride. Cap the openings with aluminum foil and store in a close cupboard.

12.0 Method Performance

- Extraction Holding Time: The elapsed time expressed in days from the date of sample collection to the date the extraction starts. The holding time is tracked in the laboratory LIMS system, and is the primary basis of prioritizing work.

- Method Detection Limit (MDL) Study: An initial method detection limit study is performed in accordance with Policy DV-QA-005P. An MDL verification is performed once a year to satisfy NELAC 2003 requirement. For DoD, AFCEE, and DOE projects, an MDL verification is performed quarterly. MDLs are stored in the LIMS.

- Initial Demonstration of Capability: IDOCs must be performed initially, before samples are analyzed, and must be repeated on an annual basis. Each analyst performing this procedure must successfully analyze four LCS QC samples using current laboratory LCS control limits. The results of the IDOC study are summarized in the NELAC format, as described in SOP DV-QA-0024. IDOCs are approved by the Quality Assurance Manager and the Technical Director. IDOC records are maintained by the QA staff in the central training files.

  Training Qualification: The Group/Team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience. See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

13.0 Pollution Prevention

- The volume of spike solutions prepared is minimized to reduce the volume of expired standard solutions requiring hazardous waste disposal.

14.0 Waste Management

All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Corporate Safety Manual, and HS-001, "Waste Management Program."

The following waste streams are produced when this method is carried out:

- Methylene chloride – Waste Stream B.
- Flammable solvents – Waste Stream C.
- Basic aqueous sample waste saturated with methylene chloride – Waste Stream X.
- Expired Standards/Reagents – Contact Waste Coordinator for guidance.

15.0 References

Method 3520C, Continuous Liquid-Liquid Extraction, Revision 3, December, 1996, SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

16.0 Miscellaneous

16.1 Modifications from SW-846 Method 3520C

- Section 7.1 of the method calls for initial sample volume to be determined volumetrically. This SOP allows the initial sample volume to be determined by weight.
- Section 7.2 of the method calls for the surrogate standard to be added to the sample while the sample is in the sample container or the graduated cylinder used for measuring the initial volume. This SOP calls for the surrogate standard to be added to the sample after the sample is transferred to the CLLE.
- Table 1 of the method calls for the secondary extraction pH for method 8270C to be >11. This SOP calls for the secondary extraction pH for method 8270C to be ≥12.

16.2 Modifications from Previous Version

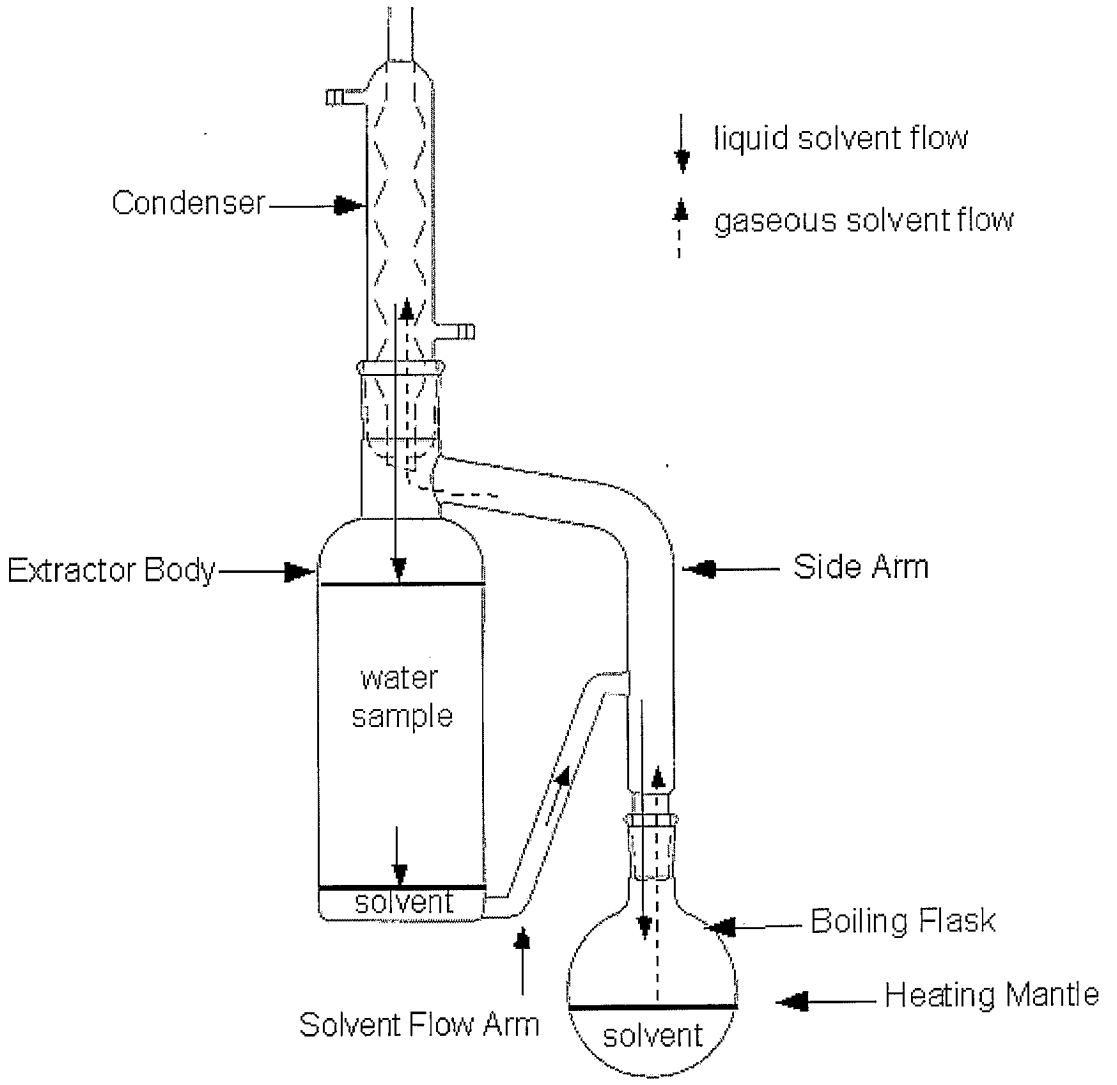
- Snyder columns are now in use on the CLLEs to help monitor the extraction rate and to prevent loss of lighter compounds.

17.0 Tables, Figures, and Attachments

Figure 1. Continuous Liquid-Liquid Extractor (CLLE)

Figure 2. Organic Extraction Checklist

Figure 1. Continuous Liquid-Liquid Extractor (CLLE)



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ORGANIC EXTRACTION CHECKLIST			
Batch:	**Test:**		**Date:**
	EXTRACTIONIST	WITNESS	CONCENTRATIONIST / REVIEWER
Reviewed Client Requirements			
Reviewed QC Code			
Reviewed QAS, QAPP Requirements			
Client/Program Required QA/QC Samples were performed			
The number of samples in this batch does not exceed the acceptable QC to Sample ratio for this method or project/program			
Correct Spike Used			
Correct Surrogate Used			
Mechanical pipette set at correct volume			
The correct solvents were used for this method			
Clean-up procedures used were applicable to the method			
The final sovlent and final extract volume are appropriate for the method			
NCMs written for any anomalies, hold time violations			
Handwritten initial weights, volumes, and calculation entered into LIMS verified			Reviewed by 2nd Person:
Handwritten solvent lot#s, SOP#s, and other notes entered into LIMS			Reviewed by 2nd Person:
Labels checked.			
Extracts are acceptable for delivery			

L:\QA\Edit\FORMS\Organic Prep\Organic Extraction Checklist 5/4/07

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THE LEADER IN ENVIRONMENTAL TESTING

Title: GC/MS Analysis Based On Methods 8270C and 625

Approvals (Signature/Date):

William Rhoades 5/28/08 William Rhoades Date Technical Manager	Adam W. Alban 12 June 08 Adam Alban Date Health & Safety Manager / Coordinator
Karen Kuoppala 6-13-08 Karen Kuoppala Date Quality Assurance Manager	Robert C. Hanisch 6/13/08 Robert C. Hanisch Date Laboratory Director

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1.0 Scope and Application

1.1 This method is based upon standard method SW846 8270C, and is applicable to the determination of the concentration of semivolatile organic compounds in extracts prepared from solid and aqueous matrices.

1.1.1 The modifications presented in Appendix A may be followed for analysis of wastewater following method 625.

1.1.2 The modifications presented in Appendix B may be followed for analysis of wastewater following method 8270 (best practices).

1.1.3 The modifications presented in Appendix C may be followed for analysis of wastewater following the isotopic dilution method.

1.1.4 Direct injection of a sample may be used in limited applications.

1.1.5 Refer to Tables 1 and 2 for the list of compounds applicable for this method. Note that the compounds are listed in approximate retention time order. This method may be amenable to additional compounds. If non-standard analytes are required, they must be validated by the procedures described in section 13 before sample analysis.

1.2 The following compounds may require special treatment when being determined by this method:

- Benzidine can be subject to oxidative losses during solvent concentration and exhibits poor chromatography. Neutral extraction should be performed if this compound is expected.
- Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.
- N-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be distinguished from diphenylamine.
- Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, benzoic acid, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.
- 3-Methylphenol cannot be separated from 4-methylphenol by the conditions specified in this method. They are reported as ¾-methylphenol.
- Hexachlorophene and famphur analysis are not quantitatively reliable by this method.
- Kepone should be analyzed by GC/ECD.

1.3 The standard reporting limit (SRL) of this method for determining an individual compound is approximately 0.33 mg/kg (wet weight) for soil/sediment samples, 1 - 200 mg/kg for wastes (dependent on matrix and method of preparation), and 10 µg/L for groundwater samples. Some compounds have higher reporting limits. Refer to Tables 1 and 2 for

specific SRLs. Reporting limits will be proportionately higher for sample extracts that require dilution.

2.0 Summary of Method

2.1 Aqueous samples are extracted with methylene chloride using a continuous extractor or a separatory funnel.

2.2 Solid samples are extracted with methylene chloride / acetone using sonication or Soxhlet extraction. The extract is dried, concentrated to a volume of 1 mL, and analyzed by GC/MS.

2.3 Waste dilution is used for samples that are miscible with the solvent.

2.4 Extraction procedures are detailed in the following SOPs:

DV-OP-0006	Extraction of Aqueous Samples by Separatory Funnel, SW846 3510C and EPA 600 Series
DV-OP-0007	Concentration of Organic Extracts, SW846 3510C, 3520C, 3540C, 3550B, and EPA 600 Series
DV-OP-0008	Extraction of Aqueous Samples by Continuous Liquid/Liquid Extraction (CLLE) by Method SW-846 3520C and Methods 625 and 607
DV-OP-0009	Ultrasonic Extraction of Solid Samples, SW846 3550B
DV-OP-0010	Soxhlet Extraction of Solid Samples, SW846 3540C

2.5 Qualitative identification of the analytes in the extract is performed using the retention time and the relative abundance of characteristic ions. Quantitative analysis is performed using the internal standard technique with a single characteristic ion.

3.0 Definitions

3.1 CCC (Calibration Check Compounds) - A subset of target compounds used to evaluate the calibration stability of the GC/MS system. A maximum percent deviation of the CCCs is specified for calibration acceptance.

3.2 SPCC (System Performance Check Compounds) - Target compounds designated to monitor chromatographic performance, sensitivity, and compound instability or degradation on active sites. Minimum response factors are specified for acceptable performance.

3.3 Batch - The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The Quality Control batch must contain a matrix spike / matrix spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank (MB). In some cases, at client request, the MS/MSD may be replaced with a matrix spike and sample duplicate. Batches are defined at the sample preparation stage. Batches should be kept together through the whole analytical process to the extent possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. Refer to the TAL QC Program document (DV-QA-003P) for further details of the batch definition.

3.4 Method Blank (MB) - An analytical control consisting of all reagents, internal standards and surrogate standards that is carried through the entire analytical procedure. The method blank is used to define the level of laboratory background and reagent contamination.

3.5 Laboratory Control Sample (LCS) - A blank matrix (reagent water or Ottawa Sand) spiked with the analytes of interest that is carried through the entire analytical procedure. Analysis of this sample with acceptable recoveries of the spiked analytes demonstrates that the laboratory techniques for this method are acceptable.

3.6 Matrix Spike (MS) – An aliquot of a matrix (water or soil) fortified (spiked) with known amounts of specific analytes and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

3.7 Matrix Spike Duplicate (MSD) - A second aliquot of the same sample as the matrix spike (above) that is spiked in order to determine the precision of the method by measuring the relative percent difference (RPD) between the MS and MSD results.

3.8 Surrogates - Organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples. Each sample, blank, LCS, MS, and MSD is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits.

4.0 Interferences

4.1 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the sample. Cleanup procedures may help to eliminate select interferences, as follows:

- Method 3640A, Gel-Permeation Chromatography - Removes higher molecular weight hydrocarbons by size exclusion chromatography, which is most frequently used for biological samples
- Method 3660B, Sulfur Cleanup – If a sulfur peak is detected, copper or mercury can be used to treat the extract and remove the sulfur
- Other, more aggressive cleanup procedures listed in SW-846 may be used for select compounds listed in this procedure, but may cause degradation of some of the more reactive compounds. Consult with a technical expert in the laboratory for more difficult interference problems.

Details concerning cleanup steps are described in the organic extraction SOPs (see Section 2.4).

4.2 Contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts may cause method interferences. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section (Section 9.3). Raw GC/MS data from all blanks, samples, and spikes must be evaluated for

interferences. If an interference is detected, it is necessary to determine if the source of interference is in the preparation and/or cleanup of the samples; then take corrective action to eliminate the problem.

4.3 The use of high purity reagents, solvents, and gases helps to minimize interference problems.

4.4 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between samples. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross contamination.

4.5 Phthalate contamination is commonly observed in this analysis and its occurrence should be carefully evaluated as an indicator of a contamination problem in the sample preparation step of the analysis.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

NOTE: Latex and vinyl gloves provide no protection against the organic solvents used in this method. Nitrile or similar gloves must be used.

5.1.2 The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.1.3 The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

5.1.4 There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power

before performing any maintenance.

5.1.5 The use of separatory funnels to extract aqueous samples with methylene chloride creates excessive pressure very rapidly. Initial venting should be done immediately after the sample container has been sealed and inverted. Vent the funnel into the hood away from people and other samples. This is considered a high-risk activity, and must be done either in a hood with the sash down to chest level or while wearing a face shield over safety glasses.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating.

NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.

A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Materials with Significant or Serious Hazard Rating

Material (1)	Hazards	Exposure Limit (2)	Signs and Symptoms of Exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.

Material (1)	Hazards	Exposure Limit (2)	Signs and Symptoms of Exposure
Sodium Hydroxide	Corrosive	2 mg/m3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/m3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
(1) Always add acid to water to prevent violent reactions. (2) Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Gas chromatograph/mass spectrometer system: an analytical system complete with a temperature-programmable gas chromatograph suitable for split/splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to the source.

6.2 Column: 30 m x 0.25 mm I.D., 0.5-µm film thickness fused-silica capillary column coated with 5% diphenyl/95% dimethyl polysiloxane(Restek Rtx®-5MS or equivalent). Alternate columns are acceptable if they provide acceptable performance.

6.3 Mass Spectrometer: Capable of scanning from 35 to 500 u (previously "amu") every one second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) that meets all of the criteria in Table 4 when 25 ng of the GC/MS tuning standard is injected through the GC.

6.4 Autosampler: LEAP Technologies CTC A200S, HP7683 Autosampler or equivalent.

6.5 GC/MS Interface: Any GC-to-MS interface that gives acceptable calibration points and achieves acceptable tuning performance criteria may be used.

6.6 Data System: A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as the Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIH Mass Spectral Library is recommended.

6.7 Syringe: 10 µL or 5µL Hamilton Laboratory grade syringes or equivalent. The 5 µL syringe is used for the Agilent ALS to be able to inject 0.5 µL.

6.8 Carrier gas: Ultra high-purity helium.

7.0 Reagents and Standards

7.1 A minimum five-point calibration curve is prepared when average response factors or linear regression curve fitting is used. Six calibration points are required for second-order curve fits. The low point should be at or below the reporting limit. Refer to tables 11 and 12 for typical calibration levels for all analytes. Other calibration levels may be used, depending on instrument capability, but the low standard must support the reporting limit and the high standard defines the range of the calibration.

7.2 An internal standard (IS) solution is prepared. Compounds in the IS Mix are acenaphthene-d10, chrysene-d12, 1,4-dichlorobenzene-d4, naphthalene-d8, perylene-d12, and phenanthrene-d10.

7.2.1 Internal standards are added to all standards and extracts to result in a final concentration of 40 µg/mL. For example, if the volume of an extract aliquot used was 200 µL, 20 µL of a 400 µg/mL internal standard solution would be added to the aliquot. See Appendix B for the levels used for the 8270 best practice method.\

7.3 Surrogate Standard Spiking Solution: Prepare as indicated in the extraction SOPs (refer to Section 2.4 for extraction SOPs numbers). Surrogate compounds and levels are listed in Table 9.

Acid Surrogates	Base Surrogates
2-Fluorophenol	2-Fluorobiphenyl
2,4,6-tribromophenol	Terphenyl-d4
Phenol-d5	Nitrobenzene-d5
2-chlorophenol-d4	1,2,-Dichlorobenzene-d4

7.4 GC/MS Tuning Standard: A methylene chloride solution containing 50 µg/mL of decafluorotriphenylphosphine (DFTPP) is prepared. Pentachlorophenol, benzidine, and DDT should also be included in the Tuning Standard at 50 µg/mL.

7.5 Laboratory Control Spiking Solution: Prepare as indicated in the extraction SOPs (refer to Section 2.4 for extraction SOPs numbers). LCS compounds and levels are listed in Table 7.

7.6 Matrix Spike Solution: Prepare as indicated in the extraction SOPs (refer to Section 2.4 for extraction SOPs numbers). The matrix spike compounds and levels are the same as the LCS compounds.

7.7 The standards listed in sections 7.1 to 7.6 must be refrigerated at -10°C to -20°C if it can be demonstrated that analytes do not fall out of solution at these temperatures. If not stable, the standards should be stored at 4 ± 2 °C. The standard stock solutions expire after one year from preparation date or at the earliest expiration date assigned by the vendor to any parent standard, whichever is earlier. The continuing calibration standard should be replaced every week, when there are visible signs of degradation, or when the standard fails to meet QC criteria. The continuing calibration standard is stored at -10°C to -20°C.

8.0 Sample Collection, Preservation, Shipment and Storage

Matrix	Sample Container	Min. Sample Size	Preservation	Extraction Holding Time	Analysis Holding Time	Reference
Waters	1 liter amber	1 Liter	Cool 4 ± 2°C	7 Days	40 Days from extraction	40 CFR Part 136.3
Soils	4oz Jar	30 grams	Cool 4 ± 2°C	14 Days	40 Days from extraction	N/A

9.0 Quality Control

9.1 Initial Performance Studies

9.1.1 Before analyzing samples, the laboratory must establish a method detection limit (MDL). See Section 13 for a discussion of detection limit studies.

9.1.2 In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on the instrument they will be using. On-going proficiency must be demonstrated by each analyst on an annual basis. See Section 13 for more details.

9.2 Control Limits

9.2.1 In-house historical control limits must be determined for surrogates, matrix spikes, and laboratory control samples (LCS). These limits are determined every 6 months. The recovery limits are the mean recovery ±3 standard deviations for surrogates, MS, and LCS. Precision limits for the MS/MSD pair results is the absolute value of the mean relative percent difference (RPD) +3 standard deviations.

9.2.2 These limits do not apply to dilutions, but surrogate and matrix spike recoveries will be reported unless the dilution is 4x or more.

9.2.3 All surrogate, LCS, and MS recoveries (except for dilutions) must be entered into the LIMS or other database so that accurate historical control limits can be generated. For multiple dilutions reported from the same extract, surrogates will be reported for all dilutions of less than 4x. For tests without a separate extraction, surrogates and matrix spikes will be reported for all dilutions.

9.2.4 Refer to the QC program document, DV-QA-003P, for further details of control limits.

9.3 Method Blank (MB)

For aqueous sample batches, the method blank is reagent water; for solid sample batches, the method blank is clean sand. In either case, the method blank is free of the analytes of interest and is spiked with the surrogates. At least one method blank must be

processed with each preparation batch.

Acceptance Criteria: The result for the method blank must be less than ½ of the reporting limit or less than 10% of the analyte concentration found in the associated samples, whichever is higher. When a compound is above ½ the reporting limit a NCM needs to be completed.

NOTE: All programs require that the maximum blank concentration must be less than one-half of the reporting limit or less than 10% of the lowest sample concentration.

Corrective Action: Re-preparation and reanalysis of all samples associated with an unacceptable method blank. If the analyte was not detected in the samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative.

9.4 Instrument Blank

Instruments must be evaluated for contamination during each 12-hour analytical run. This may be accomplished by analysis of a method blank. If a method blank is not available, an instrument blank must be analyzed. An instrument blank consists of methylene chloride with the internal standards added. It is evaluated in the same way as the method blank.

9.5 Laboratory Control Sample (LCS)

The LCS is prepared using reagent water for aqueous methods and Ottawa sand for solid sample methods. A laboratory control sample (LCS) is prepared and analyzed with every batch of samples. The LCS is spiked with the compounds listed in Tables 9 and 10 unless specified by a client or agency. The compounds must be spiked at a concentration equivalent to 100 or 150 µg/L, depending on the analyte, unless a special QAS states a specific level. Ongoing monitoring of the LCS provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

Acceptance Criteria: All analytes must be within established control limits. See QC Policy DV-QA-003P for details on establishing control limits.

Corrective Action: If any analyte in the LCS is outside the laboratory-established historical control limits or project-specific control limits, as applicable, corrective action must occur. Corrective action may include re-extraction and reanalysis of the batch.

- If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. An example of acceptable reasons for not reanalyzing might be that the matrix spike and matrix spike duplicate are acceptable, and sample surrogate recoveries are good, demonstrating that the problem was confined to the LCS. This type of justification

should be reviewed and documented with the client before reporting.

- If re-extraction and reanalysis of the batch are not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

The matrix spike is a second aliquot of one of the samples in the batch. The matrix spike duplicate is a third aliquot of the same sample. The MS and MSD are spiked with the same analytes as the LCS (See Tables 9 and 10). An MS/MSD pair is prepared and analyzed with every batch of samples.

Acceptance Criteria: The percent recovery (%R) must fall within either historical limits or project-specific limits, as applicable. The relative percent difference (RPD) between the MS and MSD results must be less than or equal to the established historical or project-specific limit. See QC Policy DV-QA-003P for details on establishing control limits

Corrective Action: If any individual recovery or RPD fails the acceptance criteria, then corrective action must occur. Initially check the recovery of the analyte in question in the LCS. Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is considered to be in control and analysis may proceed. The reasons for accepting the batch must be documented.

- If the recovery for any analyte fails acceptance criteria for the MS, MSD, and the LCS, the laboratory operation is considered to be out of out of control and corrective action must be taken. Corrective action will normally include re-preparation and reanalysis of the batch.
- If it is not possible to prepare both an MS and MSD due to limitations of sample amount, then a duplicate LCS should be prepared and analyzed. The RPD between the LCS and LCSD must be less than or equal to the RPD limit established for the MS/MSD.
- The MS/MSD pair must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted to concentrations below the calibration range.

9.7 Surrogates

9.7.1 Each sample, blank, and QC sample is spiked with the surrogate standards. Surrogate compounds must be spiked at either 100 or 150 ug/L, depending on the surrogate. The compounds routinely included in the surrogate spiking solution, along with recommended standard concentrations, are listed in Table 9. For the Best Practice method, see

table B-4 in Appendix B.

Acceptance Criteria: Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits.

Corrective Action: If any surrogates are outside of the limits, then the following corrective actions must take place (except for dilutions):

- Check all calculations for error.
- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze the extract if either of the above checks reveals a problem.
- Re-extract and reanalyze the sample or flag the data as "Estimated Concentration" if neither of the above resolves the problem.

NOTE: The decision to reanalyze or flag the data should be made in consultation with the client. It is only necessary to reprepare / reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out-of-control results are not due to matrix effect.

9.7.2 If the sample with failed surrogate recoveries was a sample used for an MS/MSD pair and the surrogate recoveries in the MS/MSD are also outside of the control limits, then the sample and the MS and the MSD do not require reanalysis. This phenomenon indicates a possible matrix problem.

9.7.3 If the sample is reanalyzed and the surrogate recoveries in the reanalysis are acceptable, then the problem was within the analyst's control and only the reanalyzed data should be reported. (Unless the reanalysis was outside holding times, in which case reporting both sets of results may be appropriate).

9.7.4 If the reanalysis does confirm the original results, the original analysis is reported and the data flagged as estimated due to matrix effects.

9.8 Nonconformance and Corrective Action

9.8.1 Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA department also receives NCMs by e-mail for tracking and trending purposes. The nonconformance shall be addressed in the case narrative, and the NCM shall be filed in the project file. The NCM process is described in more detail in SOP DV-QA-0031.

9.8.2 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the

project documents. and approved by a supervisor and QA Manager.

9.8.3 Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

9.9 Quality Assurance Summaries (QAS) or Program Distillations

Certain clients may require specific project or program QC that may supersede the requirements presented in this section. Quality Assurance Summaries (also known as Program Distillations) should be developed to address these requirements.

9.10 TAL QC Program

Details of the TAL Denver Quality Control Program, including corrective action guidelines, are presented in DV-QA-003P, Quality Control Program. Refer to this document if in doubt regarding corrective actions.

10.0 Procedure

10.1 Sample Preparation

Samples are prepared according to the following organic preparation SOPs, as applicable:

DV-OP-0006	Extraction of Aqueous Samples by Separatory Funnel, SW846 3510C and EPA 600 Series
DV-OP-0007	Concentration of Organic Extracts, SW846 3510C, 3520C, 3540C, 3550B, and EPA 600 Series
DV-OP-0008	Extraction of Aqueous Samples by Continuous Liquid/Liquid Extraction (CLLE) by Method SW-846 3520C and Methods 625 and 607
DV-OP-0009	Ultrasonic Extraction of Solid Samples, SW846 3550B

10.2 Sample Analysis Procedure

10.2.1 Calibrate the instrument as described in Section 10. Depending on the target compounds required by the client, it may be necessary to use more than one set of calibration standards.

10.2.2 All samples must be analyzed using the same instrument conditions as the preceding continuing calibration verification (CCV) standard.

10.2.3 Add internal standard to an aliquot of the extract to result in a 40-ng/µL concentration (for example, 20 µL of internal standard solution at, 400 µg/mL in 200 µL of extract). Mix thoroughly before injection into the instrument.

10.2.4 Inject the aliquot into the GC/MS system using the same injection technique as used for the standards.

10.2.5 The data system will determine the concentration of each analyte in the extract using calculations equivalent to those in Section 12. Quantitation is based on the initial calibration, not the continuing calibration verification.

10.2.6 Identified compounds are reviewed for proper integration. Manual integrations are performed if necessary and are documented by the analyst (see DV-QA-0033, Acceptable Manual Integration Practices) or

automatically by the data system. The minimum documentation required includes a hard copy of original data system peak integration and a similarly scaled hard copy showing the manual integration with analyst initials and date.

10.2.7 Target compounds identified by the data system are evaluated using the criteria listed in Section 12.1.

10.2.8 Library searches of peaks present in the chromatogram that are not target compounds, i.e., Tentatively Identified Compounds (TIC), may be performed if required by the client. They are evaluated using the criteria in Section 12.2.

10.3 Dilutions

If the response for any compound exceeds the working range of the GC/MS system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

10.3.1 Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and the baseline rise is less than the height of the internal standards, or if individual non-target peaks are significantly less than two times the height of the internal standards, the sample should be reanalyzed at a more concentrated dilution. This requirement is approximate and subject to analyst judgment. For example, samples containing organic acids may need to be analyzed at a higher dilution to avoid destroying the column.

10.3.2 Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will be reported only at client request.

10.4 Perform all qualitative and quantitative measurements. When the extracts are not being used for analyses, freeze them at <-10°C, protected from light in screw cap vials equipped with unpierced Teflon lined septa.

10.5 Retention Time Criteria for Samples

10.5.1 If the retention time for any internal standard changes by more than 0.5 minutes from the last continuing calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

10.5.2 If the retention time of any internal standard in any sample varies by more than 0.1 minute from the preceding continuing calibration standard, the data must be carefully evaluated to ensure that no analytes have shifted outside their retention time windows.

10.6 Percent Moisture

Analytical results may be reported as dry or wet weight, as required by the client. Percent moisture must be determined if results will be reported as dry weight. Refer to SOP DV-WC-0023 for determination of percent moisture.

10.7 Procedural Variations

One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA department also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described

10.8 Troubleshooting Guide

10.8.1 Daily Instrument Maintenance

In addition to the checks listed in Appendix D, the following daily maintenance should be performed.

- Clip Column as necessary.
- Install new or cleaned injection port liner as necessary.
- Install new septum as necessary.
- Install new or cleaned gold seal and washer as necessary.
- Perform mass calibration as necessary.

10.8.2 Major Maintenance

A new initial calibration is necessary following certain maintenance procedures. These maintenance procedures include changing the column, cleaning the repeller, cleaning the source, replacing the multiplier, and replacing the "top board" or RF-related electronics. Refer to the manufacturer's manual for specific guidance.

11.0 Calibration

11.1 Summary

The instrument is tuned for DFTPP, calibrated initially with a minimum of a five levels, and verified each 12-hour shift with one or more continuing calibration standard(s). Recommended instrument conditions are listed in Table 3.

11.2 All standards and extracts are allowed to warm to room temperature before injecting.

11.3 Instrument Tuning

At the beginning of every twelve-hour shift when analyses are to be performed, the GC/MS system must be checked to see if the acceptance criteria are achieved for DFTPP (decafluorotriphenylphosphine). See Table 4.

11.3.1 Inject 25 ng of the GC/MS tuning standard (Section 7.4) into the GC/MS system. Obtain a background-corrected mass spectra of DFTPP and confirm that all the key m/z criteria in Table 4 are achieved. If all the

criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are achieved. The performance criteria must be achieved before any samples, blanks, or standards are analyzed.

11.3.2 The GC/MS tuning standard should also be used to evaluate the inertness of the chromatographic system. The acceptance criteria for the peak tailing factor for benzidine is < 3.0 and pentachlorophenol is < 5.0. DDT breakdown must be <20%. Refer to section 12 for the appropriate calculations.

11.4 Initial Calibration

11.4.1 Internal Standard (IS) Calibration Procedure: Internal standards are listed in Table 5. Use the base peak m/z as the primary m/z for quantitation of the standards. If interferences are noted, use one of the next two most intense masses for quantitation.

11.4.2 Compounds are assigned to the IS with the closest retention time.

11.4.3 Prepare calibration standards at a minimum of five concentration levels for each parameter of interest when average response factors or linear regression curve fits are used. Six standards must be used for a quadratic least-squares calibration. It may also be useful to analyze six calibration levels and use the lower five for most analytes and the upper five for analytes that have poor response.

11.4.4 For AFCEE projects, the five calibration levels will be those shown in Table 10. The table also lists a sixth calibration level that is used if a second-order regression fit is needed. The only exceptions would be for the AFCEE projects requiring special reporting limits, i.e., reporting limits different than those in the AFCEE program QAPP. Additional calibration points may be required for special projects.

11.4.5 Rejection of Calibration Points

11.4.5.1 Generally, it is NOT acceptable to remove points from a calibration. If calibration acceptance criteria are not met, the normal corrective action is to examine conditions such as instrument maintenance and accuracy of calibration standards. Any problems must be fixed and documented in the run log or maintenance log. Then the calibration standard(s) must be reanalyzed.

11.4.5.2 If no problems are found or there is documented evidence of a problem with a calibration point (e.g., obvious misinjection explained in the run log), then one point might be rejected, but only if all of the following conditions are met:

- The rejected point is the highest or lowest on the curve, i.e., the remaining points used for calibration must be contiguous; and

- The lowest remaining calibration point is still at or below the project reporting limit; and

- The highest remaining calibration point defines the upper concentration of the working range, and all samples producing results above this concentration are diluted and reanalyzed; and

- The calibration must still have the minimum number of calibration levels required by the method, i.e. five levels for calibrations modeled with average response factors or linear regressions, or six levels for second-order curve fits.

11.4.6 Add the internal standard mixture to result in a 40-ng/µL final concentration. (For example, if the volume of the calibration standard used is 0.5 mL, add 50 µL of the 400 µg/mL internal standard). The concentrations of all analytes are listed in Tables 11 and 12. For the Best Practice method, see Table B-5 in Appendix B.

11.4.7 Analyze each calibration standard and tabulate the area of the primary characteristic m/z against the concentration for each compound and internal standard. Calculate the response factors (RF), average response factors, and the percent RSD of the response factors for each compound using the equations in section 12. Verify that the CCC and SPCC criteria, which are specified in Sections 11.4.8 and 11.4.9 are met. No sample analysis may be performed unless these criteria are met.

11.4.8 System Performance Check Compounds (SPCCs)

The minimum average RF for semivolatile SPCCs is 0.050. If the minimum response factors are not met, the system must be evaluated and corrective action must be taken before sample analysis begins. Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before analysis begins.

11.4.8.1 SPCC Compounds:

N-nitroso-di-n-propylamine
Hexachlorocyclopentadiene
2,4-Dinitrophenol
4-Nitrophenol

11.4.9 Calibration Check Compounds (CCCs)

The %RSD of the response factors for each CCC in the initial calibration must be less than 30% for the initial calibration to be considered valid. This criterion must be met before sample analysis begins. Problems similar to those listed under SPCCs could affect this check.

11.4.9.1 If none of the CCCs are required analytes, then project-specific calibration specifications (which may include the use of the CCCs listed in Section 11.4.9.2) must be implemented with concurrence from the client.

11.4.9.2 CCC Compounds:

Phenol
Acenaphthene
1,4-Dichlorobenzene
N-nitrosodiphenylamine
2-Nitrophenol
Pentachlorophenol
2,4-Dichlorophenol
Fluoranthene
Hexachlorobutadiene
Di-n-octylphthalate
4-Chloro-3-methylphenol
Benzo(a)pyrene
2,4,6-Trichlorophenol

11.4.10 If the average of all RSDs in the initial calibration is < 15%, then all analytes may use average response factor for calibration.

11.4.10.1 If the software in use is capable of routinely reporting curve coefficients for data validation purposes, and the necessary calibration reports can be generated, then the analyst should evaluate analytes with RSD > 15% for calibration on a curve. If it appears that substantially better accuracy would be obtained using quantitation from a curve fit, then the appropriate curve should be used for quantitation.

11.4.10.2 If the average of all the RSDs in the initial calibration is > 15%, then calibration using a curve fit, must be used for those analytes with RSD > 15%. Linear or quadratic curve fits may be used. Use of 1/Concentration2 weighting is recommended to improve the accuracy of quantitation at the low end of the curve. The analyst should consider instrument maintenance to improve the linearity of response.

11.4.10.3 If a linear regression equation is used, the correlation coefficient r must be greater than 0.990, and r square (r2) greater than 0.9801 Use of second-order regression equations may be used on rare occasions. In these cases, the intercept and degree of curvature should be examined to be sure that results will be reliable throughout the working range, and the coefficient of determination must be greater than 0.990.

Note: South Carolina can only be analyzed using linear calibration.

11.4.10.4 An initial calibration verification containing all components from a second source (an alternate vendor, or, a unique lot from the same vendor, or, the same source but prepared by an alternate analyst) must be analyzed after the initial calibration. Acceptance criteria for ICV percent recovery (%R) are 75-125% for DoD projects (e.g., AFCEE); 65-135% for non-DoD projects (e.g., 625/8270C HSL

components); and 45-155% for poor performers (e.g., 8270C AP9, Custom, Refinery, DBP, benzaldehyde).

11.4.11 Weighting of Calibration Data Points

In a linear or quadratic calibration fit, the points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason, it is preferable to increase the weighting of the lower concentration points. 1/Concentration2 weighting (often called 1/X2 weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability. Because the data system does not indicate the type of weighting used, the analyst must make a notation on the initial calibration form as to the weighting used (e.g. 1/x or $1/x^2$).

11.4.12 If time remains in the 12-hour period initiated by the DFTPP injection before the initial calibration, samples may be analyzed. Otherwise, proceed to continuing calibration, Section 11.5.

NOTE: Quantitation is performed using the calibration curve or average response factor from the initial curve, not the continuing calibration.

11.5 Continuing Calibration Verification (CCV)

11.5.1 At the start of each 12-hour period, the GC/MS tuning standard must be analyzed. A 25-ng injection of DFTPP must result in a mass spectrum for DFTPP, which meets the criteria given in Table 4.

11.5.2 Following a successful DFTPP analysis, the continuing calibration verification (CCV) standard(s) are analyzed. The standard(s) must contain all semivolatile analytes, including all required surrogates. A mid level calibration standard is used for the CCV.

11.5.3 The following criteria must be met for the CCV to be acceptable:

- The SPCC compounds must have a response factor ≥ 0.050.
- The percent difference or drift (%D) of the CCC compounds must be ≤ 20%. (See Section 12 for calculations.)
- For compounds of interest, reliably performing compounds (see Table 14, List 1 Reliably Performing Compounds) should have a %D ≤ 35%. Poorly performing compounds (see Table 15, List 2 Poorly Performing Compounds) should have a %D ≤ 50%, with allowance for up to 6 target analytes to have %D values greater than the applicable limit. Any compound of interest that does not meet the applicable criteria will be narrated.
- The internal standard response of the CCV must be within 50 - 200% of the response in the same level of the corresponding calibration.
- If any internal standard retention time in the CCV changes by more than 30 seconds from that of the same level of the corresponding initial calibration, the chromatographic system must be inspected for malfunctions and corrections made, as required.

11.5.3.1 If none of the CCCs are required analytes, project-specific calibration requirements (which may include the use of the CCCs listed in Section 11.4.9.2) must be implemented with concurrence from the client.

11.5.4 Once the above criteria have been met, sample analysis may begin. Initial calibration average RFs (or the calibration curve) will be used for sample quantitation, not the continuing calibration RFs. Analysis may proceed until 12 hours from the injection of the DFTPP have passed. (A sample injected less than or equal to 12 hours after the DFTPP is acceptable.)

12.0 Calculations / Data Reduction

12.1 Qualitative Identification

An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NBS library. Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC retention time as the standard component; and (2) correspondence of the sample component and the standard component characteristic ions.

NOTE: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.

12.1.1 The sample component relative retention time must compare to within ± 0.06 RRT units of the relative retention time of the standard component. For reference, the standard must be run within the same twelve hours as the sample.

12.1.2 All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) should be present in the sample spectrum.

12.1.3 The characteristic ions of a compound must maximize in the same scan or within one scan of each other.

12.1.4 The relative intensities of ions should agree to within ±30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20% and 80%.)

12.1.5 If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst the identification is correct, the analyst shall report that identification and proceed with quantitation.

12.2 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the type of analyses being

conducted. Computer generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample spectra with the nearest library searches shall the mass spectral interpretation specialist assign a tentative identification. Following are guidelines for making tentative identification:

12.2.1 Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.

12.2.2 The relative intensities of the major ions should agree to within ±20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance should be between 30%and 70%.)

12.2.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

12.2.4 Ions present in the sample spectrum, but not in the reference spectrum, should be reviewed for possible background contamination or the presence of co-eluting compounds.

12.2.5 Ions present in the reference spectrum, but not in the sample spectrum, should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

12.2.6 Automatic background subtraction can severely distort spectra from samples with unresolved hydrocarbons.

12.3 Isomers with identical mass spectra and close elution times pose problems for definitive identification. The following compounds fall into this category:

Aniline and bis(2-chloroethyl) ether
Dichlorobenzenes
Methylphenols
Trichlorophenols
Phenanthrene, anthracene
Fluoranthene, pyrene
Benzo(b) and (k)fluoranthene
Chrysene, benzo(a)anthracene

Identification of these compounds requires both experience and extra precautions on the part of the analyst. Specifically, the analyst must more closely scrutinize the comparison of retention times between the unknown and the calibration standard. The analyst must also check that all isomers have distinct retention times.

12.4 A second category of problem compounds consist of the poor responders or compounds that chromatograph poorly. The integrations for these types of compounds should be checked manually. The following compounds are included in this category:

Benzoic acid
Chloroanilines
Nitroanilines
2,4-Dinitrophenol
4-Nitrophenol
Pentachlorophenol
3,3'-Dichlorobenzidine
Benzyl alcohol
4,6-Dinitro-2-methylphenol
Ariazine
Famphur
Benzidine

12.5 Calculating the Percent Relative Standard Deviation for Initial Calibration

$$\%RSD = \frac{SD}{RF} \times 100\%$$

Where:

RF = Mean of RFs from the initial calibration for a compound
SD = Standard deviation for the mean RF from the initial calibration for a compound

$$SD = \sqrt{\frac{\sum_{i=1}^{n}\left(RF_i - \overline{RF}\right)^2}{n-1}}$$

RFi = RF for each of the calibration levels
n = Number of RF values

12.6 Calculating the Continuing Calibration Percent Drift

$$\%Drift = \frac{C_{actual} - C_{found}}{C_{actual}} \times 100\%$$

Where:

Cactual = Known concentration in standard
Cfound = Measured concentration using selected quantitation method

12.7 Calculating the Concentration in the Extract

The concentration of each identified analyte and surrogate in the extract is calculated from the linear or quadratic curve fitted to the initial calibration points, or from the average RF of the initial calibration.

12.7.1 Average Response Factor Calibration

If the average of all the RSDs of the response factors in the initial calibration is ≤15%, the average response factor from the initial calibration may be used for quantitation.

$$C_{ex} = \frac{R_x C_{is}}{R_{is} \overline{RF}}$$

Where:

Cex	=	Concentration in the extract, µg/mL
Rx	=	Response for the analyte
Ris	=	Response for the internal standard
Cis	=	Concentration of the internal standard
$\overline{RF}$	=	Average response factor

12.7.2 Linear Fit Calibration

$$C_{ex} = A + B\frac{(R_x C_{is})}{R_{is}}$$

Where:

Cex	=	Concentration in the extract, µg/mL
Rx	=	Response for the analyte
Ris	=	Response for the internal standard
Cis	=	Concentration of the internal standard
A	=	Intercept of linear calibration line
B	=	Slope of linear calibration line

12.7.3 Quadratic Fit Calibration

$$C_{ex} = A + B\left(\frac{R_x C_{is}}{R_{is}}\right) + C\left(\frac{R_x C_{is}}{R_{is}}\right)$$

Where:

Cex	=	Concentration in the extract, µg/mL
Rx	=	Response for the analyte
Ris	=	Response for the internal standard
Cis	=	Concentration of the internal standard
A	=	Intercept
B	=	Factor for the linear term of the quadratic calibration function
C	=	Factor for the curvature term of the quadratic calibration function

12.8 Calculating the Concentration in the Sample

12.8.1 Calculation for Aqueous Samples

$$\text{Concentration, } \mu g / L = \frac{C_{ex} V_t}{V_o}$$

Where:

Cex = Concentration in the extract

Vt = Volume of total extract in µL, taking into account dilutions (i.e., a 1-to-10 dilution of a 1-mL extract will mean that Vt = 10,000 µL. If half of the base/neutral extract and half of the acid extract are combined, then Vt = 2,000.)

Vo = Volume of the sample that was extracted (mL)

12.8.2 Calculation for Sediment, Soil, Sludge, and Waste Samples

Results for sediments, sludges, and soils are usually calculated on a dry-weight basis, and for waste, on a wet-weight basis.

$$\text{Concentration, } \mu g / kg = \frac{C_{ex} V_t}{W_s D}$$

Where:

Cex = Concentration in the extract

Vt = Volume of total extract in µL, taking into account dilutions (i.e., a 1-to-10 dilution of a 1-mL extract will mean that Vt = 10,000 µL. If half of the base/neutral extract and half of the acid extract are combined, then Vt = 2,000.)

Ws = Weight of sample extracted or diluted in grams

D = (100 - % moisture in sample)/100, for a dry-weight basis or 1 for a wet-weight basis

12.9 MS/MSD Percent Recovery Calculation

$$\text{Matrix Spike Recovery} = \frac{S_{SR} - S_R}{S_A} \times 100\%$$

Where:

SSR = Spike sample result

SR = Sample result

SA = Spike added

12.10 Calculating the Relative Percent Difference (RPD) MS/MSD Pair

$$RPD = \frac{MS_R - MSD_R}{1/2(MS_R + MSD_R)} \times 100$$

Where:

RPD	=	Relative percent difference
MSR	=	Matrix spike result
MSDR	=	Matrix spike duplicate result

12.11 Relative Response Factor Calculation

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:

Ax = Area of the characteristic ion for the compound being measured
Ais = Area of the characteristic ion for the specific internal standard
Cx = Concentration of the compound being measured (µg/L)
Cis = Concentration of the specific internal standard (µg/L)

12.12 Calculation of TICs

The calculation of TICs (tentatively identified compounds) is identical to the above calculation (12.11) with the following exceptions:

Ax = Area of the total ion chromatogram for the compound being measured
Ais = Area of the total ion chromatogram for the nearest internal standard without interference
RF = 1

12.13 Calculating Percent DDT Breakdown

$$\%\,\text{DDT breakdown} = \frac{\text{DDEarea} + \text{DDDarea}}{\text{DDTarea} + \text{DDEarea} + \text{DDDarea}}$$

The areas for the 235 ion are used for this calculation.

12.14 Calculating the Peak Tailing Factor

$$TailingFactor = \frac{BC}{AB}$$

Where:

Peak width (AC) is measured at 10% peak height, and divided into two line segments at the peak centroid, so that .

AC = AB + BC, with
AB = left-hand segment
BC = right-hand segment

13.0 Method Performance

13.1 Method Detection Limit Study (MDL)

An initial MDL study must be performed on each instrument before samples can be analyzed. MDL studies are conducted annually as follows

13.1.1 Prepare seven replicates at three to five times the estimated MDL concentration.

13.1.2 Extract and analyze the MDL standards as described in Section 10.

13.1.3 Calculate the mean concentration found (X) in µg/L, and the standard deviation of the mean concentration in µg/L, for each analyte. Then calculate the MDL (single-tailed, 99% confidence level, as described in Policy DV-QA-005P) for each analyte.

13.1.4 MDL studies are repeated annually, and MDL results are stored in the laboratory LIMS system. See Policy DV-QA-005P for further details concerning MDL studies.

13.1.5 The current MDL value is maintained in the TestAmerica Denver LIMS.

13.2 MDL Verification (MDLV)

Calculated MDLs from the annual studies are subject to quarterly verification by analyzing an MDLV standard prepared at 1-2 times the calculated MDL concentration. An MDLV standard is analyzed immediately after each MDL study and quarterly thereafter. This standard is subject to the entire preparation and analysis process.

Acceptance Criteria:	The calculated MDL is verified if the MDLV standard is detected, nominally signal to noise ratio ≥ 3, under routine instrument conditions.
Corrective Actions:	If the first MDLV is not detected, the MDLV standard will be reprepared and analyzed at twice the original concentration. The lowest concentration that produces a detectable signal will then be reported as the MDL.

13.3 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows:

13.3.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid-level calibration.

13.3.2 Calculate the mean recovery and standard deviation for each analyte of interest.

13.3.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.3.4 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

13.4 Training Requirements

13.4.1 Training Qualification

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience. Further details concerning the training program are described in SOP DV-QA-0024.

13.4.2 Non-standard Analytes

For non-standard analytes, an MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration should include the analysis of an extracted standard at the reporting limit and a single point calibration.

14.0 Pollution Control

14.1 Standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

15.0 Waste Management

15.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Corporate Safety Manual, and HS-001, "Waste Management Program."

15.2 The following waste streams are produced when this method is carried out

15.2.1 Expired Chemicals/Reagents/Standards – Contact Waste Coordinator

15.2.2 Methylene Chloride- B

15.2.3 Flammable Solvent- Waste Stream C

15.2.4 Used vials- Waste Stream A

NOTE: Radioactive, mixed waste, and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

16.0 References / Cross-References

16.1 SW846, Test Methods for Evaluating Solid Waste, Third Edition, Update III, December 1996, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique, Method 8270C.

16.2 40CFR, part 136, Appendix A, "Base/Neutrals and Acids", Method 625.

17.0 Method Modifications:

17.1 Modifications from Reference Method

17.1.1 A retention time window of 0.2 minutes is used for all components, since some data systems do not have the capability of using the relative retention time units specified in the reference method.

17.1.2 The quantitation and qualifier ions for some compounds have been changed from those recommended in SW-846 in order to improve the reliability of qualitative identification.

17.1.3 This procedure includes the option for weighted linear regression curves using 1/concentration2 weighting factors. Section 7.5.2 of Method 8000B discusses the use of weighted least square regression based on 1/standard deviation2 weighting factors, which would require multiple analyses of each standard to determine the standard deviation. IAETL has presented information to the EPA Office of Solid Waste demonstrating that the variance (standard deviation2) is proportional to the standard concentration. EPA accepted this argument and issued a letter in July 1998, which authorizes the use of 1/concentration2 weighting factors.

18.0 Attachments

Table 1. TAL Primary Standard and Standard Reporting Limits
Table 2. TAL Appendix IX Standard Reporting Limits
Table 3. Suggested Instrument Conditions
Table 4. DFTPP Key Ions and Ion Abundance Criteria
Table 5. Characteristic Ions, Primary Standard (in approximate retention time order)
Table 6. Characteristic Ions, Appendix IX Standard (in approximate retention time order)
Table 7. 8270C LCS Compounds
Table 8. TCLP LCS Compounds
Table 9. 8270C Surrogate Compounds
Table 10. Calibration Levels for AFCEE Projects, µg/mL
Table 11. Calibration Levels, Primary Standard, µg/mL
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Table 13. Initial Demonstration Recovery and Precision Limits
Table 14. List 1 Reliably Performing Compounds
Table 15. List 2 Poorly Performing Compounds
APPENDIX A. Modifications Required for Analysis of Wastewater Following Method 625
Table A-1. TAL Method 625 Standard Reporting List and Reporting Limits
Table A-2. Method 625 LCS and MS Compounds and Spike Concentrations
APPENDIX B. Modifications Required for Analysis of Wastewater Following Method 8270 Best Practice (8270BP)
Table B-1. TAL Method 8270BP Standard Reporting Limits
Table B-2. Method 8270BP Calibration Levels
Table B-3. Method 8270BP LCS Spike Concentrations

Table B-4. 8270BP Surrogate Compounds

Table B-5. Suggested Instrument Conditions for 8270BP
APPENDIX C. Instrument Maintenance Schedules - Mass Spectrometer & Gas Chromatograph

19.0 Revision History

- Revision 5, dated 20 March 2008\

    - Integration for TestAmerica and STL operations.
    - Revised Tables 1 and 2 to reflect current reporting limits.
    - Removed the use of average average from the calibration section 11.4.10.

Changes fromPrevious Major Revision

- Removed the modifications for 1,4-dioxane by isotope dilution, and included this compound in Appendix B, 8270 Best Practice.

Table 1.

TAL Primary Standard and Standard Reporting Limits

Analytes	CAS Number	Standard Reporting Limits	
		Aqueous (µg/L)	Low Soil/Sediment (µg/kg)
Pyridine	110-86-1	20	660
N-nitrosodimethylamine	62-75-9	10	330
Aniline	62-53-3	10	330
Phenol	108-95-2	10	330
Bis(2-chloroethyl)ether	111-44-4	10	330
2-Chlorophenol	95-57-8	10	330
1,3-Dichlorobenzene	541-73-1	4	330
1,4-Dichlorobenzene	106-46-7	4	330
Benzyl alcohol	100-51-6	10	330
1,2-Dichlorobenzene	95-50-1	4	330
2-Methylphenol	95-48-7	10	330
2,2'-oxybis(1-chloropropane)2	108-60-1	10	330
4-Methylphenol	106-44-5	10	330
N-Nitroso-di-n-propylamine	621-64-7	10	330
Hexachloroethane	67-72-1	10	330
Nitrobenzene	98-95-3	10	330
Isophorone	78-59-1	10	330
2-Nitrophenol	88-75-5	10	330
2,4-Dimethylphenol	105-67-9	10	330
Benzoic acid	65-85-0	50	1600
Bis(2-chloroethoxy)methane	111-91-1	10	330
2,4-Dichlorophenol	120-83-2	10	330
1,2,4-Trichlorobenzene	120-82-1	10	330
Naphthalene	91-20-3	10	330
4-Chloroaniline	106-47-8	10	330
Hexachlorobutadiene	87-68-3	10	330
4-Chloro-3-methylphenol	59-50-7	10	330
2-Methylnaphthalene	91-57-6	10	330
Hexachlorocyclopentadiene	77-47-4	50	1600
2,4,6-Trichlorophenol	88-06-2	10	330
2,4,5-Trichlorophenol	95-95-4	10	330
2-Chloronaphthalene	91-58-7	4	330
2-Nitroaniline	88-74-4	10	1600
Dimethyl phthalate	131-11-3	4	330
Acenaphthylene	208-96-8	4	330
3-Nitroaniline	99-09-2	10	1600
Acenaphthene	83-32-9	4	330
2,4-Dinitrophenol	51-28-5	30	1600
4-Nitrophenol	100-02-7	10	1600
Dibenzofuran	132-64-9	4	330
2,4-Dinitrotoluene	121-14-2	10	330

Table 1.

TAL Primary Standard and Standard Reporting Limits (cont.)

Analytes	CAS Number	Standard Reporting Limits	
		Aqueous (µg/L)	Low Soil/Sediment (µg/kg)
2,6-Dinitrotoluene	606-20-2	10	330
Diethylphthalate	84-66-2	4	330
4-Chlorophenyl phenyl ether	7005-72-3	10	330
Fluorene	86-73-7	4	330
4-Nitroaniline	100-01-6	10	1600
4,6-Dinitro-2-methylphenol	534-52-1	20	1600
N-Nitrosodiphenylamine	86-30-6	10	330
Azobenzene	103-33-3	10	330
4-Bromophenyl phenyl ether	101-55-3	10	330
Hexachlorobenzene	118-74-1	10	330
Pentachlorophenol	87-86-5	50	1600
Phenanthrene	85-01-8	4	330
Anthracene	120-12-7	4	330
Carbazole	86-74-8	4	330
Di-n-butyl phthalate	84-74-2	4	330
Fluoranthene	206-44-0	4	330
Benzidine	92-87-5	100	3300
Pyrene	129-00-0	10	330
Butyl benzyl phthalate	85-68-7	4	330
3,3'-Dichlorobenzidine	91-94-1	50	1600
Benzo(a)anthracene	56-55-3	4	330
Bis(2-ethylhexyl)phthalate	117-81-7	10	330
4,4-Methylenebis(2-chloroaniline)	101-14-4	100	330
Chrysene	218-01-9	4	330
Di-n-octylphthalate	117-84-0	4	330
Benzo(b)fluoranthene	205-99-2	4	330
Benzo(k)fluoranthene	207-08-9	4	330
Benzo(a)pyrene	50-32-8	4	330
Indeno(1,2,3-cd)pyrene	193-39-5	4	330
Diethyl phthalate	84-66-2	4	660
Dibenz(a,h)anthracene	53-70-3	4	330
Benzo(g,h,i)perylene	191-24-2	4	330
Acetophenone	98-86-2	10	330
3/4-Methylphenol	108-39-4	10	330
1,4-Dioxane	54841-74-6	20	660

1. The TAL primary standard is the standard normally used at TAL. Additional standards, such as the Appendix IX standard may be necessary to include all target analytes required for some clients.
2. 2,2'oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether.

Table 2.

TAL Appendix IX Standard Reporting Limits

Semivolatiles	CAS Number	Standard Reporting Limits	
		Aqueous (µg/L)	Low Soil/Sediment (µg/kg)
2-Picoline	109-06-8	20	660
N-Nitrosomethylethylamine	10595-95-6	10	330
Methyl methanesulfonate	66-27-3	10	330
N-Nitrosodiethylamine	55-18-5	10	330
Ethyl methanesulfonate	62-50-0	10	330
Pentachloroethane	76-01-7	50	1600
N-Nitrosopyrrolidine	930-55-2	10	330
N-Nitrosomorpholine	59-89-2	10	330
o-Toluidine	95-53-4	10	660
N-Nitrosopiperidine	100-75-4	10	330
o,o,o-Triethyl-Phosphorothioate	126-68-1	50	1600
a,a-Dimethyl-phenethylamine	122-09-8	50	1600
2,6-Dichlorophenol	87-65-0	10	330
Hexachloropropene	1888-71-7	100	3300
p-Phenylenediamine	106-50-3	100	1600
n-Nitrosodi-n-butylamine	924-16-3	10	330
Safrole	94-59-7	50	1600
1,2,4,5-Tetrachlorobenzene	95-94-3	10	330
Isosafrole	120-58-1	20	660
1,4-Dinitrobenzene	100-25-4	10	330
1,4-Naphthoquinone	130-15-4	50	1600
1,3-Dinitrobenzene	99-65-0	10	330
Pentachlorobenzene	608-93-5	10	330
1-Naphthylamine	134-32-7	10	330
2-Naphthylamine	91-59-8	10	330
2,3,4,6-Tetrachlorophenol	58-90-2	50	1600
5-Nitro-o-toluidine	99-55-8	20	660
Thionazin	297-97-2	10	1600
1,3,5-Trinitrobenzene	99-35-4	50	1600
Sulfotepp	3689-24-5	50	1000
Phorate	298-02-2	50	1600
Phenacetin	62-44-2	20	660
Diallate	2303-16-4	20	660
Dimethoate	60-51-5	20	660
4-Aminobiphenyl	92-67-1	50	1600
Pentachloronitrobenzene	82-68-8	50	1600
Pronamide	23950-58-5	20	660
Disulfoton	298-04-4	50	1600
2-secbutyl-4,6-dinitrophenol (Dinoseb)	88-85-7	10	660
Methyl Parathion	298-00-0	50	1600
1-chloronaphthalene	90-13-1	10	330
Biphenyl	92-51-3	10	330

Table 2.

TAL Appendix IX Standard Reporting Limits (cont.)

Semivolatiles	CAS Number	Standard Reporting Limits	
		Aqueous (µg/L)	Low Soil/Sediment (µg/kg)
4-Nitroquinoline-1-oxide	56-57-5	100	3300
Parathion	56-38-2	50	1600
Methapyrilene	91-80-5	50	1600
Aramite	140-57-8	20	660
Isodrin	465-73-6	10	330
p-(Dimethylamino)azobenzene	60-11-7	20	660
p-Chlorobenzilate	510-15-6	10	330
3,3'-Dimethylbenzidine	119-93-7	20	660
2-Acetylaminofluorene	53-96-3	100	3300
Dibenz(a,j)acridine	224-42-0	10	660
7,12-Dimethylbenz(a)anthracene	57-97-6	20	660
3-Methylcholanthrene	56-49-5	20	660
Diphenylamine	122-39-4	10	330

1. The Appendix IX standard contains additional analytes required for the Appendix IX list. The TAL primary standard must also be analyzed to include all of the Appendix IX list.
2. May also be analyzed by method 8141, which can achieve lower reporting limits.
3. May also be analyzed by method 8080 or 8081, which can achieve lower reporting limits.

Table 3.

Suggested Instrument Conditions

Mass Range:	35 - 500 amu
Scan Time:	≤ 1 second/scan
Initial Column Temperature/Hold Time:	40 °C for 1 minute
Column Temperature Program:	40 - 325 °C at 25 °C/min.
Final Column Temperature/Hold Time:	325 °C (until at least one minute after benzo(g,h,i)perylene has eluted)
Injector Temperature:	250 °C
Transfer Line Temperature:	290 °C
Source Temperature:	According to manufacturer's specifications
Injector:	Grob-type, split / splitless
Sample Volume:	0.5 µl
Carrier Gas:	Helium at 3.4 mL/min.

Table 4.

DFTPP Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
51	30 - 60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40 - 60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5 - 9% of mass 198
275	10 - 30% of mass 198
365	>1% of mass 198
441	Present, but less than mass 443
442	40 - 100% of mass 198
443	17 - 23% of mass 442

Table 5.

Characteristic Ions, Primary Standard (in approximate retention time order)

Analyte	Primary	Secondary	Tertiary
N-nitrosodimethylamine	74	42	
1,4-Dioxane	88	58	
Pyridine	79	52	
2-Fluorophenol (Surrogate Standard)	112	64	63
Phenol-d5 (Surrogate Standard)	99	42	71
Aniline	93	66	
Phenol	94	65	66
Bis(2-chloroethyl)ether	93	63	95
2-Chlorophenol	128	64	130
1,3-Dichlorobenzene	146	148	113
1,4-Dichlorobenzene-d4 (Internal Standard)	152	150	115
1,4-Dichlorobenzene	146	148	113
Benzyl Alcohol	108	79	77
1,2-Dichlorobenzene	146	148	113
2-Methylphenol	108	107	77
2,2'-oxybis(1-chloropropane)[1]	45	77	79
4-Methylphenol	108	107	79
N-Nitroso-di-n-propylamine	70	42	101,130
Hexachloroethane	117	201	199
Nitrobenzene-d5 (Surrogate Standard)	82	128	54
Nitrobenzene	77	123	65
Isophorone	82	95	138
2-Nitrophenol	139	65	109
2,4-Dimethylphenol	107	121	122
Benzoic Acid	122	105	77
Bis(2-chloroethoxy)methane	93	95	123
2,4-Dichlorophenol	162	164	98
1,2,4-Trichlorobenzene	180	182	145
Naphthalene-d8 (Internal Standard)	136	68	54
Naphthalene	128	129	127
4-Chloroaniline	127	129	65
Hexachlorobutadiene	225	223	227
4-Chloro-3-methylphenol	107	144	142
2-Methylnaphthalene	142	141	115
Hexachlorocyclopentadiene	237	235	271
2,4,6-Trichlorophenol	196	198	200
2,4,5-Trichlorophenol	196	198	200
2-Fluorobiphenyl (Surrogate Standard)	172	171	170
2-Chloronaphthalene	162	164	127
2-Nitroaniline	65	92	138
Dimethylphthalate	163	194	164
Acenaphthylene	152	151	153

Table 5.

Characteristic Ions, Primary Standard (in approximate retention time order) (cont.)

Analyte	Primary	Secondary	Tertiary
2,6-Dinitrotoluene	165	63	89
Acenaphthene-d10 (Internal Standard)	164	162	160
3-Nitroaniline	138	108	92
Acenaphthene	153	152	154
2,4-Dinitrophenol	184	63	154
Dibenzofuran	168	139	84
4-Nitrophenol	109	139	65
2,4-Dinitrotoluene	165	63	89
Diethylphthalate	149	177	150
Fluorene	166	165	167
4-Chlorophenylphenylether	204	206	141
4-Nitroaniline	138	92	108
4,6-Dinitro-2-methylphenol	198	105	51
N-Nitrosodiphenylamine	169	168	167
2,4,6-Tribromophenol (Surrogate Standard)	330	332	141
Azobenzene	77	182	105
4-Bromophenylphenylether	248	250	141
Hexachlorobenzene	284	142	249
Pentachlorophenol	266	264	268
Phenanthrene-d10 (Internal Standard)	188	94	80
Phenanthrene	178	179	176
Anthracene	178	179	176
Carbazole	167	166	139
Di-n-butylphthalate	149	150	104
Fluoranthene	202	101	100
Benzidine	184	92	185
Pyrene	202	101	100
Terphenyl-d14 (Surrogate Standard)	244	122	212
Butylbenzylphthalate	149	91	206
Famphur	218	93	125
Benzo(a)Anthracene	228	229	226
Chrysene-d12 (Internal Standard)	240	120	236
3,3'-Dichlorobenzidine	252	254	126
4,4-Methylenebis(2-Chloroanaline)	231	266	-
Chrysene	228	226	229
Bis(2-ethylhexyl)phthalate	149	167	279
Di-n-octylphthalate	149	167	43
Benzo(b)fluoranthene	252	253	125
Benzo(k)fluoranthene	252	253	125
Benzo(a)pyrene	252	253	125
Perylene-d12 (Internal Standard)	264	260	265
Indeno(1,2,3-cd)pyrene	276	138	277
Dibenz(a,h)anthracene	278	139	279
Benzo(g,h,i)perylene	276	138	277

Table 6.

Characteristic Ions, Appendix IX Standard (in approximate retention time order)

Analyte	Primary	Secondary	Tertiary
2-Picoline	93	66	92
N-Nitrosomethylethylamine	88	42	43
Methyl methanesulfonate	80	79	65
N-Nitrosodiethylamine	102	44	57
Ethyl methanesulfonate	79	109	97
Pentachloroethane	117	119	167
Acetophenone	105	77	120
N-Nitrosopyrrolidine	100	41	42
N-Nitrosomorpholine	116	56	86
o-Toluidine	106	107	77
3/4-Methylphenol	108	107	77
N-Nitrosopiperidine	114	42	55
o,o,o-Triethyl-Phosphorothioate	198	121	93
a,a-Dimethyl-phenethylamine	58	91	
2,6-Dichlorophenol	162	164	63
Hexachloropropene	213	215	211
p-Phenylenediamine	108	80	54
n-Nitrosodi-n-butylamine	84	57	41
Safrole	162	104	77
1,2,4,5-Tetrachlorobenzene	216	214	218
Isosafrole 1	162	104	131
Isosafrole 2	162	104	131
1,4-Dinitrobenzene	168	75	122
1,4-Naphthoquinone	158	104	102
1,3-Dinitrobenzene	168	50	76
Pentachlorobenzene	250	248	252
1-Naphthylamine	143	115	
2-Naphthylamine	143	115	
2,3,4,6-Tetrachlorophenol	232	230	131
5-Nitro-o-toluidine	152	77	106
Thionazin	97	96	143
1,3,5-Trinitrobenzene	213	75	120
Sulfotepp	97	322	202
Phorate	75	97	121
Phenacetin	108	179	109
Diallate	86	234	
Dimethoate	87	93	125
4-Aminobiphenyl	169	168	115
Pentachloronitrobenzene	237	142	214
Pronamide	173	175	255
Disulfoton	88	97	89
2-secbutyl-4,6-dinitrophenol (Dinoseb)	211	163	147
Methyl parathion	109	125	263
4-Nitroquinoline-1-oxide	190	128	160

Table 6.

Characteristic Ions, Appendix IX Standard (in approximate retention time order) (cont.)

Analyte	Primary	Secondary	Tertiary
Parathion	109	97	291
Isodrin	193	66	195
Famphur	218	125	93
Methapyrilene	97	58	
Aramite 1	185	319	
Aramite 2	185	319	
p-(Dimethylamino)azobenzene	120	225	77
p-Chlorobenzilate	251	139	253
3,3'-Dimethylbenzidine	212	106	
2-Acetylaminofluorene	181	180	223
Dibenz(a,j)acridine	279	280	
7,12-Dimethylbenz(a)anthracene	256	241	120
3-Methylcholanthrene	268	252	253

Table 7.

8270C LCS Compounds

LCS Compounds	Spiking Level, ng/µL in extract
1,2,4-Trichlorobenzene	100
Acenaphthene	100
2,4-Dinitrotoluene	100
Pyrene	100
N-Nitroso-di-n-propylamine	100
1,4-Dichlorobenzene	100
Pentachlorophenol	150
Phenol	150
2-Chlorophenol	150
4-Chloro-3-methylphenol	150
4-Nitrophenol	150

Table 8.

TCLP LCS Compounds

LCS Compounds	Spiking Level, ng/µL in extract
1,4-Dichlorobenzene	50
2,4-Dinitrotoluene	50
Hexachlorobenzene	50
Hexachlorobutadiene	50
Hexachloroethane	50
2-Methylphenol	50
3/4-Methylphenol	100
Nitrobenzene	50
Pentachlorophenol	100
Pyridine	50
2,4,5-Trichlorophenol	50
2,4,6-Trichlorophenol	50

Recovery limits for the LCS and for matrix spikes are generated from historical data and are maintained by the QA group.

Table 9.

8270C Surrogate Compounds

Surrogate Compounds	Spiking Level, ng/µL in extract
Nitrobenzene-d5	100
2-Fluorobiphenyl	100
Terphenyl-d14	100
1,2-Dichlorobenzene-d4[1]	100
Phenol-d5	150
2-Fluorophenol	150
2,4,6-Tribromophenol	150
2-Chlorophenol-d4[1]	150

1. Included in standard mix, but not routinely evaluated for method 8270C

Recovery limits for surrogates are generated from historical data and are maintained by the QA department.

Table 10.

Calibration Levels for AFCEE Projects, µg/mL

Analyte	Std Conc 1	Std Conc 2	Std Conc 3	Std Conc 4	Std Conc 5	Std Conc 6	Addnl Conc for 2nd Order ICALs
Pyridine	20	50	80	120	200	---	160
N-nitrosodimethylamine	10	20	50	80	120	200	---
Aniline	10	20	50	80	120	200	---
Phenol	10	20	50	80	120	200	---
Bis(2-chloroethyl)ether	10	20	50	80	120	200	---
2-Chlorophenol	10	20	50	80	120	200	---
1,3-Dichlorobenzene	10	20	50	80	120	200	---
1,4-Dichlorobenzene	10	20	50	80	120	200	---
Benzyl alcohol	10	20	50	80	120	200	---
1,2-Dichlorobenzene	10	20	50	80	120	200	---
2-Methylphenol	10	20	50	80	120	200	---
2,2'-oxybis(1-chloropropane)[1]	10	20	50	80	120	200	---
4-Methylphenol	10	20	50	80	120	200	---
N-Nitroso-di-n-propylamine	10	20	50	80	120	200	---
Hexachloroethane	10	20	50	80	120	200	---
Nitrobenzene	10	20	50	80	120	200	---
Isophorone	10	20	50	80	120	200	---
2-Nitrophenol	10	20	50	80	120	200	---
2,4-Dimethylphenol	10	20	50	80	120	200	---
Benzoic acid	50	50	80	120	200	---	160
Bis(2-chloroethoxy)methane	10	20	50	80	120	200	---
2,4-Dichlorophenol	10	20	50	80	120	200	---
1,2,4-Trichlorobenzene	10	20	50	80	120	200	---
Naphthalene	10	20	50	80	120	200	---
4-Chloroaniline	10	20	50	80	120	200	---
Hexachlorobutadiene	20	20	50	80	120	200	---
4-Chloro-3-methylphenol	10	20	50	80	120	200	---
2-Methylnaphthalene	10	20	50	80	120	200	---
Hexachlorocyclopentadiene	20	50	80	120	200	---	160
2,4,6-Trichlorophenol	10	20	50	80	120	200	---
2,4,5-Trichlorophenol	10	20	50	80	120	200	---
2-Chloronaphthalene	10	20	50	80	120	200	---
2-Nitroaniline	20	50	80	120	200	---	160
Dimethyl phthalate	10	20	50	80	120	200	---
Acenaphthylene	10	20	50	80	120	200	---
3-Nitroaniline	20	50	80	120	200	---	160
Acenaphthene	10	20	50	80	120	200	---
2,4-Dinitrophenol	20	50	80	120	200	---	160
4-Nitrophenol	20	50	80	120	200	---	160
Dibenzofuran	10	20	50	80	120	200	---
2,4-Dinitrotoluene	10	20	50	80	120	200	---
2,6-Dinitrotoluene	10	20	50	80	120	200	---
Diethylphthalate	10	20	50	80	120	200	---

Table 10.

Calibration Levels for AFCEE Projects, µg/mL (cont.)

Analyte	Std Conc 1	Std Conc 2	Std Conc 3	Std Conc 4	Std Conc 5	Std Conc 6	Addnl Conc for 2nd Order ICALs
4-Chlorophenyl phenyl ether	10	20	50	80	120	200	---
Fluorene	10	20	50	80	200	200	---
4-Nitroaniline	20	50	80	120	200	200	---
4,6-Dinitro-2-methylphenol	20	50	80	120	200	200	---
N-Nitrosodiphenylamine	10	20	50	80	200	200	---
Azobenzene[2]	10	20	50	80	200	200	---
4-Bromophenyl phenyl ether	10	20	50	80	200	200	---
Hexachlorobenzene	10	20	50	80	200	200	---
Pentachlorophenol	20	50	80	120	200	200	---
Phenanthrene	10	20	50	80	200	200	---
Anthracene	10	20	50	80	200	200	---
Carbazole	10	20	50	80	200	200	---
Di-n-butyl phthalate	10	20	50	80	200	200	---
Fluoranthene	10	20	50	80	200	200	---
Benzidine	50	50	80	120	200	200	---
Pyrene	10	20	50	80	200	200	---
Butyl benzyl phthalate	10	20	50	80	200	200	---
3,3'-Dichlorobenzidine	10	50	80	120	200	200	---
Benzo(a)anthracene	10	20	50	80	200	200	---
Bis(2-ethylhexyl)phthalate	10	20	50	80	200	200	---
Chrysene	10	20	50	80	200	200	---
Di-n-octylphthalate	10	20	50	80	200	200	---
Benzo(b)fluoranthene	10	20	50	80	200	200	---
Benzo(k)fluoranthene	10	20	50	80	200	200	---
Benzo(a)pyrene	10	20	50	80	200	200	---
Indeno(1,2,3-cd)pyrene	10	20	50	80	200	200	---
Dibenz(a,h)anthracene	10	20	50	80	200	200	---
Diethyl phthalate	10	20	50	80	200	200	---
Benzo(g,h,i)perylene	10	20	50	80	200	200	---

1. 2,2'oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether.
2. Azobenzene is formed by decomposition of 1,2-diphenlyhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

Table 11.

Calibration Levels, Primary Standard, µg/mL

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Pyridine	--	20	50	80	120	160	200
N-nitrosodimethylamine	10	20	50	80	120	160	200
Aniline	10	20	50	80	120	160	200
Phenol	10	20	50	80	120	160	200
Bis(2-chloroethyl)ether	10	20	50	80	120	160	200
2-Chlorophenol	10	20	50	80	120	160	200
1,3-Dichlorobenzene	10	20	50	80	120	160	200
1,4-Dichlorobenzene	10	20	50	80	120	160	200
Benzyl alcohol	10	20	50	80	120	160	200
1,2-Dichlorobenzene	10	20	50	80	120	160	200
2-Methylphenol	10	20	50	80	120	160	200
2,2'-oxybis(1-chloropropane)[1]	10	20	50	80	120	160	200
4-Methylphenol	10	20	50	80	120	160	200
N-Nitroso-di-n-propylamine	10	20	50	80	120	160	200
Hexachloroethane	10	20	50	80	120	160	200
Nitrobenzene	10	20	50	80	120	160	200
Isophorone	10	20	50	80	120	160	200
2-Nitrophenol	10	20	50	80	120	160	200
2,4-Dimethylphenol	10	20	50	80	120	160	200
Benzoic acid	--	20	50	80	120	160	200
Bis(2-chloroethoxy)methane	10	20	50	80	120	160	200
2,4-Dichlorophenol	10	20	50	80	120	160	200
1,2,4-Trichlorobenzene	10	20	50	80	120	160	200
Naphthalene	10	20	50	80	120	160	200
4-Chloroaniline	10	20	50	80	120	160	200
Hexachlorobutadiene	10	20	50	80	120	160	200
4-Chloro-3-methylphenol	10	20	50	80	120	160	200
2-Methylnaphthalene	10	20	50	80	120	160	200
Hexachlorocyclopentadiene	--	20	50	80	120	160	200
2,4,6-Trichlorophenol	10	20	50	80	120	160	200
2,4,5-Trichlorophenol	10	20	50	80	120	160	200
2-Chloronaphthalene	10	20	50	80	120	160	200
2-Nitroaniline	--	20	50	80	120	160	200
Dimethyl phthalate	10	20	50	80	120	160	200
Acenaphthylene	10	20	50	80	120	160	200
3-Nitroaniline	--	20	50	80	120	160	200
Acenaphthene	10	20	50	80	120	160	200
2,4-Dinitrophenol	--	20	50	80	120	160	200
4-Nitrophenol	--	20	50	80	120	160	200
Dibenzofuran	10	20	50	80	120	160	200
2,4-Dinitrotoluene	10	20	50	80	120	160	200
2,6-Dinitrotoluene	10	20	50	80	120	160	200
Diethylphthalate	10	20	50	80	120	160	200
4-Chlorophenyl phenyl ether	10	20	50	80	120	160	200

Table 11.

Calibration Levels, Primary Standard, µg/mL (cont.)

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Fluorene	10	20	50	80	120	160	200
4-Nitroaniline	--	20	50	80	120	160	200
4,6-Dinitro-2-methylphenol	--	20	50	80	120	160	200
N-Nitrosodiphenylamine	10	20	50	80	120	160	200
Azobenzene[2]	10	20	50	80	120	160	200
4-Bromophenyl phenyl ether	10	20	50	80	120	160	200
Hexachlorobenzene	10	20	50	80	120	160	200
Pentachlorophenol	--	20	50	80	120	160	200
Phenanthrene	10	20	50	80	120	160	200
Anthracene	10	20	50	80	120	160	200
Carbazole	10	20	50	80	120	160	200
Di-n-butyl phthalate	10	20	50	80	120	160	200
Fluoranthene	10	20	50	80	120	160	200
Benzidine	--	20	50	80	120	160	200
Pyrene	10	20	50	80	120	160	200
Butyl benzyl phthalate	10	20	50	80	120	160	200
3,3'-Dichlorobenzidine	--	20	50	80	120	160	200
Benzo(a)anthracene	10	20	50	80	120	160	200
Bis(2-ethylhexyl)phthalate	10	20	50	80	120	160	200
Chrysene	10	20	50	80	120	160	200
4,4-Methylenebis(2-chloroaniline)	10	20	50	80	120	160	200
Di-n-octylphthalate	10	20	50	80	120	160	200
Benzo(b)fluoranthene	10	20	50	80	120	160	200
Benzo(k)fluoranthene	10	20	50	80	120	160	200
Benzo(a)pyrene	10	20	50	80	120	160	200
Indeno(1,2,3-cd)pyrene	10	20	50	80	120	160	200
Dibenz(a,h)anthracene	10	20	50	80	120	160	200
Diethyl phthalate	10	20	50	80	120	160	200
Benzo(g,h,i)perylene	10	20	50	80	120	160	200

1. 2,2'oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether
2. Azobenzene is formed by decomposition of 1,2-diphenlyhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

Table 11.

Calibration Levels, Primary Standard, µg/mL (cont.)

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Fluorene	10	20	50	80	120	160	200
4-Nitroaniline	--	20	50	80	120	160	200
4,6-Dinitro-2-methylphenol	--	20	50	80	120	160	200
N-Nitrosodiphenylamine	10	20	50	80	120	160	200
Azobenzene[2]	10	20	50	80	120	160	200
4-Bromophenyl phenyl ether	10	20	50	80	120	160	200
Hexachlorobenzene	10	20	50	80	120	160	200
Pentachlorophenol	--	20	50	80	120	160	200
Phenanthrene	10	20	50	80	120	160	200
Anthracene	10	20	50	80	120	160	200
Carbazole	10	20	50	80	120	160	200
Di-n-butyl phthalate	10	20	50	80	120	160	200
Fluoranthene	10	20	50	80	120	160	200
Benzidine	--	20	50	80	120	160	200
Pyrene	10	20	50	80	120	160	200
Butyl benzyl phthalate	10	20	50	80	120	160	200
3,3'-Dichlorobenzidine	--	20	50	80	120	160	200
Benzo(a)anthracene	10	20	50	80	120	160	200
Bis(2-ethylhexyl)phthalate	10	20	50	80	120	160	200
Chrysene	10	20	50	80	120	160	200
4,4-Methylenebis(2-chloroaniline	10	20	50	80	120	160	200
Di-n-octylphthalate	10	20	50	80	120	160	200
Benzo(b)fluoranthene	10	20	50	80	120	160	200
Benzo(k)fluoranthene	10	20	50	80	120	160	200
Benzo(a)pyrene	10	20	50	80	120	160	200
Indeno(1,2,3-cd)pyrene	10	20	50	80	120	160	200
Dibenz(a,h)anthracene	10	20	50	80	120	160	200
Diethyl phthalate	10	20	50	80	120	160	200
Benzo(g,h,i)perylene	10	20	50	80	120	160	200

1. 2,2'oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether
2. Azobenzene is formed by decomposition of 1,2-diphenlyhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

Table 12.
Calibration Levels, Appendix IX Standard, µg/mL

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
2-Picoline	10	20	50	80	120	160	200
N-Nitrosomethylethylamine	10	20	50	80	120	160	200
Methyl methanesulfonate	10	20	50	80	120	160	200
N-Nitrosodiethylamine	10	20	50	80	120	160	200
Ethyl methanesulfonate	10	20	50	80	120	160	200
Pentachloroethane	--	20	50	80	120	160	200
Acetophenone	10	20	50	80	120	160	200
N-Nitrosopyrrolidine	10	20	50	80	120	160	200
N-Nitrosomorpholine	10	20	50	80	120	160	200
o-Toluidine	10	20	50	80	120	160	200
3-Methylphenol	10	20	50	80	120	160	200
N-Nitrosopiperidine	10	20	50	80	120	160	200
o,o,o-Triethyl-Phosphorothioate	--	20	50	80	120	160	200
a,a-Dimethyl-phenethylamine	--	20	50	80	120	160	200
2,6-Dichlorophenol	10	20	50	80	120	160	200
Hexachloropropene	--	20	50	80	120	160	200
p-Phenylenediamine	--	20	50	80	120	160	200
n-Nitrosodi-n-butylamine	10	20	50	80	120	160	200
Safrole	--	20	50	80	120	160	200
1,2,4,5-Tetrachlorobenzene	10	20	50	80	120	160	200
Isosafrole 1 + 2	10	20	50	80	120	160	200
1,4-Dinitrobenzene	10	20	50	80	120	160	200
1,4-Naphthoquinone	--	20	50	80	120	160	200
1,3-Dinitrobenzene	10	20	50	80	120	160	200
Pentachlorobenzene	10	20	50	80	120	160	200
1-Naphthylamine	10	20	50	80	120	160	200
2-Naphthylamine	10	20	50	80	120	160	200
2,3,4,6-Tetrachlorophenol	--	20	50	80	120	160	200
5-Nitro-o-toluidine	10	20	50	80	120	160	200
Thionazin	10	20	50	80	120	160	200
1,3,5-Trinitrobenzene	--	20	50	80	120	160	200
Sulfotepp	--	20	50	80	120	160	200
Phorate	--	20	50	80	120	160	200
Phenacetin	10	20	50	80	120	160	200
Diallate 1 + 2	10	20	50	80	120	160	200
Dimethoate	10	20	50	80	120	160	200
4-Aminobiphenyl	--	20	50	80	120	160	200
Pentachloronitrobenzene	--	20	50	80	120	160	200
Pronamide	10	20	50	80	120	160	200
Disulfoton	--	20	50	80	120	160	200
2-secbutyl-4,6-dinitrophenol (Dinoseb)	10	20	50	80	120	160	200
Methyl parathion	--	20	50	80	120	160	200
4-Nitroquinoline-1-oxide	--	20	50	80	120	160	200
Parathion	--	20	50	80	120	160	200

Table 12.

Calibration Levels, Appendix IX Standard, µg/mL (cont.)

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Isodrin	10	20	50	80	120	160	200
Methapyrilene	--	20	50	80	120	160	200
Aramite 1 and 2	10	20	50	80	120	160	200
p-(Dimethylamino) azobenzene	10	20	50	80	120	160	200
p-Chlorobenzilate	10	20	50	80	120	160	200
3,3'-Dimethylbenzidine	10	20	50	80	120	160	200
2-Acetylaminofluorene	--	20	50	80	120	160	200
Dibenz (a,j)acridine	10	20	50	80	120	160	200
7,12-Dimethylbenz(a) anthracene	10	20	50	80	120	160	200
3-Methylcholanthrene	10	20	50	80	120	160	200

Table 13.

Initial Demonstration Recovery and Precision Limits

Compound	Spiking Concentration, µg/L	Limit for Relative Standard Deviation	Limit for Average Recovery, %
Acenaphthene	60	27.6	60.1-132.3
Acenaphthylene	60	40.2	53.5-126.0
Aldrin[1]	60	39.0	7.2-152.2
Anthracene	60	32.0	43.4-118.0
Benz(a)anthracene	60	27.6	41.8-133.0
Benzo(b)fluoranthene	60	38.8	42.0-140.4
Benzo(k)fluoranthene	60	32.3	25.2-145.7
Benzo(a)pyrene	60	39.0	31.7-148.0
Benzo(ghi)perylene	60	58.9	D-195.0
Benzylbutyl phthalate	60	23.4	D-139.9
B-BHC[1]	60	31.5	41.5-130.6
d-BHC[1]	60	21.6	D-100.0
Bis(2-chloroethyl) ether	60	55.0	42.9-126.0
Bis(2-chloroethoxy)methane	60	34.5	49.2-164.7
Bis(2-chloroisopropyl) ether	60	46.3	62.8-138.6
Bis(2-ethylhexyl) phthalate	60	41.1	28.9-136.8
4-Bromophenyl phenyl ether	60	23.0	64.9-114.4
2-Chloronaphthalene	60	13.0	64.5-113.5
4-Chlorophenyl phenyl ether	60	33.4	38.4-144.7
Chrysene	60	48.3	44.1-139.9
4,4'-DDD[1]	60	31.0	D-134.5
4,4'-DDE[1]	60	32.0	19.2-119.7
4,4'-DDT[1]	60	61.6	D-170.6
Dibenzo(a,h)anthracene	60	70.0	D-199.7

Table 13.

Initial Demonstration Recovery and Precision Limits (cont.)

Compound	Spiking Concentration, µg/L	Limit for Relative Standard Deviation	Limit for Average Recovery, %
Di-n-butyl phthalate	60	16.7	8.4-111.0
1,2-Dichlorobenzene	60	30.9	48.6-112.0
1,3-Dichlorobenzene	60	41.7	16.7-153.9
1,4-Dichlorobenzene	60	32.1	37.3-105.7
3,3'-Dichlorobenzidine	60	71.4	8.2-212.5
Dieldrin[1]	60	30.7	44.3-119.3
Diethyl phthalate	60	26.5	D-100.0
Dimethyl phthalate	60	23.2	D-100.0
2,4-Dinitrotoluene	60	21.8	47.5-126.9
2,6-Dinitrotoluene	60	29.6	68.1-136.7
Di-n-octylphthalate	60	31.4	18.6-131.8
Endosulfan sulfate[1]	60	16.7	D-103.5
Endrin aldehyde	60	32.5	D-188.8
Fluoranthene	60	32.8	42.9-121.3
Fluorene	60	20.7	71.6-108.4
Heptachlor[1]	60	37.2	D-172.2
Heptachlor epoxide[1]	60	54.7	70.9-109.4
Hexachlorobenzene	60	24.9	7.8-141.5
Hexachlorobutadiene	60	26.3	37.8-102.2
Hexachloroethane	60	24.5	55.2-100.0
Indeno(1,2,3-cd)pyrene	60	44.6	D-150.9
Isophorone	60	63.3	46.6-180.2
Naphthalene	60	30.1	35.6-119.6
Nitrobenzene	60	39.3	54.3-157.6
N-Nitrosodi-n-propylamine	60	55.4	13.6-197.9
PCB-1260[1]	60	54.2	19.3-121.0
Phenanthrene	60	20.6	65.2-108.7
Pyrene	60	25.2	69.6-100.0
1,2,4-Trichlorobenzene	60	28.1	57.3-129.2
4-Chloro-3-methylphenol	60	37.2	40.8-127.9
2-Chlorophenol	60	28.7	36.2-120.4
2,4-Chlorophenol	60	26.4	52.5-121.7
2,4-Dimethylphenol	60	26.1	41.8-109.0
2,4-Dinitrophenol	60	49.8	D-172.9
2-Methyl-4,6-dinitrophenol	60	93.2	53.0-100.0
2-Nitrophenol	60	35.2	45.0-166.7
4-Nitrophenol	60	47.2	13.0-106.5
Pentachlorophenol	60	48.9	38.1-151.8
Phenol	60	22.6	16.6-100.0
2,4,6-Trichlorophenol	60	31.7	52.4-129.2

1. Organochlorine pesticides and PCBs project DQOs generally require better sensitivity than is provided by 8270C, so methods 8081 and 8082 are used instead. These compounds will not be included in the initial demonstration of capability for method 8270C.

Table 14.

List 1 Reliably Performing Compounds

Acenaphthene	Dibenzofuran	1H-Indene
Acenaphthylene	1,4-Dioxane	Indeno(1,2,3-cd)pyrene
Acetophenone	n-Dodecane	Isophorone
Alachlor	n-Docosane	1-Methylnaphthalene
Aniline	1,2-Dichlorobenzene	2-Methylnaphthalene
Anthracene	1,3-Dichlorobenzene	2-Methylphenol
Atrazine	1,4-Dichlorobenzene	4-Methylphenol
Benzo(a)anthracene	2,3-Dichlorobenzeneamine	Methylstyrene
Benzo(a)pyrene	3,3'-Dichlorobenzidine	Naphthalene
Benzo(b)fluoranthene	2,4-Dichlorophenol	2-Nitroaniline
Benzo(k)fluoranthene	Diethyl phthalate	3-Nitroaniline
Benzo(g,h,i)perylene	2,4-Dimethylphenol	4-Nitroaniline
Benzoic acid	Dimethyl phthalate	Nitrobenzene
Benzyl alcohol	Di-n-butyl phthalate	2-Nitrophenol
Bis(2-chloroethoxy)methane	4,6-Dinitro-2-methylphenol	4-Nitrophenol
Bis(2-chloroethyl)ether	2,4-Dinitrophenol	N-Nitrosodimethylamine
Bis(2-ethylhexyl)phthalate	2,4-Dinitrotoluene	N-Nitroso-di-n-propylamine
4-Bromophenyl phenyl ether	2,6-Dinitrotoluene	N-Nitrosodiphenylamine
Butyl benzyl phthalate	1,2-Diphenylhydrazine (as Azobenzene)	2,2'-Oxybis(1-chloropropane) aka "bis(2-chloroisopropyl) ether"
Caprolactam	Di-n-octyl phthalate	n-Octadecane
Carbazole	n-Eicosane	Pentachlorophenol
4-Chloroaniline	Famphur	Phenanthrene
4-Chloro-3-methylphenol	Fluoranthene	Phenol
2-Chloronaphthalene	Fluorene	Pyrene
2-Chlorophenol	Hexachlorobenzene	Pyridine
4-Chlorophenyl phenyl ether	Hexachlorocyclopentadiene	n-Tetradecane
Chrysene	Hexachlorobutadiene	1,2,4-Trichlorobenzene
n-Decane	Hexachloroethane	2,4,5-Trichlorophenol
Dibenz(a,h)anthracene	n-Hexadecane	2,4,6-Trichlorophenol

Table 15.

List 2 Poorly Performing Compounds

2-Acetylaminofluorene	Diphenylamine	N-Nitrosopyrrolidine
Acrylamide	Disulfoton	Parathion
4-Aminobiphenyl	2-Ethoxyethanol	Pentachlorobenzene
Aramite (#1)	Ethyl methanesulfonate	Pentachloroethane
Aramite (#2)	Hexachlorophene	Pentachloronitrobenzene
Benzenethiol	Hexachloropropene	Perylene
Benzidine	Isosafrole (#1)	Phenacetin
Benzyl chloride	Isosafrole (#2)	p-Phenylenediamine
Biphenyl	Isodrin	Phorate
Carbofuran phenol	Methapyrilene	Phthalic anhydride
Chlorobenzilate	Methomyl	2-Picoline
Diallate (#1)	3-Methylcholanthrene	Pronamide
Diallate (#2)	6-Methylchrysene	Quinoline
Dibenz(a,h)acridine	4,4"-Methylenebis(2-chloroaniline)	Safrole
Dibenz(a,j)acridine	Methyl methanesulfonate	2-secbutyl-4,6-dinitrophenol (Dinoseb)
Dibenzo(a,e)pyrene	Methyl Parathion	Sulfotepp
Tris(2,3-Dibromopropyl) phosphate	1-Naphthylamine	1,2,4,5-Tetrachlorobenzene
2,6-Dichlorophenol	2-Naphthylamine	2,3,4,6-Tetrachlorophenol
Dimethoate	1,4-Naphthoquinone	Thionazin
p-(Dimethylamino)azobenzene	5-Nitro-o-toluidine	o-Toluidine
7,12-Dimethylbenz(a)anthracene	4-Nitroquinoline-1-oxide	2,4- and 2,6-Toluenediamine
3,3'-Dimethylbenzidine	N-Nitrosodiethylamine	Triethylamine
N,N-Dimtheylformamide	n-Nitrosodi-n-butylamine	Triethylphosphate
a,a-Dimethyl-phenethylamine	N-Nitrosomethylethylamine	o,o,o-Triethylphosphorothioate
1,3-Dinitrobenzene	N-Nitrosomorpholine	1,3,5-Trinitrobenzene
1,4-Dinitrobenzene	N-Nitrosopiperidine	

APPENDIX A

Modifications Required for Analysis of Wastewater Following Method 625

REQUIREMENTS FOR METHOD 625

- Method 625 is required for demonstration of compliance with NPDES wastewater discharge permits or other CWA compliance situations. The standard analyte list and reporting limits are listed in Table A-1.
- This method can be applied to only aqueous matrices.
- The tune period for this method is defined as 24 hours.
- Initial calibration curve requirements are as follows:
  - The initial calibration curve for this method requires at least three points.
  - Target compounds must have RSD ≤ 35%.
  - If this requirement cannot be met, a regression curve must be constructed for the non-compliant compounds.
- Continuing calibration verification requirements are as follows:
  - All target compounds must have %D ≤ 20%.
- Matrix Spike and LCS requirements are as follows:
- A full analyte spike is required for method 625. The spiking levels are given in Table A-2.

Table A-1.

TAL Method 625 Standard Reporting List and Reporting Limits

Analytes	CAS Number	Aqueous, µg/L
Phenol	108-95-2	10
Bis(2-chloroethyl)ether	111-44-4	10
2-Chlorophenol	95-57-8	10
1,3-Dichlorobenzene	541-73-1	10
1,4-Dichlorobenzene	106-46-7	10
1,2-Dichlorobenzene	95-50-1	10
2,2'-oxybis(1-chloropropane)	108-60-1	10
N-Nitroso-di-n-propylamine	621-64-7	10
Hexachloroethane	67-72-1	10
Nitrobenzene	98-95-3	10
Isophorone	78-59-1	10
2-Nitrophenol	88-75-5	10
2,4-Dimethylphenol	105-67-9	10
Bis(2-chloroethoxy)methane	111-91-1	10
2,4-Dichlorophenol	120-83-2	10
1,2,4-Trichlorobenzene	120-82-1	10
Naphthalene	91-20-3	10
Hexachlorobutadiene	87-68-3	10
4-Chloro-3-methylphenol	59-50-7	10
Hexachlorocyclopentadiene	77-47-4	20
2,4,6-Trichlorophenol	88-06-2	10
2-Chloronaphthalene	91-58-7	10
Dimethyl phthalate	131-11-3	10
Acenaphthylene	208-96-8	10
Acenaphthene	83-32-9	10
2,4-Dinitrophenol	51-28-5	50
4-Nitrophenol	100-02-7	50
2,4-Dinitrotoluene	121-14-2	10
2,6-Dinitrotoluene	606-20-2	10
Diethylphthalate	84-66-2	10
4-Chlorophenyl phenyl ether	7005-72-3	10
Fluorene	86-73-7	10
4,6-Dinitro-2-methylphenol	534-52-1	50
N-Nitrosodiphenylamine	86-30-6	10
4-Bromophenyl phenyl ether	101-55-3	10
Hexachlorobenzene	118-74-1	10
Pentachlorophenol	87-86-5	50

Table A-1.

TAL Method 625 Standard Reporting List and Reporting Limits (cont.)

Analytes	CAS Number	Aqueous, µg/L
Phenanthrene	85-01-8	10
Anthracene	120-12-7	10
Di-n-butyl phthalate	84-74-2	10
Fluoranthene	206-44-0	10
Benzidine	92-87-5	100
Pyrene	129-00-0	10
Butyl benzyl phthalate	85-68-7	10
3,3'-Dichlorobenzidine	91-94-1	50
Benzo(a)anthracene	56-55-3	10
Bis(2-ethylhexyl)phthalate	117-81-7	10
Chrysene	218-01-9	10
Di-n-octylphthalate	117-84-0	10
Benzo(b)fluoranthene	205-99-2	10
Benzo(k)fluoranthene	207-08-9	10
Benzo(a)pyrene	50-32-8	10
Indeno(1,2,3-cd)pyrene	193-39-5	10
Dibenz(a,h)anthracene	53-70-3	10
Benzo(g,h,i)perylene	191-24-2	10
N-Nitrosodimethylamine	62-75-9	10

Table A-2.

Method 625 LCS and MS Compounds and Spike Concentrations

LCS Compounds	Spiking Level, ng/µL in extract[1]
Phenol	100
Bis(2-chloroethyl)ether	100
2-Chlorophenol	100
1,3-Dichlorobenzene	100
1,4-Dichlorobenzene	100
1,2-Dichlorobenzene	100
2,2'-oxybis(1-chloropropane)	100
N-Nitroso-di-n-propylamine	100
Hexachloroethane	100
Nitrobenzene	100
Isophorone	100
2-Nitrophenol	100
2,4-Dimethylphenol	100
Bis(2-chloroethoxy)methane	100
2,4-Dichlorophenol	100
1,2,4-Trichlorobenzene	100
Naphthalene	100
Hexachlorobutadiene	100
4-Chloro-3-methylphenol	100
Hexachlorocyclopentadiene	100
2,4,6-Trichlorophenol	100
2-Chloronaphthalene	100
Dimethyl phthalate	100
Acenaphthylene	100
Acenaphthene	100
2,4-Dinitrophenol	100
4-Nitrophenol	100
2,4-Dinitrotoluene	100
2,6-Dinitrotoluene	100
Diethylphthalate	100
4-Chlorophenyl phenyl ether	100
Fluorene	100
4,6-Dinitro-2-methylphenol	100
N-Nitrosodiphenylamine	100
4-Bromophenyl phenyl ether	100
Hexachlorobenzene	100
Pentachlorophenol	100

Table A-2.

Method 625 LCS and MS Compounds and Spike Concentrations (cont.)

LCS Compounds	Spiking Level, ng/µL in extract[1]
Phenanthrene	100
Anthracene	100
Di-n-butyl phthalate	100
Fluoranthene	100
Benzidine	100
Pyrene	100
Butyl benzyl phthalate	100
3,3'-Dichlorobenzidine	100
Benzo(a)anthracene	100
Bis(2-ethylhexyl)phthalate	100
Chrysene	100
Di-n-octylphthalate	100
Benzo(b)fluoranthene	100
Benzo(k)fluoranthene	100
Benzo(a)pyrene	100
Indeno(1,2,3-cd)pyrene	100
Dibenz(a,h)anthracene	100
Benzo(g,h,i)perylene	100
N-Nitrosodimethylamine	100

APPENDIX B

Modifications Required for Analysis of Wastewater Following Method 8270 Best Practice (8270BP)

REQUIREMENTS FOR METHOD 8270 BEST PRACTICE (8270BP)

- Method Best Practice is utilized to obtain lower reporting limits while still providing full scan data.. The standard analyte list and reporting limits are listed in Table B-1.
- This method can be applied to only aqueous matrices.
- The extraction is the same with one exception. The final volume of the extract is 2 mL.
- The tune period for this method is defined as 12 hours.
- Initial calibration curve requirements are as follows:
  - Same as for 8270 detailed in Section 11.4 of this SOP.
  - The calibrations levels are shown in Table B-2.
- Continuing calibration verification requirements are as follows:
  - Same as for 8270 detailed in Section 11.5 of this SOP, except the level 7 calibration point is used.
- Matrix Spike and LCS requirements are as follows:
  - The spike levels are listed in Table B-3.
- Internal Standards: The internal standard concentrations are listed in Table B-4.
- Surrogates: The surrogate concentrations are listed in Table B-5.
- Instrument Conditions are shown in Table B-6.

Table B-1.

TAL Method 8270BP Standard Reporting Limits

Analytes	CAS Number	Aqueous, µg/L
Pyridine	110-86-1	20
N-nitrosodimethylamine	62-75-9	5
Aniline	62-53-3	5
Phenol	108-95-2	10
Bis(2-chloroethyl)ether	111-44-4	1
2-Chlorophenol	95-57-8	5
Benzyl alcohol	100-51-6	5
2-Methylphenol	95-48-7	5
2,2'-oxybis(1-chloropropane)[2]	108-60-1	5
4-Methylphenol	106-44-5	5
N-Nitroso-di-n-propylamine	621-64-7	5
Hexachloroethane	67-72-1	5
Nitrobenzene	98-95-3	5
Isophorone	78-59-1	5
2-Nitrophenol	88-75-5	5
Benzoic acid	65-85-0	10
Bis(2-chloroethoxy)methane	111-91-1	5
2,4-Dichlorophenol	120-83-2	5
1,2,4-Trichlorobenzene	120-82-1	5
Naphthalene	91-20-3	5
4-Chloroaniline	106-47-8	5
Hexachlorobutadiene	87-68-3	5
4-Chloro-3-methylphenol	59-50-7	5
2-Methylnaphthalene	91-57-6	5
Hexachlorocyclopentadiene	77-47-4	5
2,4,6-Trichlorophenol	88-06-2	5
2,4,5-Trichlorophenol	95-95-4	5
2-Chloronaphthalene	91-58-7	5
2-Nitroaniline	88-74-4	5
Dimethyl phthalate	131-11-3	5
Acenaphthylene	208-96-8	5
3-Nitroaniline	99-09-2	5
Acenaphthene	83-32-9	5
2,4-Dinitrophenol	51-28-5	5
4-Nitrophenol	100-02-7	5
Dibenzofuran	132-64-9	5
2,4-Dinitrotoluene	121-14-2	5
2,6-Dinitrotoluene	606-20-2	5
4-Chlorophenyl phenyl ether	7005-72-3	5
Fluorene	86-73-7	5
4-Nitroaniline	100-01-6	5
4,6-Dinitro-2-methylphenol	534-52-1	10
N-Nitrosodiphenylamine	86-30-6	5
Azobenzene	103-33-3	5
4-Bromophenyl phenyl ether	101-55-3	5

Table B-1.

TAL Method 8270BP Standard Reporting Limits (cont.)

Analytes	CAS Number	Aqueous, µg/L
Hexachlorobenzene	118-74-1	1
Pentachlorophenol	87-86-5	10
Phenanthrene	85-01-8	1
Anthracene	120-12-7	5
Carbazole	86-74-8	5
Di-n-butyl phthalate	84-74-2	5
Fluoranthene	206-44-0	1
Benzidine	92-87-5	1
Pyrene	129-00-0	5
Butyl benzyl phthalate	85-68-7	5
3,3'-Dichlorobenzidine	91-94-1	5
Benzo(a)anthracene	56-55-3	1
Bis(2-ethylhexyl)phthalate	117-81-7	5
Chrysene	218-01-9	1
Di-n-octylphthalate	117-84-0	5
Benzo(b)fluoranthene	205-99-2	5
Benzo(k)fluoranthene	207-08-9	5
Benzo(a)pyrene	50-32-8	5
Indeno(1,2,3-cd)pyrene	193-39-5	5
Diethyl phthalate	84-66-2	5
Dibenz(a,h)anthracene	53-70-3	5
Benzo(g,h,i)perylene	191-24-2	5
1,4-Dioxane	123-91-2	1

Table B-2.

Method 8270BP Calibration Levels

Calibration Level	Calibration Concentration, µg/mL
1	0.25
2	0.40
3	1.00
4	2.50
5	5.00
6	7.50
7	10.
8	12.5
9	20.0
10	40.0
SSV	5.0

Table B-3.

Method 8270BP LCS Spike Concentrations

LCS Compounds	Spiking Level, ng/µL in extract[1]
Phenol	10
Bis(2-chloroethyl)ether	10
2-Chlorophenol	10
1,3-Dichlorobenzene	10
1,4-Dichlorobenzene	10
1,2-Dichlorobenzene	10
2,2'-oxybis(1-chloropropane)	10
N-Nitroso-di-n-propylamine	10
Hexachloroethane	10
Nitrobenzene	10
Isophorone	10
2-Nitrophenol	10
2,4-Dimethylphenol	10
Bis(2-chloroethoxy)methane	10
2,4-Dichlorophenol	10
1,2,4-Trichlorobenzene	10
Naphthalene	10
Hexachlorobutadiene	10
4-Chloro-3-methylphenol	10
Hexachlorocyclopentadiene	10
2,4,6-Trichlorophenol	10
2-Chloronaphthalene	10
Dimethyl phthalate	10
Acenaphthylene	10
Acenaphthene	10
2,4-Dinitrophenol	10
4-Nitrophenol	10
2,4-Dinitrotoluene	10
2,6-Dinitrotoluene	10
Diethylphthalate	10
4-Chlorophenyl phenyl ether	10
Fluorene	10
4,6-Dinitro-2-methylphenol	10
N-Nitrosodiphenylamine	10
4-Bromophenyl phenyl ether	10
Hexachlorobenzene	10
Pentachlorophenol	10
Phenanthrene	10
Anthracene	10
Di-n-butyl phthalate	10
Fluoranthene	10
Benzidine	10
Pyrene	10
Butyl benzyl phthalate	10
3,3'-Dichlorobenzidine	10
Benzo(a)anthracene	10

Table B-3.

Method 8270BP LCS Spike Concentrations (cont.)

LCS Compounds	Spiking Level, ng/µL in extract[1]
Bis(2-ethylhexyl)phthalate	10
Chrysene	10
Di-n-octylphthalate	10
Benzo(b)fluoranthene	10
Benzo(k)fluoranthene	10
Benzo(a)pyrene	10
Indeno(1,2,3-cd)pyrene	10
Dibenz(a,h)anthracene	10
Benzo(g,h,i)perylene	10
N-Nitrosodimethylamine	10
1,4-Dioxane	10

Table B-4.

8270BP Surrogate Compounds

Surrogate Compounds	Spiking Level, ng/µL in extract
Nitrobenzene-d5	5
2-Fluorobiphenyl	5
Terphenyl-d14	5
1,2-Dichlorobenzene-d4[1]	5
Phenol-d5	7.5
2-Fluorophenol	7.5
2,4,6-Tribromophenol	7.5
2-Chlorophenol-d4[1]	7.5

Table B-5.

Suggested Instrument Conditions for 8270BP

Mass Range:	35 - 500 amu
Scan Time:	≤1 second/scan
Initial Column Temperature/Hold Time:	50 °C for 1 minutes
Column Temperature Program:	50 - 320 °C at 35°C/min.
Final Column Temperature/Hold Time:	325 °C/4 min hold
Injector Temperature:	275 °C
Transfer Line Temperature:	290 °C
Source Temperature:	230 °C
Injector:	Single Taper Direct Connect Liner /splitless
Sample Volume:	0.5 µl
Carrier Gas:	Helium at 1.0mL/min.
Column:	DB-5 Capillary 20m x 0.18mm x 0.36 um film thickness

APPENDIX C

Instrument Maintenance Schedules - Mass Spectrometer & Gas Chromatograph

MASS SPECTROMETER Instrument Maintenance Schedule				
Daily	Weekly	As Needed	Quarterly	Annually
Check for sufficient gas supply. Check for correct column flow and/or inlet pressure	**Check mass calibration (PFTBA or FC-43).**	**Check level of oil in mechanical pumps and diffusion pump if vacuum is insufficient. Add oil if needed between service contract maintenance.**	**Check vacuum, relays, gas pressures, and flows.**	**Replace the exhaust filters on the mechanical rough pump every 1 to 2 years.**
Check temperatures of injector, detector. Verify temperature programs.		**Replace electron multiplier when the tuning voltage approaches the maximum and/or when sensitivity falls below required levels.**		**Change the oil in the mechanical rough pump.**
Check inlets, septa.		**Clean source, including all ceramics and lenses. Source cleaning is indicated by a variety of symptoms, including inability of the analyst to tune the instrument to specifications, poor response, and high background contamination.**		**Relubricate the turbomolecular pump-bearing wick.**
Check baseline level.		**Repair/replace jet separator.**		
Check values of lens voltages, electron multiplier, and relative abundance and mass assignments of the calibration compounds.		**Replace filaments when both filaments burn out or performance indicates the need for replacement.**		

APPENDIX C

Instrument Maintenance Schedules - Mass Spectrometer & Gas Chromatograph (cont.)

GAS CHROMATOGRAPH Instrument Maintenance Schedule (For GC/MS only.)	
Daily	*As Needed*
Check for sufficient supply of carrier and detector gases. Check for correct column flow and/or inlet pressures.	***Replace front portion of column packing or guard column or break off front portion of capillary columns. Replace column if this fails to restore column performance or when column performance indicates it is required (e.g., peak tailing, poor resolution, high backgrounds, etc.).***
Check temperatures of injectors and detectors. Verify temperature programs.	***Change glass wool plug in injection port and/or replace injection port liner when front portion of column packing is changed or front portion of capillary column is removed.***
Check inlets, septa. ***Clean injector port.***	***Replace septa.***
Check baseline level.	***Perform gas purity check (if high baseline indicates that impure carrier gas may be in use).***
Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks.	***Repair or replace flow controller if constant gas flow cannot be maintained.***
	Reactivate flow controller filter dryers when the presence of moisture is suspected.
	Autosampler: Replace syringe, fill wash bottle, dispose of waste bottle contents.

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Title: Extraction of Aqueous Samples by Continuous Liquid/Liquid Extraction (CLLE) by Method SW-846 3520C and Methods 625 and 607

Approvals (Signature/Date):

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1.0 Scope and Application

This Standard Operating Procedure (SOP) is applicable to the solvent extraction of organic compounds from aqueous samples, TCLP leachates, and SPLP leachates using a continuous liquid/liquid extractor (CLLE). This SOP based on SW-846 Method 3520C and EPA Methods 625 and 607.

The determinative methods used in conjunction with this procedure are listed in Table 1. This extraction procedure may be used for additional methods when appropriate pH and spiking mixtures are used.

This procedure does not include the concentration and cleanup steps. See SOP DV-OP-0007, "Concentration of Organic Extracts", for details concerning the concentration and cleanup of extracts.

2.0 Summary of Method

A measured volume of sample, usually 1 liter, is placed in a continuous liquid/liquid extractor (CLLE). The pH is adjusted, as required, for the efficient extraction of specific compounds. The organic compounds are extracted with methylene chloride for 18 to 24 hours. A second extraction at a different pH may be also required. The water phase is discarded.

3.0 Definitions

3.1 AFCEE: Air Force Center for Environmental Excellence.

3.2 Aliquot: A part which is a definite fraction of a whole; as in a "sample aliquot for testing or analysis." "Aliquot" is also used as a verb meaning to take all or part of a sample for preparation, extraction, and/or analysis.

3.3 Extraction Holding Time: The elapsed time, expressed in days, from the date of collection of the sample to the date of extraction, i.e., the date solvent comes in contact with the sample. The holding time is tracked in the laboratory LIMS system, and is the primary basis of prioritizing work.

3.4 Preparation Batch: A group of up to 20 samples that are of the same matrix and are processed together in the same extraction event using the same procedure and lots of reagents and standards.

3.5 Quality Assurance Summary (QAS): Certain clients may require extensive specific project instructions or program QC, which are too lengthy to fit conveniently in the special instructions/client requirements field in LIMS. In those situations, laboratory Project Managers describe the special requirements in a written QAS to address these requirements. QASs are posted on a public drive for easy accessibility by all laboratory employees. Normally, QASs are

introduced to analysts in an initial project kick-off meeting to be sure that the requirements are understood.

3.6 QC Program: The QC Program is the portion of the LIMS structured analysis code (SAC) assigned to a sample that defines the client QA program that applies to the analytical work. For example,

01 = Standard commercial work
9G = AFCEE program under the Version 3.1 QAPP

As such, the QC Program defines in the LIMS database a specific set of spike compounds, and control limits. It is necessary to check the QC Program to know which spike solution and how much spike to use for batch QC.

4.0 Interferences

4.1 Chemical and physical interferences may be encountered when analyzing samples using this method.

4.2 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.3 Visual interferences or anomalies (such as foaming, emulsions, odor, etc.) must be documented.

4.4 The most common interference is laboratory contamination, which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

4.5 The decomposition of some analytes has been demonstrated under basic extraction conditions. Organochlorine pesticides may dechlorinate, phthalate esters may exchange, and phenol may react to form tannates. These reactions increase with increasing pH, and are decreased by the shorter reaction times available in Method 3510C. Method 3510C is preferred over Method 3520C for the analysis of these classes of compounds. However, using Method 3520C and performing the initial extraction at the acid pH optimizes the recovery of phenols.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and

health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 CLLE glassware is often times fragile and awkward to use. Care should be taken to prevent breakage and injury. During the coarse of the procedure it might be necessary to adjust the height of the heating mantle. This will cause the CLLE body to tilt. Do not tilt the CLLE body more than 15 degrees to avoid stressing the glass and making the CLLE unstable. See Attachment 2 for troubleshooting.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Materials with Serious or Significant Hazard Rating

Material [1]	Hazards	Exposure Limit [2]	Signs and Symptoms of Exposure
Methylene Chloride	Carcinogen Irritant	25 ppm (TWA) 125 ppm (STEL)	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting, and headache. Causes irritation, redness, and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sodium Hydroxide	Corrosive Poison	2 mg/m3	Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat, and runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes and can cause burns that may result in permanent impairment of vision, even blindness with greater exposures.

Materials with Serious or Significant Hazard Rating (continued)

Material (1)	Hazards	Exposure Limit (2)	Signs and Symptoms of Exposure
Hydrochloric Acid	Corrosive Poison	5 ppm (Ceiling)	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Sulfuric Acid	Corrosive Carcinogen	1 mg/m3	Inhalation may cause irritation of the respiratory tract with burning pain the nose and throat, coughing, wheezing, shortness of breath, and pulmonary edema. Causes chemical burns to the respiratory tract. Inhalation may be fatal as a result of spasm, inflammation, edema of the larynx and bronchi, chemical pneumonitis, and pulmonary edema. Causes skin burns. Causes severe eye burns. May cause irreversible eye injury, blindness, permanent corneal opacification.

(1) Always add acid to water to prevent violent reactions.
(2) Exposure limit refers to the OSHA regulatory exposure limit

6.0 Equipment and Supplies

NOTE: All glassware used in this procedure is cleaned following SOP DV-OP-0004. In addition, the glassware is rinsed with methylene chloride immediately prior to use. For the CLLEs used in the extraction of NDMA for analysis by GC/CI/MS/MS, please see section 11.4 for glassware pre-cleaning. The CLLEs used in the extraction of NDMA for analysis by GC/CI/MS/MS are CLLEs dedicated to only this extraction.

6.1 Supplies

- Continuous Liquid-Liquid Extractor (CLLE), equipped with ground glass joints and polytetrafluoroethylene (PTFE) stopcock. (See Figure 1.)
- 250mL boiling flask with ground glass joint.
- Boiling Chips, contaminant free, approximately 10/40 mesh, Teflon®, PTFE.
- Cooling Condensers
- Re-circulating Chiller
- Heating Mantle, Rheostat controlled.
- Balance, ≥ 1400 g capacity, accurate to ± 1 g, calibrated daily per SOP DV-QA-0014.
- pH indicator paper, wide range.
- Class A Graduated Cylinder, sizes ranging from 100 mL to 1 L.
- Teflon® stir rods
- Mechanical pipette, 1 mL, calibrated per SOP DV-QA-0008.
- Aluminum foil.
- Paper towels.

7.0 Reagents and Standards

Reagents - All materials must be reagent grade or higher quality, unless otherwise specified

7.1 Reagent Water

7.1.1 Testamerica Denver has two ELGA water purification systems. The water coming from the ELGA system should be 17-18.2 Mohm-cm. The performance of the water polishing system is checked daily and recorded per SOP DV-QA-0026.

7.1.2 For all methods except low-level NDMA by GC/CI/MS/MS, reagent water is prepared by adding approximately 1 gram of baked sodium chloride to 1 L of the organic-free de-ionized water from the ELGA water system. This is done to give the reagent water some ionic strength to more closely mimic the matrix of actual water samples. Record the lot number of the sodium chloride on the bench sheet.

7.1.3 For low-level NDMA by GC/CI/MS/MS there is a concern that free chlorine may interfere with the analysis. Therefore the organic-free de-ionized water dispensed from the ELGA system is defined as reagent water for this extraction.

7.2 Methylene Chloride

Each lot of solvent is tested following SOP S-T-001 or S-T-001 DEN-1 before it is put into use. QA personnel post the list of approved lots at solvent storage areas.

7.3 Acids and Bases

7.3.1 Sulfuric Acid (H2SO4), 1:1

Place an ice water bath on a stir plate. Place a container with a magnetic stir bar in the bath. While stirring, slowly add 1 part concentrated reagent grade sulfuric acid (36N) to 1 part reagent water.

7.3.2 Sodium Hydroxide (NaOH), 10N

Purchased at ready-to-use concentration from commercial vendors.

7.4 Sodium Thiosulfate, 0.08 g/mL solution

Dissolve 8 grams of sodium thiosulfate in 100mL of water from the ELGA system.

7.5 Baked Sodium Chloride

Bake in 400 °C oven for at least 4 hours.

Standards - Stock standards are obtained from commercial sources.

7.6 Verification

All standards are subject to verification using a second-source standard before they are used for sample analysis. This process is described in SOP DV-QA-0015.

7.7 Storage Requirements

All standards must be refrigerated at 4 ± 2 °C and protected from light. Standard solutions are allowed to adjust to room temperature before using.

7.8 Expiration Date of Standards

7.8.1 Stock standards are monitored for signs of degradation or evaporation. The standards must be replaced annually from the date of opening or earlier if the vendor indicates an earlier date.

7.8.2 Dilutions from stock standards cannot have a later expiration date than the date assigned to the parent stock solutions. The standards must be replaced at least every six months or sooner if comparison with check standards indicates a problem.

7.9 Working Spike Solutions for LCS and MS/MSD

7.9.1 If a project requires a non-routine set of spike compounds, the current working spiking standard is reviewed to see if the required compounds are already included. A new standard does not always have to be made if more compounds are included than the project requires. That is to say, the LCS and MS/MSD do not have to be evaluated for all compounds in the spike solution if only a subset of compounds is needed. It depends on how the PM and QA set up the work in LIMS based on client instructions.

7.9.2 TestAmerica Denver's QC Policy DV-QA-003P requires that the same spike solution and spike amount are used for LCSs and MS/MSDs.

7.9.3 The following sections describe the standard solutions used for preparing spikes and surrogates for each determinative method used with this SOP. Except where noted, each is designed so that 1 mL of spike solution is added to make the LCS, MS, and MSD, and 1 mL of surrogate solution is added to all samples and QC in the batch.

7.9.4 Standards for Semi-Volatile Organics in Waters and SPLP Leachates by 8270

7.9.4.1 8270 standard solutions are prepared in purge and trap grade methanol as shown in Table 2. The standards are given a 1 year expiration date.

7.9.4.2 Some clients may require samples to be spiked with a longer list of compounds (i.e., "Full List Spike"). In this case, the LCS standard for method 625 should be used (see next section).

7.9.5 Standards for Semi-Volatile Organics in Waters by Method 625

7.9.5.1 Standard solutions for Method 625 are purchased in a methanol - methylene chloride mixture (75:25) as described in Table 3. The standard is given a 1 year expiration date.

7.9.5.2 A separate benzidine spiking solution is prepared in purge and trap grade methanol as described in Table 4. The expiration date for this standard is one week from the date of preparation. 1.5 mL of this standard is added to every LCS/LCSD and MS/MSD for method 625 as needed.

7.9.5.3 The Method 625 surrogate solution is prepared in purge and trap grade methanol as described in Table 3.

7.9.6 Standards for Low-Level Semi-Volatile Organics in Waters by 8270 Best Practices

7.9.6.1 Method 8270 Best Practice spiking solution is described in Table 5. It is prepared by performing a 1:10 dilution of the Method 625 LCS spiking solution described in Table 3 in purge and trap grade methanol. It is given a 1 year expiration date.

7.9.6.2 Method 8270 Best Practices surrogate solution is prepared in purge and trap grade methanol as described in Table 5. It is given a 1 year expiration date.

7.9.7 Standards for Semi-Volatile Organics in TCLP Leachates by 8270

7.9.7.1 TCLP standard solution leachates for 8270 analysis are prepared in purge and trap grade methanol as shown in Table 6. The standard is given a 1 year expiration date.

7.9.8 Standards for Polynuclear Aromatic Hydrocarbons (PAHs) in Waters by 8270 SIM

7.9.8.1 PAH standard spike solution is prepared by performing a 1:200 dilution of the Method 625 spike solution described in Table 3 in 90:10 purge and trap methanol : methylene chloride. The standard is given a 1 year expiration date.

7.9.8.2 PAH standard surrogate solution is prepared by performing a 1:200 dilution of the 8270 standard surrogate solution described in Table 2 in purge and trap methanol. The standard is given a 1 year expiration date.

7.9.9 Standards for Pentachlorophenol in Waters by 8270 SIM

7.9.9.1 PCP standard spike solution is prepared in purge and trap methanol at a concentration of 5 ug/mL. The standard is given a 1 year expiration date.

7.9.9.2 For the extraction of PCP by 8270 SIM, the Method 8270 SIM surrogate standard described in Section 7.9.8.2 is used.

7.9.10 Standards for NDMA by 8070A and 607

7.9.10.1 NDMA standard solutions are prepared in methanol as described in Table 7. The standards are given a 6 month expiration date.

7.9.11 Standards for NDMA in Waters by GC/CI/MS/MS

7.9.11.1 NDMA standard solutions are prepared in methanol as described in Table 8. The standards are given a 6 month expiration date.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Waters	Amber Glass	1000 mL	Cool 4 ± 2°C	7 Days	40 CFR Part 136.3
TCLP Leachates	Glass	200 mL	Cool 4 ± 2°C	7 Days from the completion of the leach	SW-846 1311
SPLP Leachates	Glass	1000 mL	Cool 4 ± 2°C	7 Days from the completion of the leach	SW-846 1312

[1] Exclusive of analysis.

9.0 Quality Control

- The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS QC program code and special instructions to determine specific QC requirements that apply.

- The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, Quality Control Program.

- Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), AFCEE, etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs.

- Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via special instructions in the LIMS.

- Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.1 **Sample QC** - The following quality control samples are prepared with each batch of samples.

9.1.1 Preparation Batch

A batch can contain up to 20 field samples. Each preparation batch must contain a method blank, an LCS, and a matrix spike/matrix spike duplicate (MS/MSD) pair. Special program or project requirements can include additional requirements. Always refer to special project instructions for details before proceeding with the analysis.

Different TCLP and SPLP batches can result from leaching samples of different waste matrix types and can also have been generated using different extraction fluids. For these reasons, TCLP batches must be kept separate from other water samples.

9.1.2 Method Blank (MB)

At least one method blank must be processed with each preparation batch.

The method blank is used to identify any background interferences or contamination of the analytical system that may lead to the reporting of elevated concentration levels or false positive data.

Aqueous method blanks are prepared using 1,000 mL of reagent water spiked with the surrogates.

TCLP method blanks for method 8270 use 200 mL of leachate fluid spiked with the surrogates. SPLP method blanks for method 8270 use 1 L of leachate fluid spiked with the surrogates.

Acceptance Criteria: Acceptance criteria for the method blank are prescribed in the SOP for the appropriate determinative method.

Corrective Actions: If the method blank fails the acceptance criteria for the determinative method and/or the project, the general requirement in policy DV-QA-003P, Quality Control Program is to re-extract and reanalyze all associated samples.

9.1.3 Laboratory Control Sample (LCS)

At least one LCS must be processed with each preparation batch. Some projects require two LCSs, an LCS and an LCS duplicate (LCSD) in every batch. Check special project instructions for non-routine LCS requirements.

Methods 607 and 625 require a LCS at a 10% frequency. In other words one LCS is required for a batch of 10 or less samples. A LCSD is required for a batch of 11 or more samples.

The LCS is used to monitor the accuracy of results within the batch and long-term precision. It provides evidence that the method is being performed properly, independent of any sample matrix effects.

Aqueous LCSs are prepared using 1,000 mL of reagent water that is spiked with the compounds of interest and surrogate compounds, as shown in Tables 2-8.

TCLP LCSs for method 8270 use 200 mL of leachate fluid spiked with the surrogates and the LCS standard. SPLP LCSs for method 8270 use 1 L of leachate fluid spiked with the surrogates and the LCS standard.

Acceptance Criteria: Acceptance Criteria for LCS analysis are prescribed in the SOP for the appropriate determinative method. Acceptance criteria are typically specified in terms of statistical control limits based on historical data, as described in Policy DV-QA-003P.

Corrective Actions: Because LCSs are completely under the laboratory's control, failing LCS acceptance criteria generally requires re-extraction and reanalysis of associated samples. On-line control charts of LCS results over time are available and are useful for trouble-shooting method performance problems.

9.1.4 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked exactly as the MS) prepared and analyzed along with the sample and matrix spike. One MS/MSD pair must be processed for each preparation batch.

Methods 607 and 625 require one matrix spike for every 10 samples. These methods require a MSD if there are more than 10 samples per batch.

Note that some programs (e.g., North Carolina and South Carolina) require that MS/MSD pairs are run at a 10% frequency. For a batch of 10 or less, one MS/MSD pair is required, (2 spiked samples). For a batch of 11 or more 2 MS/MSDs pairs are required (4 spiked samples).

Samples identified as field blanks or equipment blanks cannot be used for MS/MSD analysis.

If insufficient sample volume is available for MS/MSD, an NCM must be written. For SW-846 methods a LCS/LCSD will be required in this case.

Acceptance Criteria: Acceptance Criteria for MS/MSD evaluation are prescribed in the SOP for the appropriate determinative method. Acceptance criteria are typically specified in terms of statistical control limits for both the analyte recovery and relative percent difference (RPD) between the MS and MSD based on historical data, as described in Policy DV-QA-003P.

Corrective Actions: If the method blank and LCS results are within control limits, and the MS/MSD results fail to meet acceptance criteria, the indications are that the analytical system is in control and the MS/MSD failure may be the result of a matrix effect specific to the sample from which the MS and MSD were prepared. In these cases, the general requirement is to qualify the results reported to the client using data qualifier flags and/or comments in the final report case narrative.

9.1.5 Surrogate Spikes

Each sample, method blank, LCS, and MS/MSD in the batch is spiked with surrogate standard. Surrogates are organic compounds which are similar to the target analytes in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples. Surrogates are used to monitor the accuracy of results for each sample.

Acceptance Criteria: Surrogate spike recoveries are evaluated against percent recovery control limits that are based on statistical evaluation of historical data.

Corrective Actions: If LCS and method blank results are within control limits but sample surrogate recoveries are outside of control limits, the general requirement is to qualify the results reported to the client using data qualifier flags and/or comments in the final report case narrative. However, some projects require automatic re-extraction when surrogates fail. Any special requirements such as this will be documented in special project instructions and/or the QAS.

10.0 Procedure

One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented

using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.1 Critical Procedural Considerations

10.1.1 As stated throughout this SOP, analysts must review client requirements, the LIMS test code (SAC), and any applicable QASs before starting work. This review is also documented on the Organic Extraction Checklist (see Attachment 1).

10.1.2 Analyst must focus on using clean technique throughout this procedure. Any parts or pipettes that come into direct contact with dirty surfaces or any other separatory funnel than the designated one should be cleaned or disposed of before coming into contact with the sample.

10.1.3 If problems arise during the extraction, see Attachment 2: Troubleshooting Guide.

10.2 Special Pre-Extraction Procedure for NDMA by GC/CI/MS/MS

10.2.1 For each sample and QC sample, rinse a 250 mL boiling flask with methylene chloride. Add a few boiling chips and fill with about 150 mL of methylene chloride.

10.2.2 Use only CLLEs dedicated to this extraction. For each sample and QC sample, rinse a CLLE with methylene chloride. Then fill the bottom of the CLLEs with methylene chloride to a level approximately 2 cm above the opening of the solvent flow arm. (See Figure 1.)

10.2.3 Set the CLLE on a stand in front of a heating mantle and under a slot hood. Attach the 250 mL boiling flask containing methylene chloride and boiling chips to the side arm of the CLLE and place a heating mantle under the flask.

10.2.4 With the stopcock of the CLLE open, add reagent water to each CLLE until the methylene chloride in the bottom of the CLLE starts to spill over through the solvent flow arm and into the boiling flask. Be careful not to allow water to spill over. You may need to add additional methylene chloride to the CLLE to prevent water from entering the solvent flow arm.

10.2.5 Attach the cold condenser (chilled at 10 °C). Check that the hoses are not kinked. Check the temperature and the water level of the chiller.

10.2.6 Check again for boiling chips in the boiling flask. Turn on the heating mantle. Inspect joints for leaks once solvent has begun cycling

10.2.7 Extract for approximately 18 hours.

10.2.8 At the end of the pre-extraction, turn off the heating mantle and allow the extractor to cool. Remove the boiling flasks and discard the methylene chloride in the appropriate waste container.

10.2.9 For the CLLEs to be used for the extraction of samples and MS/MSDs, remove the water and methylene chloride from the extractor body, discarding them in the appropriate waste containers. Return the CLLEs to the stands under the slot hood. For the CLLEs to be used for the extraction of method blanks and LCSs, the water and the small amount of methylene chloride are kept in the CLLE. This pre-extracted water will be used as the method blank and LCS samples.

10.2.10 Rinse a fresh 250 mL boiling flask with methylene chloride. Add a few boiling chips and fill with about 150 mL of methylene chloride. Attach these boiling flasks to the side arms of the CLLEs.

10.2.11 Fill the bottom of the CLLEs to be used for samples and MS/MSDs with methylene chloride to a level approximately 2 cm above the opening of the solvent flow arm. (See Figure 1.)

10.2.12 Label each boiling flask with the sample ID or batch QC ID.

10.2.13 Proceed to Section 10.4

10.3 Assemble and Clean the Glassware Immediately Before Use

10.3.1 For each sample and QC sample, rinse a 250 mL boiling flask with methylene chloride. Add a few boiling chips and fill with about 150 mL of methylene chloride.

10.3.2 For each sample and QC sample, rinse a CLLE with methylene chloride. Then fill the bottom of the CLLEs with methylene chloride to a level approximately 2 cm above the opening of the solvent flow arm. (See Figure 1.)

10.3.3 Set the CLLE on a stand in front of a heating mantle and under a slot hood. Attach the 250 mL boiling flask containing methylene chloride and boiling chips to the side arm of the CLLE and place a heating mantle under the flask.

10.3.4 Label each boiling flask with the sample ID or batch QC ID. Also include on the label what fraction (acid or base) if the method calls for extraction at a secondary pH. (See Section 10.8).

10.4 Aliquot Samples

10.4.1 Note the differences in aliquotting samples for SW-846 extraction methods in Section 10.4.3 versus aliquotting for 600 series methods in Section 10.4.4.

10.4.2 It should be noted that TestAmerica Denver routinely aliquots samples gravimetrically. This is done to prevent cross-contamination due to

volumetric glassware and to provide a more accurate initial volume measurement. However, some clients and regulatory programs require the laboratory to aliquot samples volumetrically. The client requirements and QASs must be read before samples are aliquotted to check for this requirement. If samples are to be aliquotted volumetrically, use Class A volumetric glassware only.

10.4.3 Aliquot Samples for SW-846 Methods

10.4.3.1 Weigh the bottle containing the approximately 1 liter sample and record the gross weight to the nearest gram.

10.4.3.2 Measure the initial sample pH with wide-range pH paper and record the pH on the extraction bench sheet. If the sample is a leachate, compare the current pH against the pH recorded on the TCLP worksheet. Note any discrepancies in an NCM.

10.4.3.3 Open the stopcock if the CLLE is equipped with one. Pour the sample into the CLLE on top of the methylene chloride. If the sample bottle contains a significant amount of sediment, decant the water, leaving the sediment in the sample container. Be careful to allow only the methylene chloride in the bottom of the CLLE to spill over into the boiling flask, and not to allow water to spill over. Add additional methylene chloride or reagent water to the CLLE if needed.

NOTE: Some programs (e.g. South Carolina, USACE, DoD, and Wisconsin) require that the entire sample is used and the sample container is rinsed with methylene chloride (decanting of solids not allowed). Client requirements and any QASs must be read before extraction begins.

10.4.3.4 Reweigh the bottle and calculate the initial sample volume by subtracting the empty bottle's weight from the full bottle's weight, assuming a density of 1g=1mL. If there is any indication that the sample's density is not 1g=1mL, then measure the density of the sample and correct the calculated initial volume accordingly. See Section 11 for the calculation. Document abnormal sample density in an NCM.

10.4.3.5 If the initial volume is less than 800 mL the sample reporting limits and method detection limits will be elevated accordingly. Document this in a NCM.

10.4.3.6 For TCLP leachates, use a Class A graduated cylinder to measure out 200 mL for method 8270 extraction into the labeled CLLE. Add reagent water to the CLLE to ensure proper flow. For SPLP leachates, use a Class A graduated cylinder to measure out 1000mL for method 8270 extraction into the labeled CLLE.

10.4.4 Aliquot Samples for 600 Series Methods

10.4.4.1 Weigh the bottle containing the approximately 1-liter sample and record the gross weight to the nearest gram.

10.4.4.2 Mix the sample well by shaking the container.

10.4.4.3 Measure the initial sample pH with wide-range pH paper and record the pH on the extraction bench sheet.

10.4.4.4 Open the stopcock if the CLLE is equipped with one. Pour the entire sample into the CLLE on top of the methylene chloride. The entire sample must be used. Be careful to allow only the methylene chloride in the bottom of the CLLE to spill over into the boiling flask, and not to allow water to spill over.

10.4.4.5 Rinse the sample bottle with methylene chloride and add it to the CLLE. Add additional methylene chloride or reagent water to the CLLE if needed.

10.4.4.6 Reweigh the bottle and calculate the initial sample volume by subtracting the empty bottle's weight from the full bottle's weight, assuming a density of 1g=1mL. If there is any indication that the sample's density is not 1g=1mL then measure the density of the sample and correct the calculated initial volume accordingly. See Section 11 for the calculation. Document abnormal sample density in an NCM.

10.4.4.7 If the initial volume is less than 800 mL the sample reporting limits and method detection limits will be elevated accordingly. Document this in a NCM.

10.5 Prepare Method Blanks

10.5.1 Add approximately 1000 mL of reagent water to CLLEs marked for the method blanks. Add enough reagent water to cause the methylene chloride to begin to pour over the arm and into the boiling flask.

NOTE: If any sample in the batch is preserved with sodium thiosulfate, then the method blank should also contain sodium thiosulfate. Add 1 mL of the 0.08 g/mL solution to the water in the CLLE. Samples that are chlorinated waters should be preserved with sodium thiosulfate.

10.5.2 For TCLP method blanks, use the appropriate TCLP leaching fluid as provided by the TCLP lab. Use 200 mL of the leach fluid for method 8270 TCLP method blanks. For SPLP method blanks, use 1 liter of the appropriate SPLP leach fluid. If necessary, add reagent water to the CLLEs to cause the methylene chloride to begin to pour through the solvent flow arm and into the boiling flask.

10.5.3 For low-level NDMA the pre-extracted water generated in Section 10.2 is used for the method blank.

10.6 Prepare LCS and MS/MSD QC Samples

NOTE: For SW-846 methods if there is not a MS/MSD pair in the batch then perform a LCS/LCSD. Methods 607 and 625 require a LCS and LCSD in batches of II samples or more.

10.6.1 For aqueous sample batches, add 1 liter of reagent water to CLLEs marked for the LCS.

NOTE: If any sample in the batch is preserved with sodium thiosulfate, then the LCS should also contain sodium thiosulfate. Add 1mL of the 0.08 g/mL solution to the water in the CLLE.

10.6.2 For TCLP batches, prepare the LCS using 200 mL of the appropriate leach fluid for method 8270 LCSs. For SPLP batches, prepare the LCS using 1 liter of the appropriate leach fluid. If necessary add reagent water to the CLLE to cause the methylene chloride to begin to pour through the solvent flow arm and into the boiling flask.

10.6.3 For low-level NDMA, the pre-extracted water generated in Section 10.2 is used for the LCS.

10.6.4 For the MS, add a second aliquot of the selected sample to a CLLE marked for the MS. For the MSD, add a third aliquot of the selected sample to a CLLE marked for the MSD.

10.6.5 Add the appropriate working LCS and MS/MSD standard (see Tables 2-8) to the CLLEs containing any LCS, LCSD, MS, and MSD samples. Record the ID of the standard used on the bench sheet. Note that the standard should be allowed to come to room temperature before spiking the samples.

10.6.6 Spiking events must be witnessed by a second person, and the witnessing is documented on the Organic Extraction Checklist (see Attachment 1).

10.7 Add Surrogates to All Field Samples and QC Samples

10.7.1 Add 1.0 mL of the appropriate working surrogate standard (see Tables 2-8) to the CLLEs for each field sample and QC sample. Record the ID of the standard used on the bench sheet.

NOTE: Some programs (e.g., South Carolina) require that the surrogate is added to the sample while the sample is still in the sample container. Client requirements and any QASs must be read before extraction begins.

10.8 Adjust pH of Field Samples and QC Samples

Adjust the sample pH as indicated in the chart below using a minimum amount of 1:1 sulfuric acid or 10 N sodium hydroxide, as necessary. Use a Teflon® stir rod to mix the sample after the addition of the acid or base. Check the pH using wide-range pH paper. Record the adjusted pH and the lot number of the acid or base on the bench sheet.

Method	Initial Extraction pH	Secondary Extraction pH
SW-846 8270 and Method 625 (Method Codes QL & DP)	1 – 2	11 - 12
SW-846 8270 SIM PAHs (Method Code IV)	As Received	None
SW-846 8270 SIM Pentachlorophenol (Method Code 6C)	1 – 2	None
SW-846 8270 Best Practice Low-Level Semi-Volatile Organics (Method Code 5M, Prep Code 49)	1 – 2	12 – 14
SW-846 8270 Best Practice Low-Level 1,4-Dioxane Only (Method Code 5M, Prep Code 10)	12 – 14	None
SW-846 8070A and Method 607	As Received	None
NDMA by GC/CI/MS/MS	As Received	None

10.9 Start the Initial Extraction

10.9.1 Attach the cold condenser (chilled at 10°C). Check that the hoses are not kinked. Check the temperature and water level of the chiller.

10.9.2 Turn on the heating mantle. Check again for boiling chips in the boiling flask and inspect joints for leaks once solvent has begun cycling. The methylene chloride in the round bottom flask should be boiling steadily, but not too rapidly that the condenser cannot condense the vapor as fast as it is being generated.

10.9.3 Record the date and the time the extraction started on the benchsheet.

10.9.4 For method 625, extract for 24 hours. For SW-846 methods, method 607, and low-level NDMA by GC/CI/MS/MS, extract for 18-24 hours. In order to consistently meet the DoD QSM control limits for the method 8270 surrogate 2-fluorobiphenyl, a 24 hour extraction is required.

10.9.5 At the end of the extraction, turn off the heating mantle and allow the extractor to cool. Record the date and time the extraction stopped on the benchsheet.

10.9.6 Remove the boiling flask, cap with aluminum foil, and store refrigerated until concentration. If extraction at a second pH is required continue on to Section 10.10. If extraction at a second pH is not required, go to Section 10.11.

10.10 Start the Secondary Extraction

10.10.1 Attach a boiling flask with fresh methylene chloride and boiling chips to the CLLE. Label each boiling flask with the sample ID or batch QC ID.

10.10.2 Remove the condenser and adjust the pH of the sample in the extractor body to the pH indicated in Section 10.8 with the minimum amount of 10N NaOH. Usually 10mL of base is needed. Use a Teflon ® stir rod to mix the sample. Measure with wide-range pH paper and record the adjusted pH and the lot number of the base used on the benchsheet.

10.10.3 Re-attach the cold condenser, turn on the heating mantle, and inspect the joints for leaks. Record the date and time the extraction started on the benchsheet.

10.10.4 For SW-846 methods, extract for 18-24 hours. For method 625, extract for 24 hours. In order to consistently meet the DoD QSM control limits for the method 8270 surrogate 2-fluorobiphenyl, a 24 hour extraction is required.

10.10.5 At the end of the second extraction, turn off the heating mantle and allow the extractor to cool. Record the date and time the extraction stopped on the benchsheet.

10.10.6 Remove the boiling flask, cap with aluminum foil, and store refrigerated until concentration.

10.11 Disposal of Waste

10.11.1 Dispose of the methylene chloride remaining in the CLLE body in the methylene chloride Waste Stream B.

10.11.2 Dispose of the solvent-saturated water remaining in the CLLE in Waste Stream X if pH is neutral or basic or in Waste Stream Y if pH is acidic.

10.12 Initial weights and volumes of samples are entered into LIMS, and the transcribed data must be verified by a second person. This verification is documented on the Organic Extraction Checklist (see Attachment 1).

10.13 Wash glassware following SOP DV-OP-0004.

11.0 Data Analysis and Calculations

$$InitialVolume(mL) = \frac{FullBottle(g) - EmptyBottle(g)}{Density(g/mL)}$$

12.0 Method Performance

12.1 Before analyzing samples, the laboratory must establish a method detection limit (MDL). See Policy DV-QA-005P, "Determination of Method Detection Limits", for more information on the method detection limit studies.

12.2 An initial demonstration of capability (IDOC) must be performed by each analyst. On-going proficiency must be demonstrated by each analyst on an annual basis. See DV-QA-0024, "Employee Training", for more information on the IDOCs.

12.3 Training Qualification

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience. Further details concerning the training program are described in SOP DV-QA-0024.

12.4 Calibration
N/A

12.5 Sample Analysis
N/A

13.0 Pollution Control

The volume of spike solutions prepared is minimized to reduce the volume of expired standard solutions requiring hazardous waste disposal.

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health and Safety Manual, and HS-001, "Waste Management Program."

14.2 The following waste streams are produced when this method is carried out:

14.2.1 Methylene chloride – Waste Stream B

14.2.2 Neutral aqueous sample waste saturated with methylene chloride – Waste Stream X.

14.2.3 Basic aqueous sample waste saturated with methylene chloride – Waste Stream X.

14.2.4 Acidic aqueous sample waste saturated with methylene chloride – Waste Stream Y.

14.2.5 Expired Standards/Reagents – Contact Waste Coordinator for guidance

NOTE: Radioactive waste, mixed waste, and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of these materials.

15.0 References / Cross-References

15.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005, Method 3520C, Continuous Liquid-Liquid Extraction, Revision 3, December 1996.

15.2 Code of Federal Regulations, Title 40 – Protection of the Environment, Part 136 – Guidelines Establishing Test Procedures for the Analysis of Pollutants, Appendix A – Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, Method 625.

15.3 Code of Federal Regulations, Title 40 – Protection of the Environment, Part 136 – Guidelines Establishing Test Procedures for the Analysis of Pollutants, Appendix A – Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, Method 607.

16.0 Modifications:

16.1 Modifications from SW846 Method 3520C

16.1.1 Section 7.1 of the method calls for initial sample volume to be determined volumetrically. This SOP allows the initial sample volume to be determined by weight.

16.1.2 Section 7.2 of the method calls for the surrogate standard to be added to the sample while the sample is in the sample container or the graduated cylinder used for measuring the initial volume. This SOP calls for the surrogate standard to be added to the sample after the sample is transferred to the CLLE.

16.1.3 Section 7.7 of the method states that if the acid and base extracts are not to be analyzed separately, then the boiling flask and solvent does not need to be changed, but may be used for the second pH extraction. This is currently not

the practice here at STL Denver in order to prevent loss of some of the compounds that would extract in the first 18-hour extraction.

16.1.4 Table 1 of the method calls for the secondary extraction pH for method 8270C to be >11. This SOP calls for the secondary extraction pH for method 8270C to be ≥12.

16.2 Modifications from 40 CFR Method 625

16.2.1 Section 2.1 of the method calls for extracting the method at a pH greater than 11 and then again at a pH less than 2. This SOP calls for the acid extraction to be performed first followed by the base extraction. This is done to improve the recoveries of the phenols.

16.2.2 Section 11.2 of the method calls for initial sample volume to be determined volumetrically. This SOP allows the initial sample volume to be determined by weight.

16.2.3 The Method calls for the acid and base extracts to be concentrated and analyzed separately. This SOP calls for the extracts to be combined, then concentrated and analyzed.

16.3 Modifications from 40 CFR Method 607

16.3.1 The method calls for the extraction to be performed by separatory funnel. However it is STL Denver's experience that CLLE extraction produces better recoveries.

16.3.2 Section 10.1 of the method calls for initial sample volume to be determined volumetrically. This SOP allows the initial sample volume to be determined by weight.

17.0 Attachments

Figure 1. Continuous Liquid-Liquid Extractor (CLLE)

Table 1. Determinative Methods Using CLLE Extractions

Table 2. Working Standards for Waters and SPLP Leachates by 8270.

Table 3. Working Standards for Waters by 625 and AFCEE 8270

Table 4. Working Benzidine Spike Standards for 625

Table 5. Working Standards for 8270 Best Practice

Table 6. Working Standards for TCLP 8270.

Table 7. Working Standards for 8070A and 607

Table 8. Working Standards for NCMA by GC/CI/MS/MS

Attachment 1. Organic Extraction Checklist

Attachment 2. Trouble-shooting Guide

18.0 Revision History

- Revision 3, dated 20 March 2008
- Integration for TestAmerica and STL operations.

18.1 Modifications from Previous Revision.

18.1.1 Clarified the definition of holding time in Section 3.3

18.1.2 Added Attachment 2 – Troubleshooting Guide

18.1.3 Updated section 7.9 to include the expiration dates of all standards.

18.1.4 Table 2 was revised to add additional compounds in the method 8270 spike solution.

18.1.5 Table 6 was revised to lower the spike concentration of hexachlorobenzene and 2,4-dinitrotoluene. This was done to spike at or below the TCLP regulatory level for these compounds.

18.1.6 Table 7 was revised to include the surrogate compound n-Nitrosodiethylamine. This was done to provide a surrogate that elutes before the target compound n-nitrosodimethylamine.

18.1.7 Section 9.0 was updated to clarify the frequency requirement for LCS/LCSDs in method 607 and 625

18.1.8 Section 9.0 was revised to instruct the lab that for SW-846 method batches if a MS/MSD is not performed a LCS/LCSD is needed for precision.

18.1.9 Section 10.4 was revised to give more detail on the labs procedure for aliquoting samples grammatically.

18.1.10 Sections 10.9 and 10.10 were modified to require a 24 hour extraction for DoD samples for method 8270.

FIGURE 1.

Continuous Liquid-Liquid Extractor (CLLE)

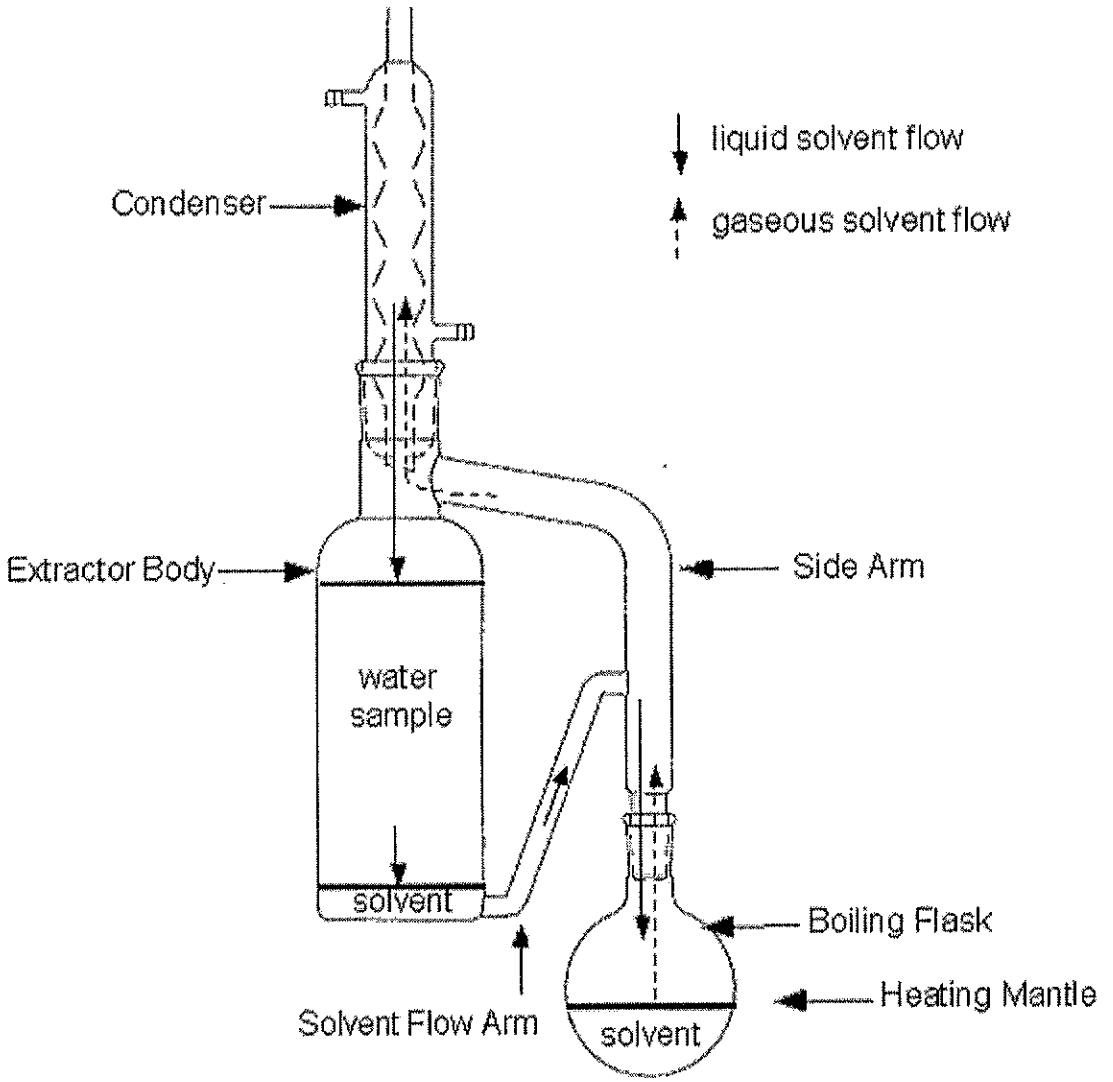

TABLE 1.

Determinative Methods Using CLLE Extractions

Method Description	*Determinative Method*	*SOP*
Semi-Volatiles by GC/MS	SW-846 8270 EPA Method 625	DV-MS-0011
Low-Level Semi-Volatiles by GC/MS Best Practice	SW-846 8270	DV-MS-0011
Polynuclear Aromatic Hydrocarbons by GC/MS SIM	SW-846 8270 SIM	DV-MS-0002
n-Nitrosodimethylamine by GC	SW-846 8070A EPA Method 607	DV-GC-0018
n-Nitrosodimethylamine by GC/CI/MS/MS	Per SOP	DV-LC-0019

TABLE 2.

Working Standards for Waters & SPLP Leachates by Method 8270

Spike Solution:	
Compound	**Concentration (μg/mL)**
1,2,4-Trichlorobenzene	100
1,4-Dichlorobenzene	100
2,4-Dinitrotoluene	100
2-Chlorophenol	100
4-Chloro-3-Methylphenol	100
4-Nitrophenol	100
Acenaphthene	100
N-nitrosodi-n-propylamine	100
Pentachlorophenol	100
Phenol	100
Pyrene	100
2.4.6-Trichlorophenol	100
2-Methylnaphthalene	100
2-Methylphenol	100
Anthracene	100
Carbazole	100
Surrogate Solution:	
2-Chlorophenol-d4	150
2-Fluorobiphenyl	100
2-Fluorophenol	150
Nitrobenzene-d6	100
p-Terphenyl-d14	100
Phenol-d6	150
1,2-Dichlorobenzene-d4	100
2,4,6-Tribromophenol	150

TABLE 3.

Working Standards for Waters by AFCEE Method 8270 and Method 625

Spike Solution:		
Compound	**Concentration (µg/mL)**	**Method 625 Standard Control Compounds**
1,2,4-trichlorobenzene	100	X
1,2-dichlorobenzene	100	X
1,3-dichlorobenzene	100	X
1,4-dichlorobenzene	100	X
2,4,5-trichlorophenol	100	
2,4,6-trichlorophenol	100	X
2,4-dichlorophenol	100	X
2,4-dimethylphenol	100	X
2,4-dinitrophenol	100	X
2,4-dinitrotoluene	100	X
2,6-dinitrotoluene	100	X
2-chloronaphthalene	100	X
2-chlorophenol	100	X
2-methyl-4,6-dinitrophenol	100	X
2-methylnaphthalene	100	
2-methylphenol	100	X
2-nitroaniline	100	
2-nitrophenol	100	X
3-nitroaniline	100	
4-bromophenylphenyl ether	100	X
4-chloro-3-methylphenol	100	X
4-chloroaniline	100	
Bis(2-chloroethyl)ether	100	X
Bis(2-chloroisopropyl)ether	100	X
Bis(2-ethylhexyl)phthalate	100	X
Butyl benzyl phthalate	100	X
Carbazole	100	
Chrysene	100	X
di-n-butyl phthalate	100	X
di-n-octyl phthalate	100	X
Dibenz(a,h)anthracene	100	X
Dibenzofuran	100	
Diethyl phthalate	100	X
Dimethyl phthalate	100	X
Fluoranthene	100	X
Fluorene	100	X
Hexachlorobenzene	100	X
Hexachlorobutadiene	100	X
Hexachlorocyclopentadiene	100	X
Hexachloroethane	100	X
Indeno(1,2,3-cd)pyrene	100	X
Isophorone	100	X
n-nitrosodi-n-propylamine	100	X

TABLE 3.

Working Standards for Waters for AFCEE Method 8270 and Method 625 (continued)

Compound	Concentration (µg/mL)	Method 625 Standard Control Compounds
n-nitrosodimethylamine	100	X
Naphthalene	100	X
Nitrobenzene	100	X
Pentachlorophenol	100	X
Phenanthrene	100	X
Phenol	100	X
Pyrene	100	X
1,4-dioxane	100	
2,3-dichloroaniline	100	
Acetophenone	100	
Alachlor	100	
Alpha-methyl styrene	100	
Atrazine	100	
Caprolactam	100	
Indene	100	
n-decane	100	
n-docosane	100	
n-dodecane	100	
n-eicosane	100	
n-octadecane	100	
n-tetradecane	100	
Aniline	100	
Benzoic acid	100	
Benzyl alcohol	100	
Pyridine	100	
a-methylnaphthalene	100	
n-nitrosodiphneylamine	100	X
3,3-dichlorobenzidine	100	X
3,3-dimethylbenzidine	100	
Surrogate Solution:		
2-Chlorophenol-d4	150	X
2-Fluorobiphenyl	100	X
2-fluorophenol	150	X
Nitrobenzene-d6	100	X
p-Terphenyl-d14	100	X
Phenol-d6	150	X
1,2-dichlorobenzene-d4	100	X
1,2,6-Tribromophenol	150	X

TABLE 4.

Working Benzidine Spike Standard for Method 625

Spike Solution:	
Compound	**Concentration (μg/mL)**
Benzidine	100

TABLE 5.

Working Spike Standard for 8270 Best Practices

Spike Solution:	
Compound	**Concentration (µg/mL)**
1,2,4-trichlorobenzene	10
1,2-dichlorobenzene	10
1,3-dichlorobenzene	10
1,4-dichlorobenzene	10
2,4,5-trichlorophenol	10
2,4,6-trichlorophenol	10
2,4-dichlorophenol	10
2,4-dimethylphenol	10
2,4-dinitrophenol	10
2,4-dinitrotoluene	10
2,6-dinitrotoluene	10
2-chloronaphthalene	10
2-chlorophenol	10
2-methyl-4,6-dinitrophenol	10
2-methylnaphthalene	10
2-methylphenol	10
2-nitroaniline	10
2-nitrophenol	10
3-nitroaniline	10
4-bromophenylphenyl ether	10
4-chloro-3-methylphenol	10
4-chloroaniline	10
Bis(2-chloroethyl)ether	10
Bis(2-chloroisopropyl)ether	10
Bis(2-ethylhexyl)phthalate	10
Butyl benzyl phthalate	10
Carbazole	10
Chrysene	10
di-n-butyl phthalate	10
di-n-octyl phthalate	10
Dibenz(a,h)anthracene	10
Dibenzofuran	10
Diethyl phthalate	10
Dimethyl phthalate	10
Fluoranthene	10
Fluorene	10
Hexachlorobenzene	10
Hexachlorobutadiene	10
Hexachlorocyclopentadiene	10
Hexachloroethane	10
Indeno(1,2,3-cd)pyrene	10
Isophorone	10
n-nitrosodi-n-propylamine	10

TABLE 5.

Working Spike Standard for 8270 Best Practices (continued)

Compound	Concentration (µg/mL)
n-nitrosodimethylamine	10
Naphthalene	10
Nitrobenzene	10
Pentachlorophenol	10
Phenanthrene	10
Phenol	10
Pyrene	10
1,4-dioxane	10
2,3-dichloroaniline	10
Acetophenone	10
Alachlor	10
Alpha-methyl styrene	10
Atrazine	10
Caprolactam	10
Indene	10
n-decane	10
n-docosane	10
n-dodecane	10
n-eicosane	10
n-octadecane	10
n-tetradecane	10
Aniline	10
Benzoic acid	10
Benzyl alcohol	10
Pyridine	10
a-methylnaphthalene	10
n-nitrosodiphneylamine	10
3,3-dichlorobenzidine	10
3,3-dimethylbenzidine	10
Surrogate Solution:	
2-Chlorophenol-d4	7.5
2-Fluorobiphenyl	5
2-fluorophenol	7.5
Nitrobenzene-d6	5
p-Terphenyl-d14	5
Phenol-d6	7.5
1,2-dichlorobenzene-d4	5
1,2,6-Tribromophenol	7.5

TABLE 6.

Working Standards for TCLP 8270

Spike Solution:	
Compound	**Concentration (μg/mL)**
1,4-dichlorobenzne	50
2,3,5-trichlorophenol	50
2,4,6-trichlorophenol	50
2,4-dinitrotoluene	20
2-methylphenol	50
3-methylphenol	50
4-methylphenol	50
Hexachlorobenzene	20
Hexachlorobutadiene	50
Hexachloroethane	50
Nitrobenzene	50
Pentachlorophenol	100
Pyridine	50
Surrogate Solution:	
2-Chlorophenol-d4	150
2-Fluorobiphenyl	100
2-fluorophenol	150
Nitrobenzene-d6	100
p-Terphenyl-d14	100
Phenol-d6	150
1,2-dichlorobenzene-d4	100
1,2,6-Tribromophenol	150

TABLE 7.

Working Standards for NDMA by Method 8070A and 607

Spike Solution:	
Compound	**Concentration (µg/mL)**
n-Nitrosodimethylamine	10
Surrogate Solution:	
n-Nitrosodiethylamine	10
n-Nitrosodiphethylamine	10

TABLE 8.

Working Standards for NDMA by GC/CI/MS/MS

Spike Solution:	
Compound	**Concentration (µg/mL)**
n-Nitrosodimethylamine	0.025
Surrogate Solution:	
n-Nitrosodimethylamine-d6	0.050

ATTACHMENT 1.

Organic Extraction Checklist

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

ORGANIC EXTRACTION CHECKLIST			
Batch:	**Test:**		**Date:**
	EXTRACTIONIST	WITNESS	CONCENTRATIONIST / REVIEWER
Reviewed Client Requirements			
Reviewed QC Code			
Reviewed QAS, QAPP Requirements			
Client/Program Required QA/QC Samples were performed			
The number of samples in this batch does not exceed the acceptable QC to Sample ratio for this method or project/program			
Correct Spike Used			
Correct Surrogate Used			
Mechanical pipette set at correct volume			
The correct solvents were used for this method			
Clean-up procedures used were applicable to the method			
The final sovlent and final extract volume are appropriate for the method			
NCMs written for any anomalies, hold time violations			
Handwritten initial weights, volumes, and calculation entered into LIMS verified			Reviewed by 2nd Person:
Handwritten solvent lot#s, SOP#s, and other notes entered into LIMS			Reviewed by 2nd Person:
Labels checked.			
Extracts are acceptable for delivery			

L:\QA\Edit\FORMS\Organic Prep\Organic Extraction Checklist 5/4/07

ATTACHMENT 2.

Trouble-shooting Guide

Burn-ups - *Whenever one of these things happen. Document it in an NCM, and on the benchsheet!*

- **Is the stopcock closed?** If yes, then the sample never extracted. The solvent just went up the arm, condensed, and collected in the body of the CLLE. At this point the boiling flask can be very hot because it was boiled dry, so turn off the heating mantle. It is best if you replace the boiling flask with a new empty flask with new boiling chips because the old boiling chips could be ruined. Once the new empty boiling flask is on the arm, open the stopcock and the solvent will spill over the arm and into the flask. Turn on the heating mantle and start the 18 or 24 extraction clock over.
- **Is the condenser warm?** If yes, then the extraction is most likely ruined, especially if the boiling flask was dry. If there is sample volume and hold time remaining, then the sample should be re-extracted. If there is no sample volume remaining, then get a cold condenser on the CLLE, add more solvent and continue with the extraction. If there is sample remaining, but hold time has expired we need to save this extraction because it is the only one in hold. So get a cold condenser on the CLLE, but start the re-extraction ASAP.
- **Is there a loose joint?** If yes, then the extraction is most likely ruined. Follow corrective actions for warn condenser above.
- **Was the CLLE not topped off?** If the answers to numbers 1, 2, and 3 above is no, then look at the solvent return arm. If the return arm is not full, then maybe the CLLE was never topped off and the solvent just went up the arm, condensed, and collected in the body of the CLLE. Follow corrective actions for closed stopcock above.

Flooded Arms

- If the arm is equipped with a Snyder column, the arm might have become flooded because the solvent was boiling too fast and flooded the chambers of the Snyder column. When this happens, the solvent level in the round bottom flask might actually be low because the solvent is boiling out of the flask faster than it can flow back in. If this is the case, turn down the temperature of the heating mantle.
- If the flask and arm contain only solvent, just let the extraction finish. At the end of the extraction time, close the stopcock while the heating mantle is still on. Monitor the CLLE closely as the solvent boils and collects in the body of the CLLE. When the solvent level is low enough to remove the flask, turn off the heating mantle and let it cool. Remove the flask and cap with foil. If a second extraction is needed, place an empty boiling flask on the arm and open the stopcock. The excess solvent from the first extraction will flow into the empty boiling flask. Add more solvent if needed.
- If the flask and arm contain solvent and water, turn off the heating mantle and let the boiling flask cool. If there remains enough solvent in the boiling flask, close the stopcock and boil off enough solvent so you can remove the boiling flask. Once solvent level is low enough, then turn off heating mantle and allow it to cool. Then remove boiling flask and pour the water and solvent from the flask into the CLLE body while the stopcock is closed. Re-attach the boiling flask, open the stopcock, and allow the solvent to flow into the flask. If this doesn't work, the sample and all the solvent needs to be transferred to a larger CLLE.

- If the flask and arm contains solvent and water, and the step above didn't work or there is not enough solvent left in the boiling flask to attempt the step above, turn off the heating mantle and let the boiling flask cool. Rinse a 2L separatory funnel with methylene chloride. When the boiling flask is cool, remove the condenser from the CLLE, grasp the CLLE at the body and at the joint with the boiling flask and pour the sample and the solvent into the rinsed separatory funnel. This needs to be done in a hood and you will need to have someone help you. Add a few more boiling chips to the boiling flask and place the empty CLLE back on the stand under the slot hood. Drain the methylene chloride from the separatory funnel into the body of the CLLE. Once all the methylene chloride has been transferred, continue to drain the water into the CLLE body on top of the methylene chloride. The water level in the CLLE will reach a high enough level that it will force the solvent over into the boiling flask. If the solvent level in the body of the CLLE gets too low, stop and add more solvent to the CLLE body to prevent water from going into the boiling flask. Rinse the separatory funnel with methylene chloride and add it to the CLLE.

Stuck Joints

- **Stuck Condenser** If the condenser is stuck, first close the stopcock and remove the boiling flask. Put on cut-resistant gloves. Try to loosen the joint with the cut-resistant gloves. Try both clock-wise and counter-clock-wise. If this doesn't work, heat the outside of the joint with the torch, but be sure not to keep the flame on one part of the glass, instead keep moving the flame. Watch for vapor bubbles between the glass joints as an indication the joint is loosening. Try to loosen the joint again. Repeat a few times. If heating the outside of the joint is not working, let the glass cool for about 30 minutes then come back and try again. If it is still stuck, unplug the condenser and empty the CLLE. Then soak the stuck joint in soapy water overnight.
- **Stuck Boiling Flask** If the boiling flask is stuck, first close the stopcock and remove the condenser. Put on cut-resistant gloves. Try to loosen the joint with the cut-resistant gloves. Try to loosen the joint both clock-wise and counter-clock-wise. If this doesn't work, tap the joint downward with a heavy object and try again. A plastic media-bottle cap can be used.
- **Stuck CLLE Body** If the CLLE body is stuck to the stand, close the stopcock and remove the condenser and the boiling flask. If possible, remove the plastic support from the rack with by removing the screws that hold it in place. Then lift the CLLE and the plastic support off the rack and empty the CLLE. Run hot water over the CLLE and plastic support to separate the glass from the plastic.