

SAMPLING AND ANALYSIS PLAN for the BIOREMEDIATION PILOT TEST

SITE 5

NAVAL AIR STATION JOINT RESERVE BASE WILLOW GROVE, PENNSYLVANIA



Naval Facilities Engineering Command Mid-Atlantic

Contract No. N62467-04-D-0055
Contract Task Order 411

SEPTEMBER 2008



TETRA TECH

Project-Specific SAP
Site Name/Project Name: NAS JRB Willow Grove Site 5
Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study
Revision Number: 0
Revision Date: September 2008

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

FINAL
SAMPLING AND ANALYSIS PLAN
(Field Sampling Plan and Quality Assurance Project Plan)
September 2008

Site 5 Bioremediation Pilot Study
NAS JRB Willow Grove

Prepared for:
Naval Facilities Engineering Command (NAVFAC) Mid-Atlantic

Prepared by:
Megan Ritchie / Tetra Tech NUS, Inc.
234 Mall Boulevard, Suite 260
King of Prussia, PA 19406
610-382-1527

Prepared under:
N62467-04-D-0055
CTO 411

Review Signatures: *T. E. Johnston* 10-15-08
Tom Johnston / INUS CLEAN QAM / Date
Kevin Kilmartin 10/15/08
Kevin Kilmartin / INUS PM / Date
ENG.SHERRI.
R.1229438936
Digitally signed by ENG.SHERRI.R.1229438936
DN: cn=ENG.SHERRI.R.1229438936, c=US,
o=U.S. Government, ou=DoD
Date: 2008.09.25 08:18:02 -0400

Approval Signatures: Sheri Eng / NAVFAC QAO / Date
FRYE.CURTIS.ALAN.1037277009
Digitally signed by FRYE.CURTIS.ALAN.1037277009
DN: cn=FRYE.CURTIS.ALAN.1037277009, o=U.S. Government, ou=DoD, ou=PM, ou=USNA, email=FRYE.CURTIS.
ALAN.1037277009
Date: 2008.10.03 15:56:19 -0400

Curtis Frye 10/15/08
Curtis Frye / NAVFAC RPM / Date
Lisa Cunningham 10/15/08
Lisa Cunningham / EPA RPM / Date
Charles Clark 10/15/08
Charles Clark / PADEP RPM / Date

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5
Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

EXECUTIVE SUMMARY

Tetra Tech NUS (TtNUS) has prepared this Sampling and Analysis Plan (SAP) for a pilot study for in situ bioremediation of the groundwater at Site 5 (the former Fire Training Area) at the Naval Air Station Joint Reserve Base (NAS JRB) Willow Grove, Pennsylvania. The principal contaminants associated with Site 5 soil and groundwater are volatile organic compounds (VOCs) historically disposed or spilled near the site's former drum storage area. The groundwater and soil analytical data obtained in the vicinity of the former burn ring indicate that the competent base of the tank prevented the release of the volatile liquids into the surrounding soils. Drums of liquids to be burned were temporarily stored approximately 150 feet west of the former burn ring. The groundwater and soil analytical data obtained in the vicinity of the former drum storage area indicate that volatile liquids were released into the soils and created the source area for the groundwater plume that is the subject of this pilot study.

The draft Feasibility Study (FS) for Site 5 - Fire Training Area Groundwater Operable Unit 2 (OU 2) (TtNUS, October 2004) included a remedial alternative of in situ enhanced biological anaerobic reductive dehalogenation treatment (e.g., bioremediation) and natural attenuation to promote the in situ remediation of the VOCs in groundwater. This SAP contains the technical scope of work and associated sampling plan to perform a (1) pre-remediation site characterization and (2) a pilot test (including treatability testing).

The intent of the pre-remediation characterization is to understand the source location and architecture to effectively understand and subsequently design the optimal remediation system. The purpose of the pilot test is to design, evaluate the performance and ultimately prove the applicability of a biostimulation or bioaugmentation strategy. The information derived from both aspects of this project will be used to aid in the design and installation of a full-scale biostimulation/bioaugmentation remedy. A field pilot study is imperative because the extraction and/or injection system parameters are unique to site. The data collected for both of these aspects of the project include collection of subsurface soil samples, collection of groundwater levels, and collection of groundwater samples (e.g., contaminant chemistry and geochemistry) to determine site characteristics and pilot study performance.

This SAP outlines the organization, project management and objectives, planned activities, measurement/data acquisition, assessment/oversight, and data review procedures associated with pilot study activities. This SAP specifies requirements for fieldwork related to field operations, the collection of soil samples, measurement of groundwater levels, the collection of samples from monitoring wells at Site 5, and field and laboratory analyses of soil and groundwater. This SAP includes 37 worksheets required by the Uniform Federal Policy for QAPPs (UFP-QAPP) guidance as the main body of the document. Relevant appendices (A through G) are included behind the worksheets. Tables and Figures that are not included in the 37 worksheets are provided in the back of the document. A complete list of Worksheets, appendices, tables, and figures is included in the following Table of Contents.

TABLE OF CONTENTS

Acronyms

SAP Worksheets

#1 -- Title and Approval Page.....	1
#2 -- SAP Identifying Information.....	7
#3 -- Distribution List.....	10
#4 -- Project Personnel Sign-Off Sheet.....	12
#5 -- Project Organizational Chart.....	14
#6 -- Communication Pathways.....	15
#7 -- Personnel Responsibilities and Qualifications Table.....	16
#8 -- Special Personnel Training Requirements Table.....	18
#9 -- Project Scoping Session Participants Sheet.....	19
#10 -- Problem Definition.....	20
#11 -- Project Quality Objectives/Systematic Planning Process Statements.....	35
#12 -- Measurement Performance Criteria Table.....	38
#13 -- Secondary Data Criteria and Limitations Table.....	40
#14 -- Summary of Project Tasks.....	41
#15 -- Reference Limits and Evaluation Table.....	56
#16 -- Project Schedule / Timeline Table (optional format).....	63
#17 -- Sampling Design and Rationale.....	65
#18 -- Sampling Locations and Methods/SOP Requirements Table.....	67
#19 -- Analytical SOP Requirements Table.....	75
#20 -- Field Quality Control Sample Summary Table.....	78
#21 -- Project Sampling SOP References Table.....	79
#22 -- Field Equipment Calibration, Maintenance, Testing, and Inspection Table.....	80
#23 -- Analytical SOP References Table.....	81
#24 -- Analytical Instrument Calibration Table.....	83
#25 -- Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table.....	86
#26 -- Sample Handling System.....	89
#27 -- Sample Custody Requirements Table.....	90
#28 -- QC Samples Table.....	91
#29 -- Project Documents and Records Table.....	102
#30 -- Analytical Services Table.....	103
#31 -- Planned Project Assessments Table.....	105
#32 -- Assessment Findings and Corrective Action Responses.....	106
#33 -- QA Management Reports Table.....	107
#34 -- Verification (Step I) Process Table.....	108
#35 -- Validation (Steps IIa and IIb) Process Table.....	109
#36 -- Validation (Steps IIa and IIb) Summary Table.....	110
#37 -- Usability Assessment.....	111

Tables

- Table 1-1 – Well Construction Details
- Table 1-2 – Summary of Historical Data

Figures

- Figure 1-1 – Site Location Map
- Figure 1-2 – Location Map for Current & Historical Site Features
- Figure 1-3 – Monitoring Well Locations
- Figure 1-4 – Groundwater Elevation Map Shallow Groundwater Zone
- Figure 1-5 – Groundwater Elevation Map Intermediate Groundwater Zone
- Figure 1-6 – Soil Boring and Well Locations
- Figure 1-7 – Total VOC Concentrations Shallow Groundwater Zone

Figures continued

- Figure 1-8 – Total VOC Concentrations Intermediate Groundwater Zone
- Figure 1-9 – Groundwater Detections Exceeding MCLs Summer 2005
- Figure 1-10 – Hydrogeologic Cross-Section A-A'
- Figure 1-11 – Hydrogeologic Cross-Section B-B'
- Figure 1-12 – Generalized Chlorinated Ethene and Ethane Degradation Pathways
- Figure 2-1 – Proposed Soil Boring Locations and Maximum Concentrations of Select VOCs in Historical Soil Borings
- Figure 2-2 – Proposed Monitoring Well Locations
- Figure 4-1 – Pilot System Site Plan
- Figure 4-2 – Piping and Instrumentation Diagram
- Figure 4-3 – General Schedule of Activities
- Figure 4-4 – Decision Matrix for Soil Sample Selection
- Figure 5-1 – Extraction Well Detail
- Figure 5-2 – Injection Well Detail

Appendices

- Appendix A – TtNUS SOPs**
- Appendix B – Laboratory SOPs**
- Appendix C – Historical Concentration Trends for Selected Site Wells**
- Appendix D – Material Safety Data Sheets (MSDS)**
- Appendix E – Chemical Solution Makeup Table**
- Appendix F – Estimated Process Equipment List**
- Appendix G – Daily Log Sheet**

References.....113

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5
Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study
Revision Number: 0
Revision Date: September 2008

Acronyms

°C	Degrees Centigrade
%R	Percent Recovery
ACT-POC	Activity Point of Contact
ALSI	Analytical Laboratory Services, Inc.
ARS	Air Reserve Station
CCC	Calibration Check Compound
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act of 1980
CLEAN	Comprehensive Long-term Environmental Action Navy
CLP	Contract Laboratory Program
COC	Chain of Custody
CSM	Conceptual Site Model
CT	Threshold Cycle
DCA	1,1-Dichloroethane
DCE	Dichloroethene
Dhb	<i>Dehalobactor</i>
Dhc	<i>Dehalococcoides</i>
DNAPL	Dense Non-aqueous Phase Liquid
DO	Dissolved Oxygen
DoD QSM	Department of Defense Quality Systems Manual
DON	Department of the Navy
DPT	Direct Push Technology
DQI	Data Quality Indicator
DVM	Data Validation Manager
EICP	Extracted Ion Current Profile
FOL	Field Operations Leader
FS	Feasibility Study
FTMR	Field Task Modification Request
GC/FID/TCD	Gas Chromatograph/Flame Ionization Detector/Thermal Conductivity Detector
GC/MS	Gas Chromatograph/Mass Spectrometer
GFAA	Graphite Furnace Atomic Absorption
GIS	Geographical Information System
gpm	gallons per minute
H ₃ PO ₄	Phosphoric Acid
HASP	Health and Safety Plan
HCl	Hydrochloric Acid
HDPE	High Density Polyethylene
HNO ₃	Nitric Acid
HSM	Health and Safety Manager
IC	Ion Chromatograph
ICP	Inductively Coupled Plasma
ICP-AES	Inductively Couple Plasma-Atomic Emission Spectroscopy
ICV	Initial Calibration Verification
IDL	Instrument Detection Limit
IDW	Investigation-Derived Waste
IS	Internal Standard
L	Liter
LCS	Laboratory Control Sample
MBT	Molecular Biological Tools
MCL	Maximum Contaminant Level
MDL	Method Detection Limit
MI	Microbial Insights
mL	Milliliter
MNA	Monitored Natural Attenuation
MPC	Measurement performance criteria
MSD	Matrix Spike Duplicate

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5
Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study
Revision Number: 0
Revision Date: September 2008

MS	Matrix Spike
MSDS	Material Safety Data Sheet
NA	Not Applicable
Na ₃ PO ₄	Trisodium phosphate
NaOH	Sodium Hydroxide
NAS JRB	Naval Air Station Joint Reserve Base
NAVFAC	Naval Facilities Engineering Command
NELAP	National Environmental Laboratory Accreditation Program
NFESC	Naval Facilities Engineering Service Center
NR	Not Recorded
O&M	Operations and Maintenance
ORP	Oxidation Reduction Potential
OSHA	Occupational Safety and Health Administration
OU	Operating Unit
PADEP	Pennsylvania Department of Environmental Protection
PCR	Polymerase Chain Reaction
PDF	Portable Document Format
P.G.	Professional Geologist
PID	Photoionization Detector
PM	Project Manager
PQL	Project Quantitation Limit
PQOs	Project Quality Objectives
PVC	Polyvinyl chloride
PWC-DET	Public Works Center Detachment
QA	Quality Assurance
QAM	Quality Assurance Manager
QC	Quality Control
QL	Quantitation Limit
RBC	Risk Based Concentration
RI	Remedial Investigation
RF	Response Factor
RL	Reporting Limit
ROICC	Resident Officer in Charge of Construction
RPD	Relative Percent Difference
RPM	Remedial Project Manager
RSD	Relative Standard Deviation
SAP	Sampling and Analysis Plan
SDG	Sample Delivery Group
SOP	Standard Operating Procedure
SPCC	System Performance Check Compound
SQL	Simple Query Language
SSO	Site Safety Officer
TBD	To Be Determined
TCA	1,1,1-Trichloroethane
TCE	Trichloroethene
TCL	Target Compound List
TOC	Total Organic Carbon
TtNUS	Tetra Tech NUS, Inc.
UFP-QAPP	Uniform Federal Policy for Quality Assurance Project Plans
USCS	Unified Soil Classification System
USEPA	United States Environmental Protection Agency
USGS	United States Geological Survey
U.S. Navy	United States Navy
VOC	Volatile Organic Compound
WP	Work Plan
WS	Worksheet

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

Site Name/Number: Site 5
Operable Unit: NA
Contractor Name: Tetra Tech NUS, Inc.
Contract Number: N62467-04-D-0055
Contract Title: CLEAN
Work Assignment Number: CTO 411

1. This SAP was prepared in accordance with the requirements of the *Uniform Federal Policy for Quality Assurance Plans (UFP-QAPP)* (U.S. EPA 2005) and *EPA Guidance for Quality Assurance Project Plans, EPA QA/G-5, QAMS (U.S. EPA 2002)*.

2. Identify regulatory program: CERCLA

3. This SAP is a project-specific SAP.

4. List dates of scoping sessions that were held:

Scoping Session	Date
<u>Project Planning and Field Sampling Preparation (TtNUS)</u>	<u>7/16/2007</u>
<u>Project Planning and Field Sampling Preparation (TtNUS)</u>	<u>7/24/2007</u>
<u>UFP-SAP Preparation (Navy and TtNUS)</u>	<u>10/25/2007</u>

5. List dates and titles of any SAP documents written for previous site work that are relevant to the current investigation.

Title	Date
<u>NA</u>	<u></u>
<u></u>	<u></u>

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

BRAC PMO (lead), EPA (regulatory oversight), PADEP (regulatory oversight), NAVFAC (property owner), Tetra Tech NUS (pilot study contractor)

7. Lead organization (see WS 7 for detailed list of data users)

BRAC PMO

8. If any required SAP elements or required information are not applicable to the project or are provided elsewhere, then note the omitted SAP elements and provide an explanation for their exclusion below:

NA

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

UFP-QAPP Worksheet #	Required Information	Crosswalk to Related Information
A. Project Management		
<i>Documentation</i>		
1	Title and Approval Page	Not applicable, worksheet used
2	Table of Contents SAP Identifying Information	Not applicable, worksheet used
3	Distribution List	Not applicable, worksheet used
4	Project Personnel Sign-Off Sheet	Not applicable, worksheet used
<i>Project Organization</i>		
5	Project Organizational Chart	Not applicable, worksheet used
6	Communication Pathways	Not applicable, worksheet used
7	Personnel Responsibilities and Qualifications Table	Not applicable, worksheet used
8	Special Personnel Training Requirements Table	Not applicable, worksheet used
<i>Project Planning/ Problem Definition</i>		
9	Project Planning Session Documentation (including Data Needs tables) Project Scoping Session Participants Sheet	Not applicable, worksheet used
10	Problem Definition, Site History, and Background. Site Maps (historical and present)	Not applicable, worksheet used
11	Site-Specific Project Quality Objectives	Not applicable, worksheet used
12	Measurement Performance Criteria Table	Not applicable, worksheet used
13	Sources of Secondary Data and Information Secondary Data Criteria and Limitations Table	Not applicable, worksheet used
14	Summary of Project Tasks	Not applicable, worksheet used
15	Reference Limits and Evaluation Table	Not applicable, worksheet used
16	Project Schedule/Timeline Table	Not applicable, worksheet used
B. Measurement Data Acquisition		
<i>Sampling Tasks</i>		
17	Sampling Design and Rationale	Not applicable, worksheet used
18	Sampling Locations and Methods/ SOP Requirements Table Sample Location Map(s)	Not applicable, worksheet used
19	Analytical Methods/SOP Requirements Table	Not applicable, worksheet used
20	Field Quality Control Sample Summary Table	Not applicable, worksheet used
21	Project Sampling SOP References Table Sampling SOPs	Not applicable, worksheet used
22	Field Equipment Calibration, Maintenance, Testing, and Inspection Table	Not applicable, worksheet used
<i>Analytical Tasks</i>		
23	Analytical SOPs Analytical SOP References Table	Not applicable, worksheet used
24	Analytical Instrument Calibration Table	Not applicable, worksheet used
25	Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table	Not applicable, worksheet used
<i>Sample Collection</i>		
26	Sample Handling System, Documentation Collection, Tracking, Archiving and Disposal Sample Handling Flow Diagram	Not applicable, worksheet used

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5
 Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

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

27	Sample Custody Requirements, Procedures/SOPs Sample Container Identification Example Chain-of-Custody Form and Seal	Not applicable, worksheet used
<i>Quality Control Samples</i>		
28	QC Samples Table Screening/Confirmatory Analysis Decision Tree	Not applicable, worksheet used
<i>Data Management Tasks</i>		
29	Project Documents and Records Table	Not applicable, worksheet used
30	Analytical Services Table Analytical and Data Management SOPs	Not applicable, worksheet used
C. Assessment Oversight		
31	Planned Project Assessments Table Audit Checklists	Not applicable, worksheet used
32	Assessment Findings and Corrective Action Responses Table	Not applicable, worksheet used
33	QA Management Reports Table	Not applicable, worksheet used
D. Data Review		
34	Verification (Step I) Process Table	Not applicable, worksheet used
35	Validation (Steps IIa and IIb) Process Table	Not applicable, worksheet used
36	Validation (Steps IIa and IIb) Summary Table	Not applicable, worksheet used
37	Usability Assessment	Not applicable, worksheet used

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #3 -- Distribution List[\(UFP-QAPP Manual Section 2.3.1\)](#)

Name of SAP Recipients	Title/Role	Organization	Telephone Number (Optional)	E-mail Address or Mailing Address	Document Control Number (Optional)
Curtis Frye	Remedial Project Manager (RPM)	NAVFAC Mid-Atlantic	215-897-4914	curtis.frye@navy.mil	NA
Bob Lewandowski	BRAC Environmental Coordinator	NAVFAC Mid-Atlantic	215-897-4908	robert.f.lewandowski@navy.mil	NA
Dave Barclift	Technical Staff	NAVFAC Mid-Atlantic	215-897-4913	david.barclift@navy.mil	NA
Sherri Eng	NAVFAC Chemist	NAVFAC Mid-Atlantic	410-305-2746	sherri.eng@navy.mil	NA
Harold Dusen	Environmental Director Activity Point of Contact (ACT-POC)	NAS JRB Willow Grove	215-443-6937	harold.dusen@navy.mil	NA
Lt. Commander Suzanne Montgomery	Public Works Center Detachment (PWC-DET) Coordinator	PWC-DET	215-443-2229	suzanne.montgomery@navy.mil	NA
Lisa Cunningham	EPA RPM	EPA Region 3	215-814-3363	cunningham.lisa@epamail.gov	NA
Charles Clark	PADEP Project Manager	PADEP	484-250-5731	chaclark@state.pa.us	NA
Kevin Kilmartin, P.G.	Project Manager (PM)	TtNUS	610-382-1173	kevin.kilmartin@tetrattech.com	NA
Keith Henn, P.G.	Remediation Technical Specialist	TtNUS	412-921-8146	keith.henn@tetrattech.com	NA
Tom Johnston	CLEAN Quality Assurance Manager (QAM)	TtNUS	412-921-8615	tom.johnston@tetrattech.com	NA
Vince Shickora	Field Operations Leader (FOL)/Site Safety Officer(SSO)	TtNUS	610-491-9688	Vince.Shickora@tetrattech.com	NA
Megan Ritchie	Project Chemist	TtNUS	610-491-9688	megan.ritchie@tetrattech.com	NA
Jeff Jaworski	Drilling Subcontractor	Talon Drilling	609-538-0580	Talondr1@verizon.net	NA
Susan Baer	Lab Project Manager	Analytical Laboratory Services, Inc. (ALSI)	717-944-5541	sbrunk@analyticallab.com	NA

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

Name of SAP Recipients	Title/Role	Organization	Telephone Number (Optional)	E-mail Address or Mailing Address	Document Control Number (Optional)
Rachel Whitby	Lab Project Manager	Microseeps	412-826-2389	RWhitby@microseeps.com	NA
Cheryl Davis	Lab Project Manager	Microbial Insights	865-573-8188	cdavis@microbe.com	NA

NA – Not Applicable

Project-Specific SAP
 Site Name/Project Name: NAS JRB Willow Grove Site 5
 Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study
 Revision Number: 0
 Revision Date: September 2008

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

Name	Organization/Title/Role	Telephone Number (optional)	Signature/email receipt	SAP Section Reviewed	Date SAP Read
Curtis Frye	NAVFAC RPM	215-897-4914	<i>Curtis Frye</i>	ALL	8/08
Bob Lewandowski	NAVFAC BRAC Environmental Coordinator	215-897-4908	<i>Bob Lewandowski</i>	ALL	8/08
Deve Baroff	NAVFAC Technical Staff	215-897-4913	<i>Deve Baroff</i>	WS 10, 11, 1-6, 12, 14-15	4-2008
Kevin Klimartin	TINUS PM	610-982-1173	<i>Kevin Klimartin</i>	ALL	8/08
Keith Henn	TINUS Remediation Technical Specialist	412-921-8146	ON File		
Vince Shickora	TINUS FOL/SBO	810-491-9068	<i>Vince Shickora</i>		5/08
Tom Johnston	TINUS QAM	412-921-8815	ON File		
Joe Samchuck	TINUS Data Validation Manager (DVM)	412-921-8510			
Megan Ritchie	TINUS Project Chemist	610-982-1157	<i>Megan Ritchie</i>	ALL	8/08
Jeff Jaworski	Drilling Subcontractor	609-638-0580	<i>Jeff Jaworski</i>	RFP WS 14	4/08
Merin Kirach	Geophysical Subcontractor	610-524-9486	<i>Merin Kirach</i>	RFP WS 14	5/08
Pamela John	IDW Subcontractor	610-906-0740	<i>Pamela John</i>	RFP WS 14	3/08
Dennis Sikar	Surveyor	215-268-7988	<i>Dennis Sikar</i>	RFP WS 14	7/08
Susan Beer	ALSI Lab Project Manager	717-944-5541	ON File	RFP WS 12, 13, 16, 19, 20, 21, 23-30	9/07
Rachel Whitby	Microseeps Lab Project Manager	412-926-2889	<i>Rachel Whitby</i>	RFP WS 12, 16, 18, 19, 20, 21, 23-30	9/07

Name	Organization/Title/Role	Telephone Number (optional)	Signature/email receipt	SAP Section Reviewed	Date SAP Read
Cheryl Davis	Microbial Insights Lab Project Manager	865-573-8188	<i>Cheryl L. Davis</i>	RFP 2512, 15, 18, 19, 20, 21, 23-30	9/07

ALSI – Analytical Laboratory Services, Inc.
 SSO – Site Safety Officer

FOL – Field Operations Leader
 TBD – To Be Determined

PM – Project Manager QAM – Quality Assurance Manager

Project-Specific SAP

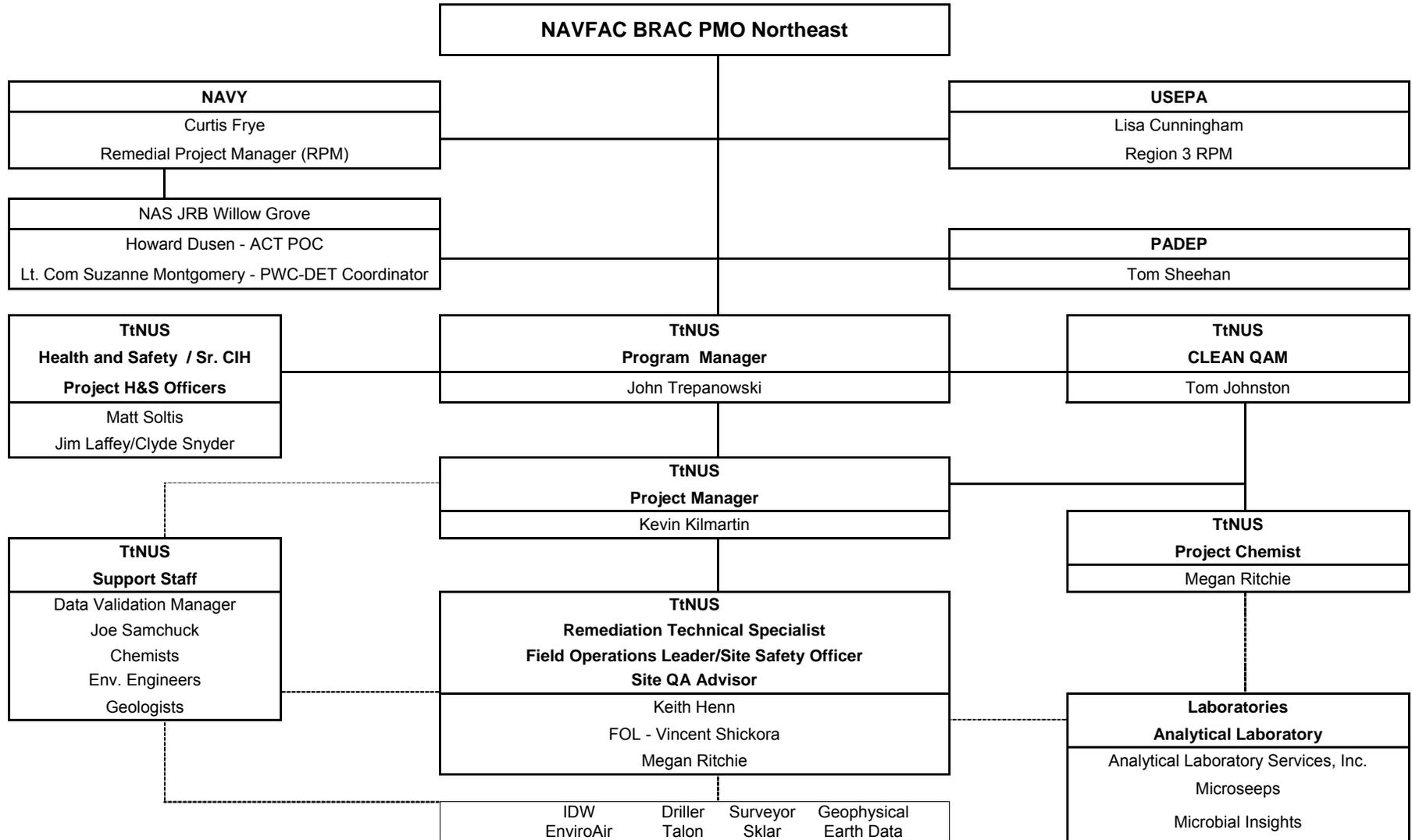
Site Name/Project Name: NAS JRB Willow Grove Site 5
 Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

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



Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5
 Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #6 -- Communication Pathways

(UFP-QAPP Manual Section 2.4.2)

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or e-mail	Procedure
Field Task Modification Requests (FTMR)	TtNUS FOL	Vince Shickora	610-491-9688	Immediately gets approval from TtNUS PM Document via FTMR form
QAPP Amendments	Navy RPM Navy Chemist	Cutis Frye Sherri Eng	215-897-4914 410-305-2746	Immediately informs TtNUS PM TtNUS documents via FTMR form Amendment is submitted to Navy Chemist for review and signature.
Changes in Schedule	TtNUS PM	Kevin Kilmartin	610-382-1173	Informs Navy via schedule impact letter as soon as impact is realized
Issues in the field that result in changes in scope of field work	TtNUS PM TtNUS Remediation Technical Specialist	Kevin Kilmartin Keith Henn, P.G.	610-382-1173 412-921-8146	FOL immediately informs PM; PM immediately informs RPM; RPM issues scope change if warranted; Scope change to be implemented before work is executed.
Recommendations to stop work and initiate work upon corrective action	TtNUS FOL TtNUS PM TtNUS QAM TtNUS HSM Navy RPM	Vince Shickora Kevin Kilmartin Tom Johnston Matt Soltis Curtis Frye	610-491-9688 610-382-1173 412-921-8615 412-921-8912 215-897-4914	Responsible Party immediately informs subcontractors, the Navy, and Project Team
Analytical data quality issues	ALSI Microseeps Microbial Insights TtNUS Project Chemist	Scott Brunk Rachel Whitby Cheryl Davis Megan Ritchie	717-944-5541 412-826-2389 865-573-8188 610-382-1527	Immediately notify TtNUS Project Chemist Notify Data Validation Staff and TtNUS PM if necessary

ALSI – Analytical Laboratory Services, Inc.
 PM – Project Manager

FOL – Field Operations Leader FTMR – Field Task Modification Request
 QAM – Quality Assurance Manager RPM – Remedial Project Manager

HSM – Health and Safety Manager
 TBD – To Be Determined

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #7 -- Personnel Responsibilities and Qualifications Table[\(UFP-QAPP Manual Section 2.4.3\)](#)

Name	Title/Role	Organizational Affiliation	Responsibilities	Education and/or Experience Qualifications (Optional)
Curt Frye	Navy RPM	NAVFAC	Oversees project, financial, and schedule, of the project.	
Sherri Eng	Navy QAO	NAVFAC	Reviews project SAP. Ensures quality aspects of the Navy and UFP-QAPP guidance.	
Kevin Kilmartin	PM	TtNUS	Oversees project, financial, schedule, and technical day to day management of the project.	M.S. Geology, 13 years of project management experience and 27 years of geological experience
Keith Henn	Remediation Technical Specialist	TtNUS	Development of the pilot study work plan, implements the plan, conducts senior evaluation of the data, and is the lead technical author of the completion report	B. S. Geology, M.S. Hydrogeology, more than 14 years of environmental experience and author of over 35 technical presentations and papers documenting innovative investigation and in situ remediation applications.
Vincent Shickora	FOL, SSO	TtNUS	Supervises, coordinates, and performs field sampling activities	
Tom Johnston	QAM	TtNUS	Reviews QAPP and data quality determination. Ensures quality aspects of the TtNUS NAVFAC Atlantic CLEAN program.	PhD Chemistry, 30 years environmental experience as technical and quality specialist.
Joe Samchuck	DVM	TtNUS	Quality assurance of data validation deliverables. Prepares lab scope, coordinates with lab, and data quality review.	B.S. Chemistry, MBA, M.S. Finance, 23 years environmental experience
Megan Ritchie	Project Chemist	TtNUS	Coordinates analyses with lab chemists, ensures the scope is followed, QA data packages, communicates with TtNUS staff.	B.S. Biology/Environmental Studies, 10 years environmental experience

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

Name	Title/Role	Organizational Affiliation	Responsibilities	Education and/or Experience Qualifications (Optional)
Matt Soltis	Health and Safety Manager (HSM)	TtNUS	Oversees CLEAN Program Health and Safety Program	B.S. Industrial Safety Sciences, 24 years of environmental experience
Jeff Jaworski	Driller	Talon Drilling	Drills boreholes for well construction under supervision of the TtNUS FOL. May also conduct Geoprobe borings.	NA
Martin Kirsch	Geophysical Subcontractor	Earth Data	Conducts geophysical borehole logging of all newly-drilled boreholes.	NA
Pamela John	IDW Subcontractor	Enviro Air	Analyzes and disposes of investigative derived waste from the pilot study operations.	NA
Dennis Sklar	Surveyor	Dennis W. Sklar, Inc.	Determines horizontal coordinates and vertical elevations for sampling locations and well locations.	NA
Susan Baer	Lab Project Manager	ALSI	Ensures laboratory analyzes and produces deliverables according to contract with TtNUS.	NA
Rachel Whitby	Lab Project Manager	Microseeps	Ensures laboratory analyzes and produces deliverables according to contract with TtNUS.	NA
Cheryl Davis	Lab Project Manager	Microbial Insights	Ensures laboratory analyzes and produces deliverables according to contract with TtNUS.	NA

CLEAN – Comprehensive Long-term Environmental Action Navy

FOL – Field Operations Leader

HSM – Health and Safety Manager

PM – Project Manager

QAM – Quality Assurance Manager

SSO – Site Safety Officer

TBD – To Be Determined

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #8 -- Special Personnel Training Requirements Table

[\(UFP-QAPP Manual Section 2.4.4\)](#)

The following table is used to identify and describe any specialized/non-routine project specific training requirements or certifications needed by personnel in order to successfully complete the project or task. OPNAV 5090.1 instructions are not considered specialized training; the OPNAV training requirements represent routine, minimum requirements that are mandatory for all Department of Navy (DON) projects.

No specialized training is required for this project. Each site worker will be required to have completed a 40-hour course (and 8-hour refresher, if applicable) in Health and Safety Training as described under Occupational Safety and Health Administration (OSHA) 29 CFR 1910.120(b)(4). Additional information about Health and Safety Training can be found in the site-specific Health and Safety Plan (HASP).

Analytical Laboratory Services, Inc (ALSI) has successfully completed the laboratory evaluation process required as part of the Naval Facilities Engineering Service Center (NFESC) Quality Assurance Program and described in the Department of Defense Quality Systems Manual (DoD QSM) (January 2006) and is additionally certified by the National Environmental Laboratory Accreditation Program (NELAP), which is the recognized certifying authority for the state of Pennsylvania. ALSI's last Navy audit was conducted on November 28 through 30, 2007. The Navy determined that ALSI's Corrective Action Plan (CAP) was acceptable, and ALSI is presently in the process of submitting the documentation supporting the CAP. The Navy has extended ALSI's certification through February of 2008, until the new certification is issued. The Navy certification letter is included in Appendix B.

The selected laboratories for the indicator parameters, Microseeps and Microbial Insights, do not have Navy certification. The methods performed by these laboratories are not audited because there are not approved EPA methods for these analyses. The methods for these analyses are either laboratory-specific or proprietary methods, and are not widely available from other laboratory sources. Microbial Insights PCR method is a proprietary method that was reviewed by Navy personnel Cliff Casey in 2005. Although, this was not an official Navy audit, Microbial Insights responded to Tetra Tech that the Navy did not express any concerns with the performance of the method. Microseeps did not indicate that they have similarly been subject to an unofficial Navy audit, but Tetra Tech has used this laboratory for similar projects and has not encountered problems. In addition, the analyses to be provided by Microbial Insights and Microseeps will essentially provide screening-level information (the viability of the bacterial community). The ultimate-decision making parameters (the concentrations of the various chemical compounds) will be performed by ALSI, which is Navy-certified.

SAP Worksheet #9 -- Project Scoping Session Participants Sheet
 (UFP-QAPP Manual Section 2.5.1) *Data needs tables were not used for these scoping sessions.

Project Name: NAS JRB Willow Grove Site 5 Projected Date(s) of Sampling: 11/12/2007 through 12/1/2009 Project Manager: Kevin Kilmartin	Site Name: NAS JRB Willow Grove Site Location: Willow Grove, Pennsylvania
--	--

Date of Session: July 16, 2007
Scoping Session Purpose: Project Planning and Field Sampling Preparation

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Kevin Kilmartin	PM	TtNUS	610-382-1173	kevin.kilmartin@tetrattech.com	Management
Keith Henn	Remediation Technical Specialist	TtNUS	412-921-8156	keith.henn@tetrattech.com	Task Management
Megan Ritchie	Project Chemist	TtNUS	610-382-1527	megan.ritchie@tetrattech.com	Chemist

Comments/Decisions: Discussed project activities. Distribute assignments to team members.
 Action Items: Kevin and Keith write work plans for Site 5 Bioremediation Pilot Study. Megan will begin preliminary QAPP writing. Team will begin thinking about project quality objectives (PQOs) which will be discussed at next session.
 Consensus Decisions: Projected internal draft deliverable will go out August 15, 2007.

Date of Session: July 24, 2007
Scoping Session Purpose: Project Planning and Field Sampling Preparation

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Kevin Kilmartin	PM	TtNUS	610-382-1173	kevin.kilmartin@tetrattech.com	Management
Keith Henn	Remediation Technical Specialist	TtNUS	412-921-8156	keith.henn@tetrattech.com	Task Management
Megan Ritchie	Project Chemist	TtNUS	610-382-1527	Megan.Ritchie@tetrattech.com	Chemist

Comments/Decisions: No tables will be repeated to eliminate errors. QAPP will refer to Tables in Work Plan.
 Action Items: Complete assigned portions of Work Plan/QAPP.

Date of Session: October 25, 2007
Scoping Session Purpose: Discuss Comments for Internal Draft

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Curt Frye	RPM	Navy	215-897-4914	curtis.frye@navy.mil	Navy PM
Bob Lewandowski	BRAC Environmental Coordinator	Navy	215-897-4908	robert.f.lewandowski@navy.mil	Management
Dave Barclift	Technical Staff	Navy	215-897-4913	david.barclift@navy.mil	SAP Reviewer
Kevin Kilmartin	PM	TtNUS	610-382-1173	kevin.kilmartin@tetrattech.com	Management
Russ Turner	PM	TtNUS	610-382-1534	russ.turner@tetrattech.com	Management
Don Whalen	Geologist	TtNUS	610-382-1536	don.whalen@tetrattech.com	Geologist
Megan Ritchie	Project Chemist	TtNUS	610-382-1527	megan.ritchie@tetrattech.com	Chemist

Consensus Decisions: TtNUS will rename QAPP to SAP and reorganize according to UFP-SAP template. Incorporate former WP into SAP.

SAP Worksheet #10 -- Problem Definition ([UFP-QAPP Manual Section 2.5.2](#))

10.1 INTRODUCTION AND PURPOSE

TtNUS has prepared this SAP for a pilot study for in situ bioremediation of the groundwater at Site 5 (the former Fire Training Area) at the Naval Air Station Joint Reserve Base (NAS JRB) Willow Grove, Pennsylvania. This work will be performed under Contract Task Order No. 411 under Contract N62467-04-D-0055, Comprehensive Long-Term Environmental Action – Navy (CLEAN).

The principal contaminants associated with Site 5 soil and groundwater are VOCs historically disposed or spilled near the site's former drum storage area. For decision making purposes under Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), the Site 5 soil has been designated Operable Unit 4 (OU 4) and the Site 5 groundwater has been designated OU 2. The pertinent analytical results of the remedial investigation work conducted to date for these media are included in the Remedial Investigation Report for Site 5 – Fire Training Area (TtNUS, February 2002), the Site 5 RI Addendum 2, Soil Investigation for Volatile Organic Compound, Soil to Groundwater Impact, Site 5 - Fire Training Area (TtNUS, March 2006), and the Site 5 Remedial Investigation Addendum - Fire Training Area Groundwater (OU 2) (TtNUS, September 2006).

The Navy completed a draft FS for Site 5 - Fire Training Area Groundwater (OU 2) (TtNUS, October 2004) that is currently being finalized. The FS included a remedial alternative of in situ enhanced biological anaerobic reductive dehalogenation treatment (e.g., bioremediation) and natural attenuation to promote the in situ remediation of the VOCs in groundwater. This SAP contains the technical scope of work to perform a bioremediation pilot test to evaluate the potential efficacy of this remedial alternative.

Prior to the implementation of this pilot test additional data collection is necessary to further develop the conceptual site model (CSM) to adequately design the pilot study, and ultimately determine the effectiveness of in situ bioremediation. Thus, this SAP also contains the scope of work to perform a pre-treatability investigation (which will be referred to as the Phase I investigation) that will include additional soil sampling and chemical analysis, the installation of monitoring wells with groundwater sampling and analyses, and aquifer testing. The soil investigation of the source area is planned to evaluate the nature and volume of residual source in the unconsolidated soil (versus the underlying bedrock), which will determine if these soils are a continuing source of VOCs to the groundwater plume. If a significant continuing source is encountered it may be removed prior to groundwater treatment implementation. Additional monitoring wells and groundwater analyses will determine the current groundwater quality (VOCs, natural attenuation parameters, and endemic bacteria population) in the pilot test area. Aquifer testing is planned to better understand the site-specific hydraulic parameters of the fractured bedrock. The pilot test design and actual implementation (which will be referred to as the Phase II investigation) are expected to be modified based upon this additional data collection and subsequent evaluation.

10.2 SCOPE AND OBJECTIVE

The objective of the pilot study is to evaluate the applicability of the in situ bioremediation strategy, and to provide the data to aid in the design, installation, and operational parameters for a full-scale recirculation system at the site. The scope of work includes the following basic elements:

- The objective of the pre-treatability soil investigation is to evaluate the lateral and vertical extent of VOC contamination of the soils at the source area. The scope of work for this task includes the drilling of soil borings and the sampling and analysis of the subsurface soils for VOCs.

SAP Worksheet #10 -- Problem Definition ([UFP-QAPP Manual Section 2.5.2](#))

- New monitoring wells will be installed to (1) better understand the nature of the groundwater contamination, and (2) provide the groundwater analytical data needed to evaluate bioremediation efficacy within the source area.
- A short-term aquifer pumping test will be performed to determine the hydraulic properties of the aquifer.
- Injection and extraction wells will be installed for use in the proposed pilot study bioremediation groundwater recirculation system.
- Biostimulants and (potentially) microbial inoculum will be injected into the aquifer via the groundwater recirculation system to stimulate anaerobic reductive dechlorination (i.e., biological destruction) of the VOCs.
- Periodic well sampling and analysis will be performed to monitor and record conditions within the aquifer, determine the optimal quantity and frequency of injected material, and to measure the performance of the bioremediation processes during the testing period.
- Periodic and routine operations and maintenance (O&M) activities (including the injection of additional nutrient, biological, or other amendments as needed) will be performed.

10.3 SITE BACKGROUND

NAS JRB Willow Grove, Pennsylvania is located in Horsham Township, Montgomery County in southeastern Pennsylvania, approximately 20 miles north of the city of Philadelphia (Figure 1-1). The Navy Base occupies approximately 1,000 acres of approximately 1,200 acres the Department of Defense (DoD) maintains at the Air Station. The Willow Grove Air Reserve Station (ARS) of the Air Force occupies approximately 200 acres of land in the northeastern section of the Air Station and shares common facilities with the NAS JRB. The Air Station is comprised of flat to slightly rolling terrain and is generally bounded by State Route 611 to the east, State Route 463 to the southwest, and Keith Valley Road to the north.

The primary mission of NAS JRB Willow Grove is to provide support for operations involving aviation activities and to train Navy reservists. NAS JRB Willow Grove supports DoD tenants such as the Marine Reserves, Pennsylvania Air National Guard, the Air Force Reserve, and the Army Reserve. The Base provides facilities, services, materials, and training in direct support of all assigned units. These units include anti-submarine warfare squadrons, a helicopter squadron, a fleet logistic support squadron, and other Navy and Marine units.

NAS JRB Willow Grove was targeted for closure by the Base Realignment and Closure (BRAC 2005) Commission in recommendations that became law on November 9, 2005. The Base is slated to close by the year 2011.

The former fire training area (Site 5) is located in the south-central portion of NAS JRB, approximately mid way between runway 10/28 and State Route 463, and covers an irregularly shaped area of approximately 1.25 acres. Fire training operations included storage and burning of flammable liquid wastes generated by the air station in the period from 1942 through 1975, when burning exercises ceased. As a result of the historical storage and burning operations, soil and groundwater have been impacted.

10.4 SITE DESCRIPTION AND SETTING

The burning area at Site 5 was located in the south-central portion of the site (Figure 1-2). The burn ring (which has been removed) consisted of a cylindrical steel tank that was cut, placed on its side, and partially buried to form an open steel ring at the surface with a competent steel bottom at the Base. The groundwater and soil analytical data obtained in the vicinity of the burn ring indicate that the competent base

SAP Worksheet #10 -- Problem Definition ([UFP-QAPP Manual Section 2.5.2](#))

of the tank prevented the release of the volatile liquids into the surrounding soils. Drums of liquids to be burned were temporarily stored approximately 150 feet west of the burn ring. The groundwater and soil analytical data obtained in the vicinity of the former drum storage area indicate that volatile liquids were released into the soils and created the source area for the groundwater plume that is the subject of this pilot study.

Site 5 is primarily covered by grasses, with some woody and brushy vegetation present within the southern portion of the area. The ground surface slopes toward the south at a grade of approximately two percent. The ground surface in the vicinity of the former drum storage area is partially covered by a paved asphalt access road that extends northward to the taxiway.

A total of 33 monitoring wells have been installed at Site 5 during the multiple phases of the RI investigation to delineate the directions of groundwater flow and the nature and horizontal and vertical extent of groundwater contamination. In addition, two monitoring wells installed at Site 3 (located hydraulically downgradient from Site 5) are incorporated into the Site 5 investigation because they are useful for defining the approximate downgradient extent of the groundwater plume. The monitoring well locations are illustrated in Figure 1-3, and their construction details are listed in Table 1-1.

10.4.1 Hydrology

The former fire training area is situated atop a southwest-northeast-trending ridge that is the highest topographic feature within the region. This ridge serves as a divide for the regional surface water bodies (watershed divide); surface water to the north of the divide flows toward the Little Neshaminy Creek, and surface water to the south of the divide flows toward the Pennypack Creek.

The ground surface in the vicinity of the former Fire Training Area slopes toward the south at a grade of approximately two percent. Runoff during normal precipitation events is minimized by the relatively gentle slope and the abundant vegetation, which serves to decrease runoff velocity and increase infiltration.

Based on the local topography, any runoff from the site area may be expected to flow off Base through a small intermittent drainage ditch that crosses the Base boundary approximately 2,000 feet south of the Fire Training Area. This drainage way, which also carries runoff from the Antenna Field Landfill, flows into Pennypack Creek approximately 3,000 feet from the Base property line.

Two small ponds are located within 100 feet south of the site in the down slope direction. The two small ponds do not always contain water and tend to dry out.

10.4.2 Geology

Soil borings drilled throughout the site encountered a variably thick overburden layer underlain by weathered bedrock of siltstone and sandstone. The overburden generally consists of silty clay to clayey silt, with minor amounts of silty sand. The thickness of the overburden (or the depth to the top of the weathered bedrock) ranges from about 7 to 19 feet.

The bedrock consists of the middle arkose member of the Triassic-age Stockton Formation, which locally is about 5,000 feet thick. The bedrock underlying the site to the current total depth of investigation (261 feet) is characterized by a generally coarse-grained lithology that consists of alternating sequences of siltstone and sandstone. Thin but laterally persistent beds of finer-grained mudstone are located within the lower portions of the monitored section.

The correlation of geophysical logs from Site 5 boreholes indicates that the bedrock strikes at North 76° East and dips at 7° Northwest, which is consistent with the attitude predicted by the regional geology.

SAP Worksheet #10 -- Problem Definition ([UFP-QAPP Manual Section 2.5.2](#))

10.4.3 Hydrogeology

Investigations conducted to date have consistently indicated that the soils beneath the former drum storage area occur entirely within the vadose zone, and that the water table at this location is encountered within the shallow, weathered bedrock at subsurface depths of about 20 to 25 feet. Although significant amounts of groundwater may be held in storage within the primary porosity of the fine- to medium-grained sandstones, groundwater migration throughout the site is dominantly through the secondary porosity created by fractures and joints and along bedding-plane partings.

The nature of the local groundwater flow is relatively complex. The site is underlain by a local groundwater divide within the shallow groundwater zone (near the water table), and a deeper, regional groundwater divide. The orientations and locations of the two groundwater divides do not coincide, resulting in multidirectional groundwater flow that varies with depth. The shallow groundwater divide is oriented east-west and generally coincides with the location and orientation of the Base runway, which is the highest local topographic feature. The regional groundwater divide is oriented southwest-northeast and closely coincides with the regional surface water divide for the Little Neshaminy Creek and Pennypack Creek watersheds.

The horizontal hydraulic gradients throughout the site are very low. For the shallow zone, the horizontal hydraulic gradient ranges from about 0.01 ft/ft to 0.001 ft/ft (depending on topographic location), and for the intermediate zone, the horizontal hydraulic gradient is about 0.001 ft/ft. Consistent with its position on a regional groundwater divide, the overall vertical hydraulic gradient at Site 5 is oriented downward, although borehole flow logs indicate that for any particular borehole, the vertical flow may be upward, downward, or both.

The groundwater flow beneath Site 5 is illustrated by groundwater elevation contour maps drawn for the shallow (Figure 1-4) and intermediate (Figure 1-5) groundwater zones. Site 5 is located south of the local shallow water divide, so groundwater at the water table and in the shallow groundwater zone (which is most directly influenced by surface topography) flows in a generally southward direction. Site 5 is also located just west of the regional groundwater divide, so groundwater in the deeper groundwater zones flows in a generally northwestward direction, consistent with the regional groundwater flow interpretation.

Groundwater flow within the Stockton Formation is markedly anisotropic, with the main direction of anisotropy oriented parallel to the strike of the bedrock. In anisotropic aquifers, the general groundwater flow direction is typically in a direction (or angle) between the direction of steepest hydraulic gradient and the dominant direction of anisotropy. This relationship is confirmed at Site 5 by the orientation of the VOC plumes (see Section 10.5.2), where the VOCs function as "tracers" indicating the site-specific, actual groundwater flow direction.

10.5 NATURE AND EXTENT OF CONTAMINATION

10.5.1 Soil

The full nature and extent of all chemical parameters (including the VOCs) in the Site 5 soils are documented and discussed in the Remedial Investigation (RI) report (TtNUS, February 2002). Additional confirmation sampling for VOCs and an investigation of the soil-to-groundwater migration potential of the VOCs is documented in a subsequent investigation (TtNUS, March 2006). The results of these investigations indicate that although the VOC concentrations in soil do not create unacceptable risks, some Site 5 soils exceed the EPA and Pennsylvania Department of Environmental Protection (PADEP) generic screening levels for the soil-to-groundwater migration pathway. In addition, the United States Geological Survey (USGS) drilled and sampled a shallow bedrock core near monitoring well 05MW01S and determined that residual VOC contamination exists in the shallow bedrock matrix beneath the drum staging area (USGS 2002).

SAP Worksheet #10 -- Problem Definition ([UFP-QAPP Manual Section 2.5.2](#))

The maximum concentrations of select VOCs in Site 5 soil borings are illustrated in Figure 1-6. VOCs are generally absent or occur in low concentrations throughout most of the site with the exception of the former drum storage area, where elevated concentrations of multiple VOCs have been detected. Most of the higher concentrations are detected in the uppermost 10 feet of soil, but concentrations of some VOCs exceeding the generic soil-to-groundwater screening levels have been detected as deep as the soil/bedrock interface (TtNUS, March 2006).

10.5.2 Groundwater

The full nature and extent of all chemical parameters (including the VOCs) in the Site 5 groundwater are documented and discussed in the RI report (TtNUS, February 2002). Additional groundwater investigation (including the resampling of existing wells and the installation and sampling of new wells) is documented and discussed in the Site 5 RI Addendum 5 (TtNUS, September 2006). The results of these investigations indicate that VOCs are the primary compounds impacting the Site 5 groundwater, and that the groundwater must be remediated because multiple VOCs occur at concentrations that create unacceptable risk.

Current Conditions

The current extent of the groundwater plume using the most recent data (Summer 2005) is illustrated in Figure 1-7 (shallow groundwater) and Figure 1-8 (intermediate groundwater). The VOC concentrations from this sampling round are summarized in Table 1-2, and the concentrations exceeding Federal Maximum Contaminant Levels (MCLs) are illustrated in Figure 1-9. The 3-dimensional extent of the plume is illustrated by a pair of hydrogeologic cross-sections in Figure 1-10 and Figure 1-11. Note that these two cross-sections should not be directly compared because they were constructed using two different sets of analytical data obtained several years apart, but the geometries of the plume can be used to illustrate how both the nature and extent of the plume vary with both the distance and direction from the source area.

The highest VOC concentrations at the water table are detected at the drum storage area (source area) near monitoring well cluster 05MW01. The migration pathway of the dissolved-phase plume emanating from the source area is consistent with the groundwater flow directions. Within the shallower depths of the aquifer, the plume flows in a generally southward direction, as it also migrates downward within the aquifer. As a result, the highest VOC concentrations within the intermediate depths of the aquifer (about 100 to 150 feet) are detected near monitoring well clusters 05MW09 and 05MW10. By this depth, however, the plume is also influenced by the regional groundwater divide and the resultant shift in the groundwater flow direction to the west to northwest. Therefore, the direction of plume migration within the intermediate depth of the aquifer also shifts toward the west-northwest, or parallel to the direction of groundwater flow.

Monitoring well cluster 03MW08 (a shallow well and a deep well) was installed as an upgradient monitoring well cluster for Site 3 (the 9th Street Landfill). However, the hydraulic head data obtained from these wells also indicates that the cluster is located nearly directly downgradient from Site 5. At 03MW08, no VOCs were detected in the shallow well, and a total VOC concentration of 14.8 ug/L (with no individual detections above their MCL) was detected in the deep well. The VOC concentrations and their vertical position within the aquifer are consistent with the conclusion that although 03MW08 is a Site 3 well cluster, it is also located in an appropriate position to identify and monitor the downgradient edge of the Site 5 groundwater plume, and to determine whether the Site 5 plume is expanding, extracting, or is at steady state. Although this well cluster does not have an extensive sampling history, the historical data from other Site 5 well clusters (see next subsection) suggest that the plume is decreasing in magnitude (absolute VOC concentrations), if not yet in areal extent.

SAP Worksheet #10 -- Problem Definition

([UFP-QAPP Manual Section 2.5.2](#))

Monitoring wells at locations 05MW14 and 05MW15 were installed along bedrock strike from the source area to investigate if the strike of the bedrock was a preferred avenue for groundwater and plume migration. The lower concentrations of VOCs at these locations relative to 05MW04 and 05MW13 indicate that under ambient conditions, the strike of the bedrock is not exerting a major structural control on the migration of the plume.

Monitoring well cluster 05MW12 was installed directly down dip from the source area at the drum staging area. The shallow well at 05MW12 was screened across the same stratigraphic interval that is monitored by 05MW01S (which is located at the source area and is the site's most highly impacted well) to investigate if the dip of the bedrock was a preferred avenue for contaminant migration, particularly if non-aqueous (DNAPL) solvent phase ever existed. The low concentration of total VOCs at 05MW12S indicates that the dip of the bedrock is not exerting a major structural control on the migration of the plume.

As illustrated in the hydrogeologic cross-sections, the "bottom" of the plume has not been defined in the sense of detecting unimpacted groundwater below the plume. However, the vertical center, or core of the plume has been well defined by the delineation of much lower VOC concentrations in the deeper groundwater that is below the zone of the most highly-impacted groundwater.

Historical Trends

The analytical results from historical sampling rounds are documented and discussed in the RI report (TtNUS, February 2002) and the Site 5 RI Addendum 5 (TtNUS, September 2006). Four full sampling rounds of Site 5 monitoring wells have been conducted since the onset of remedial investigation activities (1991, 1997, 2000, and 2005), although some wells have also been sampled at other times for various reasons.

The historical groundwater concentrations for the most highly impacted wells (05MW01S, 05MW01SI, 05MW09SI, and 05MW10SI) are illustrated in Appendix C. As discussed, these wells monitor the plume's highest VOC concentrations at the source area and immediately downgradient of the source area. The identification of trends is difficult due to the limited sampling frequency. In addition, the limited data prevent the analysis or identification of seasonal variability, so it is possible that the trends exhibited by the existing data are not representative of the true historical trends in VOC concentrations. Overall, the graphs generally indicate an overall decline in the primary VOCs such as trichloroethene (TCE) and 1,1,1-trichloroethane (TCA), and relatively constant concentrations of the compounds that are generated through the reductive dehalogenation of these compounds (daughter products, e.g., cis-1,2-dichloroethene [DCE] and 1,1-dichloroethane [DCA]), as illustrated in the generalized degradation pathways on Figure 1-10. In many cases, a decrease in the parent compounds with respect to a proportional increase in the daughter products is primary evidence of biological degradation under natural attenuation. For example, the concentrations of compounds found in nearly every well listed in Appendix C illustrates a decrease in 1,1,1-TCA concentration with respect to an increase in 1,1-DCA concentration over time. In fewer cases (e.g., 05MW01S, 05MW10SI) a decrease in TCE concentration is accompanied by an increase in the concentration of cis-1,2-DCE. This apparent lack of TCE degradation may be caused by the fact that the presence of 1,1,1-TCA has been shown to inhibit TCE degradation, particularly at the high historical concentrations of 1,1,1-TCA detected near the source area (05MW01S).

The degree of natural biological degradation currently occurring within the Site 5 VOC plume has been investigated during previous sampling events by determining and measuring the parameters that represent secondary lines of evidence of biological degradation. These results are summarized and discussed in the Site 5 RI Addendum 5 (TtNUS, September 2006), and the data summary table from that report (which includes the complete list of natural attenuation parameters collected for this site) is included as Table 1-2. The very low to non-detected concentrations of ethene, ethane, and methane (plus the absence of vinyl chloride in the historical VOC sampling rounds) indicate that the current reductive dehalogenation process within the plume either is not complete or is occurring at a very low

SAP Worksheet #10 -- Problem Definition ([UFP-QAPP Manual Section 2.5.2](#))

rate. The highest concentrations of ethane and ethene have been detected at 05MW01S, which is also the well with the highest VOC concentrations. Low aerobic to moderately reducing conditions are also present in the more highly contaminated wells, as supported by generally low dissolved oxygen (DO) concentrations (>0.5 mg/L of DO), depleted sulfate concentration with respect to background, and excessive carbon dioxide and alkalinity with respect to background. Despite these less than favorable conditions (including low total organic carbon [TOC], low pH, and likely mixed redox conditions), the data suggest that low to moderate reductive dechlorination is occurring in the highly contaminated locations, and that much less biological degradation is occurring in areas of low TOC.

As will be discussed in Worksheet 14, additional parameters and microbial populations will be collected during the Phase I investigation that will add additional insight to this analysis, but these data have not been collected in the past sampling rounds and are not yet available. It is anticipated that the limited TOC and acidic pH conditions of the groundwater are the likely factors that are limiting the biological activity at Site 5. Both of these conditions can be addressed and adjusted during the pilot study to create more favorable conditions for biological degradation.

10.6 PILOT STUDY RATIONALE

This section describes the rationale used to ensure the data from the pilot study is beneficial. In this pilot study, sodium lactate, sodium carbonate, and/or sodium bicarbonate will be injected into the subsurface to create favorable conditions to stimulate bioremediation of the contaminant plume. A microbial inoculum, including but not limited to *Dehalococcoides* (Dhc) and *Dehalobacter* (Dhb), may be injected to augment the microbial population if it is determined to be required and appropriate.

As noted in Section 10.1, additional data collection is required to further refine the site CSM prior to the implementation of the pilot test. The additional soil and groundwater chemical analysis and the aquifer

testing will be used to refine the pilot test design and implementation. For example, if the results of the aquifer testing indicate that the currently proposed rationale and methodology for the introduction of materials into the aquifer is invalid, then an alternative delivery method may need to be evaluated.

10.7 RECIRCULATION SYSTEM LAYOUT

A recirculation system was selected for Site 5 because it enables the natural flow rate of groundwater to be increased, enhances the distribution of amendments (by way of increased groundwater velocity and radius of influence), and increases the overall rate of bioremediation. It is a cost effective method in areas such as Site 5, where the hydraulic gradients and groundwater flow rates are low. The pilot system site plan and study area are illustrated in Figure 4-1, which includes the locations of existing wells, proposed injection wells, proposed extraction wells, and proposed new monitoring wells. It must be emphasized that some of the proposed locations illustrated in the site plan may be modified to accommodate existing field conditions or be adjusted as a result of the Phase I (pre-treatability study) investigation.

Changes to the well locations will be executed through the communication pathway defined in SAP Worksheet 6, under the Communication Driver "Issues in the field that result in changes in the scope of work." The TtNUS PM will immediately notify the Navy RPM of the need to change a well location. The Navy RPM will evaluate the change and decide whether the new location is a field task modification, or if a scope change is warranted. If warranted, the scope change will be implemented before the work is executed. The Navy RPM will also evaluate whether the significance of the change warrants consultation with EPA and PADEP prior to the execution of the change, or if subsequent notification is sufficient.

SAP Worksheet #10 -- Problem Definition ([UFP-QAPP Manual Section 2.5.2](#))

10.7.1 Injection / Extraction Wells

The recirculation system will include two injection wells to be installed immediately upgradient of the source area and two extraction wells to be installed downgradient of the source area. The extracted groundwater will be transferred via underground pipe to a temporary trailer-mounted feed system and reintroduced into the aquifer through the injection wells.

10.7.2 Recirculation Pumping Rate and Groundwater Zone of Capture

The groundwater pumping rate and the estimated zone of capture of the recirculation system will be determined after the results of the Phase I aquifer test are analyzed. However, based upon the currently available monitoring well purging and sampling data, it is estimated that a cumulative pumping rate of between 2 to 5 gallons per minute (gpm) may be possible. The analysis of the aquifer test will also include an estimation of the number of pore volumes that will be "flushed" at least once during the pilot study.

10.8 RECIRCULATION SYSTEM COMPONENTS

The injection products sodium lactate, sodium bicarbonate, sodium carbonate, and the microbial innoculum, will not exceed any of the drinking water standards listed in Pennsylvania. These products will be recirculated with the site groundwater, which will be of the same water quality as the current groundwater. The Material Safety Data Sheets (MSDS) for sodium lactate, sodium bicarbonate, sodium carbonate and a general microbial innoculum are included in the Appendix D.

10.8.1 Chemical Amendments

The amount of chemical amendments to be injected will be determined by the additional site data collected during the initial sampling events, including the characterization data and the geochemical baseline conditions. As illustrated in Figure 4-2, sodium lactate and sodium carbonate/bicarbonate will be proportionally added to the extracted groundwater, and this solution will be continuously injected into the pilot study area via the recirculation system. The anticipated proportions of these materials are summarized in the chemical makeup table included as Appendix E.

Sodium Lactate: The sodium lactate will be added to the recirculation system incrementally because the amount to ultimately be required is uncertain. The volume of sodium lactate to be added will be based on the Oxidation-Reduction Potential (ORP) within the pilot study area and/or the presence or absence of TCE daughter products. During the initial injection, a continuous-feed pump will introduce sodium lactate at a feed concentration of approximately 1 pound per gallon into the groundwater injection lines. As the subsurface conditions are monitored and the collected data are interpreted, periodic decisions will be made regarding the volume (if any) of additional sodium lactate to be added to the system.

Sodium Carbonate/Bicarbonate: The need for sodium carbonate and/or bicarbonate is based on the very low pH of the site groundwater, as described in Historical Trends. The sodium carbonate or bicarbonate will also be added to the recirculation system incrementally, and will be determined by the groundwater pH that is measured within the pilot study. During the initial injection, a continuous-feed pump will introduce sodium carbonate into the groundwater injection line at a feed concentration of approximately 0.5 pound per gallon. As the initial subsurface conditions are monitored and the collected data are interpreted, a decision will be made regarding the volume (if any) of additional sodium carbonate to be added to the pilot study groundwater. Also, a decision will be made whether to continue with sodium carbonate or to switch to sodium bicarbonate. If sodium bicarbonate is chosen, the same dosage rate and concentration given for sodium carbonate will be used for sodium bicarbonate.

SAP Worksheet #10 -- Problem Definition ([UFP-QAPP Manual Section 2.5.2](#))

10.8.2 Microbial Inoculum

A microbial inoculum containing, but not limited to *Dhc* and *Dhb* organisms may be injected via the recirculation injection well. To determine if *Dhc* and *Dhb* will be injected, reducing conditions will be monitored primarily by the DO concentration and ORP potential at the injection and monitoring wells. If TCE and 1,1,1-TCA daughter products are not detected after the ORP at the site has dropped to a level at or below -150 for several weeks, a microbial inoculum will be injected via the recirculation injection well. The indigenous bacterial population will be determined during Sampling Event 1 (see Worksheet 18), which will be conducted prior to the addition of any chemical amendments to the aquifer.

10.9 PILOT SCALE SAMPLING

Groundwater samples will be acquired throughout the course of the pilot study to determine the effectiveness of the treatment technology. A preliminary schedule for these events is illustrated on Figure 4-3. Groundwater samples and water level measurements will be taken from selected existing wells, the proposed new monitoring wells, and the injection and extraction wells. Field parameters (pH, DO, etc.) will be obtained during each sampling round, and will also be periodically measured on an irregular or as-needed basis throughout the scheduled operation of the recirculation system. The planned sampling rounds are as follows:

The initial round of groundwater samples for the Phase II investigation (which is identified as Round 2 on Figure 4-3, as Round 1 will be conducted during the Phase I investigation) will be taken prior to the startup of the groundwater recirculation system to provide a baseline for the later sampling rounds.

The sampling schedule presented in this report assumes that the sampling rounds will be scheduled relative to elapsed times from system startup, in order to acquire sufficient data to evaluate this technology within the total time allotted for the performance of the project. Technically, it is preferable to time the sampling rounds to coincide with the completion of pore volume flushes of the aquifer. The estimated time interval of a pore volume flush is currently unknown; this time interval will be estimated based on the results of the aquifer pumping test to be conducted during the Phase I field work. If the estimated flush interval allows for the completion of four flush volumes within the schedule that the Navy has allotted for the performance of this pilot test, then the sampling schedule will be modified to coincide with the completion of these flushes. Otherwise, the arbitrary time intervals, or possibly a hybrid of the two approaches, will be used for the sampling schedule. The actual sampling schedule (based on the results of the aquifer test) will be discussed in the Phase I Letter Report / Work Plan Addendum.

Round 3 samples will be taken approximately 4 weeks after the amendments are added to the groundwater recirculation to determine if the pilot study area has achieved reducing conditions.

Round 4 samples will be taken approximately 12 weeks after the amendment addition to determine if any initial reductions in natural attention parameters or TCE and 1,1,1-TCA concentrations are occurring. If the TCE and 1,1,1-TCA daughter product concentrations or the *Dhc/Dhb* titers do not increase after this third round, then the microbial inoculum (bioaugmentation) will be injected. If these parameters do increase to reasonable concentrations, then it is possible that the inoculum may not be injected.

Round 5 and Round 6 samples will be taken approximately 6 weeks and 20 weeks after the amendment addition and *Dhc/Dhb* inoculation to determine if the TCE and 1,1,1-TCA daughter products are being created or their concentrations are increasing, and also to determine if the injected *bacteria* are multiplying. Following an analysis of the results from the sixth round (to be conducted approximately 8 months after the startup of the recirculation system) the decision to either shut down or continue the test will be made. If it is determined that the continued operation of the system is necessary, then additional (currently unscoped) sampling round(s) may be conducted to ultimately support the decision to turn off the system.

SAP Worksheet #10 -- Problem Definition ([UFP-QAPP Manual Section 2.5.2](#))

The technical decision for shutting down the recirculation system will be based on the evaluation and integration of multiple hydrogeologic and geochemical parameters, chiefly the bioremediation indicators discussed in detail in Section 10.10.4 of Worksheet 10. TtNUS will monitor and evaluate these indicators throughout the test to evaluate the progress of the test and to determine when the efficacy of the remedial alternative has been demonstrated. Throughout this period, TtNUS will have frequent informal and unscheduled discussions with the Navy. When the time for recommended shutdown is reached, the communication pathways defined in SAP Worksheet 6 will be followed. The TtNUS PM will immediately notify the Navy RPM of TtNUS' recommendation to cease the recirculation, but the recirculation will continue until the Navy RPM notifies the TtNUS PM that the Navy concurs, and directs TtNUS to cease the recirculation. At this time, the TtNUS PM will notify the TtNUS FOL to cease the recirculation and to begin the post-shutdown monitoring tasks as soon as logistically possible. The Navy RPM will notify the EPA RPM that the recirculation system has been shut down.

The seventh and eighth sampling rounds will be conducted approximately 12 and 24 weeks after the recirculation system is shut down in order to evaluate the continued effectiveness of the treatment technology and to detect possible rebound in groundwater VOC concentrations.

Collected groundwater samples will be analyzed for chlorinated VOCs and field parameters including temperature, pH, specific conductivity, ORP, dissolved carbon dioxide, alkalinity, DO, iron, hydrogen sulfide, and water levels. Some samples will also be analyzed for dissolved gases, dissolved iron and manganese, sodium, sulfide, sulfate, nitrate, nitrite, chloride, phosphate, lactic, pyruvic, acetic, propionic, butyric, and TOC. Select samples will be analyzed for polymerase chain reaction (PCR) and functional genes to monitor the presence and abundance of Dhc or Dhb. All samples collected during each round will not be sampled for all parameters. The sampling strategy and plan is outlined in detail in Worksheet 18.

The degree of pore volume displacement and product travel within the pilot study area will also be investigated and verified during the pilot study. The groundwater specific conductivity that is measured in the field will be used to verify the product travel due to recirculation. The pore volume displacement, which is related to the groundwater velocity, will be measured by monitoring the elapsed time between the injection of sodium bicarbonate and the detection of sodium bicarbonate influences in the downgradient monitoring wells.

10.10 ENHANCED IN SITU ANAEROBIC REDUCTIVE DECHLORINATION

This section provides a brief summary of the enhanced in situ anaerobic reductive dechlorination (bioremediation) process for chlorinated VOCs. It also describes how the introduction of additional electron donors, along with the appropriate bacteria, can stimulate or accelerate the reductive dechlorination process.

10.10.1 DECHLORINATION of CHLORINATED VOCs

Reductive dechlorination is one of several processes by which chlorinated VOCs are transformed to environmentally innocuous compounds. It is widely recognized that in the presence of hydrogen, TCE can be reduced to DCE. While various DCE isomers (e.g., 1,1-DCE, cis-1,2-DCE, and trans-1,2-DCE) can be produced, it is well documented that cis-1,2-DCE is the most common degradation product of TCE. DCE can then be reduced to vinyl chloride (VC), which, in turn, can be reduced to ethene and potentially ethane, or via mineralization to carbon dioxide, water, and chloride ions. Similarly, TCA can be degraded to 1,1-DCE or 1,1-DCA and then to VC and chloroethane, respectively, and ultimately to ethane. Each of these processes are graphically illustrated in Figure 1-10.

SAP Worksheet #10 -- Problem Definition ([UFP-QAPP Manual Section 2.5.2](#))

10.10.2 BIODEGRADATION of CHLORINATED VOCs

In the presence of the appropriate bacteria, a suitable electron donor (e.g., hydrogen), and favorable geochemical conditions, microbial dechlorination can occur in the subsurface. This rigorously studied microbial process occurs under anaerobic (oxygen-deficient) conditions where organic carbon ferments and produces hydrogen. Several environmental requirements must be achieved for the successful implementation of this process.

One of the requirements for in situ reductive dechlorination is the presence of the appropriate bacteria. Without the appropriate bacteria, complete in situ reductive dechlorination will not occur. It is widely recognized that the reduction of TCE may stall at DCE because of a lack of the specific bacteria strain Dhc. Dhc bacteria and various strains are thought to be the only bacteria to successfully complete reductive dechlorination of chlorinated solvents. In response to the problem of DCE stall (where the dechlorination process slows down or ends with the production of DCE), many studies have concluded that the DCE stall can be avoided by the injection of a Dhc bacteria inoculum into the bioremediation area. Other studies have suggested that Dhc and other microbial communities are inhibited in the presence of 1,1,1-TCA and other communities (including Dhb) that effectively reduce 1,1,1-TCA.

The dechlorination process also may stall at VC because VC typically degrades more readily under aerobic conditions, and requires special conditions to degrade in an anaerobic environment. VC can be efficiently and rapidly destroyed under anaerobic conditions if sufficient donor and target bacteria are

present. Recent industry experience indicates that not only Dhc, but importantly, the Dhc reductive dehalogenases genes RDase (tceA) and VC RDase (vcrA and bvcA) should be present for complete destruction to occur. The chance for VC stall will be minimized with the bioaugmentation or injection of specific RDase VC RDase (vcrA and bvcA) into the subsurface. This reductase has been shown to be largely responsible for efficient anaerobic VC reduction.

Another requirement for in situ reductive dechlorination is the presence of a suitable electron donor. This electron donor can come from natural organic carbon or anthropogenic carbon sources such as hydrocarbon contaminants (e.g., benzene, toluene, ethylbenzene, and xylene, or BTEX). Specifically at Site 5, there is some evidence that the BTEX compounds present in the vadose and saturated zones may be one of the biostimulants fostering the degradation process to date. Lactate (a soluble electron donor) is a common substrate that may be added to an aquifer to increase the availability of electron donors. Lactate dissolves in water and is typically used quickly by indigenous microorganisms, primarily bacteria, which metabolize the lactic acid and produce hydrogen. An advantage to using soluble electron donors is that delivery and distribution is more easily achieved in a heterogeneous environment such as that existing at Site 5. Also, injection of soluble donors from a given or fixed point can arguably cover a larger area than would be treated with slow-release electron donors. The primary disadvantage of soluble electron donors is that they are generally consumed within 1 to 4 months, depending on a number of factors.

The last requirement for in situ reductive dechlorination is favorable anaerobic geochemical conditions. One of the most important geochemical parameters affecting the terminal electron acceptors that compete for the electron donor (described in greater detail in Section 3.4) is pH. The microorganisms that degrade the injected carbon substrates and produce the hydrogen essential for reductive dechlorination grow and reproduce most efficiently within a pH range of 6 to 8. If the natural pH of a site is low (as it is at Site 5), then a sodium carbonate/bicarbonate addition can increase the pH of an aquifer to within the preferred range. These materials can also add a buffering capacity to the aquifer to resist future pH change, which can be very useful because the typical carbon substrates are organic acids (such as lactic acid) and the microbial processes further reduce the pH.

SAP Worksheet #10 -- Problem Definition ([UFP-QAPP Manual Section 2.5.2](#))

10.10.3 AMENDMENT DELIVERY

Delivery of the amendments to the subsurface is paramount in the success of the implementation. There are generally three types of injection considered for source and plume treatment, including: (1) direct injection, (2) groundwater recirculation and (3) alternative methods (e.g. fracturing, reactive barriers, etc.). For Site 5, only direct injection and groundwater recirculation are considered applicable.

Direct injection utilizes intrusive tooling (e.g., direct push technology or wells) to directly inject the amendments into the ground. This approach is a one-time injection event, and if multiple injections are necessary then multiple mobilizations are required.

Groundwater recirculation is an alternate application that increases the natural flow rate of groundwater, enhances the distribution of amendments, and increases the overall rate of bioremediation. Groundwater recirculation can be cost effective in areas where the hydraulic gradient and groundwater flow rate are low, as they are at Site 5. Typically an extraction well is located downgradient of the contaminated groundwater plume segment to be treated, and an injection well is located upgradient of the treatment area. Groundwater is extracted from the extraction well and injected into the injection well to enhance groundwater flow. Depending on the rates of injection and extraction, the flow of the groundwater within the area of concern can be greatly increased, which allows the rate-limiting process of contaminant mass transport to occur more quickly.

Groundwater recirculation is also thought to more thoroughly capture groundwater within an area of contamination than would be achieved by relying solely on natural groundwater flow. An approximate "area of capture" can be determined by using modeling techniques, contaminated subsurface geological conditions, and the injection and extraction rates.

10.10.4 BIOREMEDIATION INDICATORS

Biodegradation processes can be measured through several lines of evidence. Because biodegradation processes in natural attenuation are the same as the processes in enhanced bioremediation, similar measures of performance can be used to demonstrate whether enhanced bioremediation is effectively reducing contamination.

The first line of evidence that the bioremediation is effective is a trend of decreasing contaminant mass and/or concentration. More specifically, demonstrating a decrease in the concentration of the parent compounds [for example, TCE] coupled with the generation or increase in concentration of daughter or breakdown products (cis-1,2-DCE, VC, ethene, etc.) is useful. It is important to note that because dechlorination occurs sequentially, dechlorination of a parent compound may result in a temporary increase, then decrease, in the concentrations of the various daughter products.

A secondary line of evidence that the bioremediation is effective includes the use of geochemistry data to indirectly illustrate that biodegradation is occurring. Natural attenuation (i.e., geochemical) parameters are generally used to evaluate the suitability of geochemical conditions for biodegradation and to determine if bioremediation is occurring. The complex relationship among many of these parameters is described in the following sections.

Dissolved Oxygen: DO acts as a primary substrate or co-substrate during the initial stages of metabolism. For chlorinated hydrocarbon degradation, anaerobic pathways are more efficient. If DO concentrations are greater than 0.5 to 1.0 mg/L, anaerobic bacteria cannot exist and reductive dechlorination will not occur.

Nitrate/Nitrite: After DO has been depleted through aerobic respiration, anaerobes will utilize nitrate (NO_3^-) as an electron acceptor to anaerobically degrade hydrocarbons (denitrification). This process reduces nitrate to nitrite (NO_2^-) and generates carbon dioxide.

SAP Worksheet #10 -- Problem Definition ([UFP-QAPP Manual Section 2.5.2](#))

Dissolved Manganese: After DO and nitrate have been depleted, anaerobic microbes will utilize manganese (Mn^{4+}) as an electron acceptor to anaerobically degrade hydrocarbons, generating manganese (Mn^{2+}) and carbon dioxide.

Ferric Iron/Ferrous Iron: After DO, nitrate, and manganese reduction, anaerobic microbes will utilize ferric iron (Fe^{3+}) as an electron acceptor (iron reduction), generating ferrous iron (Fe^{2+}) and carbon dioxide. Ferric iron is not typically analyzed, thus, the presence or increase in concentration of ferrous iron and carbon dioxide are indicators of iron reduction.

Sulfate/Sulfide: After dissolved oxygen, nitrate, manganese, and ferric iron have been utilized, anaerobic microbes will utilize sulfate (SO_4^{2-}) as an electron acceptor, resulting in sulfide and carbon dioxide increases. Sulfate reduction, along with methanogenesis, is one of the most important reduction pathways indicating conditions that are favorable for biodegradation of chlorinated hydrocarbons. Sulfide can be present in many forms, the three primary forms being the sulfide ion (S^{2-}) or dissolved hydrogen sulfide as either H_2S or HS^- .

Phosphate: Similar in mechanism to sulfate reduction (but to a lesser degree), phosphate (PO_4^{3-}) reduction is an anaerobic biodegradation process whereby bacteria can use phosphate as an electron acceptor. Phosphate, along with nitrogen, are also regarded as nutrients for biodegradation.

Dissolved Carbon Dioxide: Methanogenesis occurs after oxygen, nitrate, manganese, ferric iron, and sulfate have been utilized. It is one of the most important reduction pathways responsible for chlorinated VOC degradation. During methanogenesis, bacteria utilize carbon dioxide as an electron acceptor, generating methane as a byproduct. Carbon dioxide is produced by every terminal electron acceptor process.

Dissolved Methane: Because methane is not a chemical component of solvents, its presence at concentrations greater than background provides strong evidence of methanogenic fermentation (and carbon dioxide utilization). The measurement of background concentrations of methane are important because some natural sources of methane exist.

Dissolved Hydrogen: Hydrogen is produced as a result of fermentation of organic carbon. Hydrogen is then utilized by respiratory microbes such as nitrate reducers, iron reducers, sulfate reducers, and methane producers. Each microbe utilizes hydrogen more or less efficiently, leading to either a buildup or a decrease of hydrogen concentration. The hydrogen concentration therefore can provide an indication of the reduction process that is most prevalent in the subsurface at any one location.

Dissolved Ethene/Ethane: As discussed earlier, ethene and possibly ethane signify the final degradation step of chlorinated ethenes. Concentrations of ethene greater than 0.01 mg/L and ethane greater than 0.1 mg/L provide strong evidence of such degradation.

Chloride: Chloride concentrations are used to evaluate biodegradation because chloride is released into groundwater during dechlorination of chlorinated VOCs. Therefore, an increase in chloride concentration in the downgradient direction is direct evidence of dechlorination.

Oxidation-Reduction Potential: The oxidation-reduction potential (ORP) of groundwater is a measure of the relative tendency of the groundwater solution to accept or donate electrons and the amount of energy released during electron transfer. The ORP [in millivolts; (mV)] can provide evidence of the type of biodegradation processes that are active in a particular plume or area within a plume. The range of ORP values representing optimum conditions for reductive dechlorination are typically within the range of -100 to -350 mV. ORP should be used only as a qualitative indicator of the overall oxidation/reduction state.

Total Alkalinity: A result of both aerobic and anaerobic biodegradation is production of the hydrogen ion (H^+). When the hydrogen ion is produced, alkalinity will be reduced. In low alkalinity aquifers, the pH

SAP Worksheet #10 -- Problem Definition ([UFP-QAPP Manual Section 2.5.2](#))

may drop to levels outside the range of microbial activity. Thus, to have optimum conditions for microbial growth, it is essential to have a properly buffered aquifer.

pH: pH concentration is an indicator of the amount of free hydrogen ion available in a solution. Optimum conditions for microbial growth are within the pH range of 6 to 8.

Temperature: Temperature affects the metabolic activity of bacteria, as well as the solubility of geochemical species. Microbes are generally more active and efficient in warmer water. Biochemical processes are accelerated at temperatures greater than approximately 20 degrees Celsius.

A tertiary line of evidence, but argued by many as a primary line of evidence, is nucleic acid-based molecular biological tools (MBTs) used to understand the presence and the quantity of the microbiological population. For sites such as Site 5, where enhanced anaerobic bioremediation has been proposed, MBT analysis should be conducted as a part of a pre-design remedial investigation and during bioremediation to assess the site and evaluate the potential need for bioaugmentation. Dhc titers less than $10^4/L - 10^6/L$, or the requirement for faster remediation time frames, indicate bioaugmentation may be needed.

10.11 THE PROBLEM TO BE ADDRESSED BY THE PROJECT

This SAP contains the technical scope of work and associated sampling plan to perform a (1) pre-remediation site characterization and (2) a pilot test (including treatability testing). The intent of the pre-remediation characterization is to understand the source location and architecture to effectively understand and subsequently design the optimal remediation system. The purpose of the pilot test is to

design, evaluate the performance and ultimately prove the applicability of a biostimulation or bioaugmentation strategy. The information derived from both aspects of this project will be used to aid in the design and installation of a full-scale biostimulation/bioaugmentation remedy. A field pilot study is imperative because the extraction and/or injection system parameters are unique to site. The data collected for both of these aspects of the project include collection of subsurface soil samples, collection of groundwater levels, and collection of groundwater samples (e.g., contaminant chemistry and geochemistry) to determine site characteristics and pilot study performance.

The environmental questions being asked:

- **What are the VOC concentrations in the soil?** The sampling and analysis of these soils will determine the extent to which they are continuing to source the groundwater plume.
- **What are the current groundwater conditions relevant to the existence and propagation of the contaminant-reducing bacteria?** The current groundwater conditions will be analyzed to determine if the required bacteria occur naturally at the site, if the existing groundwater conditions (such as pH and oxygen content) can be adjusted to increase the bacteria population, or if the groundwater must be augmented through the addition of commercially-obtained bacteria colonies.
- **Will the biostimulation or bioaugmentation of the groundwater remediate the aquifer by reducing the concentration of dissolved VOCs?** Periodic sampling of key wells will be performed to monitor the changes in VOC concentration and associated groundwater chemistry over time throughout the performance test to determine if the VOC concentrations decline during the active portion of the test, and if they remain at these reduced levels when the active (groundwater circulation) portion of the test is completed.

Collection and evaluation of groundwater levels and groundwater samples (e.g., contaminant chemistry and geochemistry) will represent a baseline and over the course of the pilot study will be utilized to determine concentration trends as they relate in time and space to answer these questions. These data will ultimately be used as metrics of success for the pilot study and subsequently the full scale remediation.

SAP Worksheet #10 -- Problem Definition
([UFP-QAPP Manual Section 2.5.2](#))

Observations from any site reconnaissance reports:
NA

A synopsis of secondary data or information from site reports:
Historical data from the RI will be used to establish trends.

The possible classes of contaminants and the affected matrices:
VOCs in soil and groundwater.

The rationale for inclusion of chemical and non-chemical analyses:
All possible analytical fractions are included to cover items that may have been released to the groundwater. Also includes environmental indicators to determine how pilot test is functioning. Full list of field analyses is included in Worksheets 18 and 19.

Information concerning various environmental indicators: Biological indicators are explained above in Section 10.10.4. These indicators will be monitored during the before, during, and after the pilot study.

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

If soil concentrations are encountered that significantly contribute to VOC groundwater contamination, then the Navy will evaluate potential actions. Results of the Phase I soil investigation will be discussed at the quarterly NAS JRB status meetings attended by the Navy, EPA, and PADEP. In addition, informal and currently unscheduled teleconference calls will be held as needed.

If current conditions are suitable for bacteria to biodegrade the VOC contamination, then biostimulation will occur. If current conditions are not suitable, chemical amendments will be added to the aquifer to make the conditions more favorable for bacterial biodegradation, as discussed in Section 10.8.1. If current conditions indicate that bacteria are too few for VOC degradation, then biostimulation and bioaugmentation will be performed, as discussed in Section 10.8.2.

If the bioremediation pilot test causes VOC concentrations to decrease, then a full-scale bioremediation at Site 5 will be implemented. If the bioremediation pilot test does not cause VOC concentrations to decrease at Site 5, then further investigation and alternatives will be considered.

At the present time, concentrations exceed the Federal Maximum Contaminant Levels (MCLs), therefore, a (1) pre-remediation site characterization and (2) a pilot test will be implemented. If concentrations are decreased as part of the pilot study and there is a full scale remediation then long-term monitoring will be implemented. The endpoint of the monitoring is the end of the 6-month post-circulation period to look for anticipated rebounds in contaminant concentrations. The endpoint of the circulation period (and associated monitoring) will be either the end of 12 months of circulation, or some earlier termination point if the efficacy of the alternative is proven, based on the evaluation of multiple bioremediation indicators. The decision process and communication pathways for ending the circulation period are discussed in Section 10.9 of Worksheet 10. The bioremediation indicators are discussed in Section 10.10.4 of Worksheet 10.

SAP Worksheet #11 -- Project Quality Objectives/Systematic Planning Process Statements
([UFP-QAPP Manual Section 2.6.1](#))

Who will use the data?

Navy and Tetra Tech NUS (TtNUS). The regulatory agencies will evaluate the Navy's implementation plan, performance results and subsequent recommendations.

What will the data be used for?

Groundwater data will be used to determine the efficacy of bioremediation as the long-term remedial action for this site. Soil data will be used to determine whether residual VOC contamination in the soil is continuing to source the groundwater plume, and to evaluate if remediation of this soil would contribute to the remediation of the groundwater.

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

Fixed-based lab data and field tests will to be used to characterize soil and groundwater contamination. Soil and groundwater analyzed for Target Compound List (TCL) VOCs. Groundwater will also be analyzed for geochemistry and MBTs including:

Dissolved Gases – ethane, ethene, methane, and acetylene

Dissolved Iron

Dissolved Manganese

Total Sodium

Sulfide

Anions – sulfate, sulfide, nitrate, nitrite, chloride, and phosphate

TOC

Metabolic Acids – lactic, pyruvic, acetic, propionic, and butyric

Polymerase Chain Reaction (PCR) – TCE reductase, BAV1 VC reductase, and VC reductase

Field Test Parameters include:

DO

Dissolved Carbon Dioxide

Alkalinity

Ferrous Iron

Hydrogen Sulfide

Matrix: Soil/Groundwater

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

The VOC analytical data will undergo the full level of validation for 100 percent of the data because VOCs are the contaminants of concern. VOC data will be used to evaluate historical trends. All other fixed-base laboratory parameters will receive a cursory evaluation to make sure usable data is obtained. These other parameters are indicators to evaluate the characteristics and efficacy of the pilot test system and full validation is not necessary. The laboratories must hold a current NELAP accreditation in Pennsylvania and comply with the requirements of NFESC in analytical results reporting and QA/QC.

In addition to fix-based laboratory analytical work, there will be several parameters that will be measured in the field using a direct-reading meter and test kits including temperature, pH, specific conductance, ORP, ferrous iron, hydrogen sulfide, DO, dissolved carbon dioxide, and alkalinity. Specifics for field analyses are included in Worksheet 18 and 19.

SAP Worksheet #11 -- Project Quality Objectives/Systematic Planning Process Statements ([UFP-QAPP Manual Section 2.6.1](#))

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

Soil

One round of 12 baseline source area soil locations will be bored and 24 samples will be collected. The twelve locations were chosen using the analytical results of previous soil borings and using the results of historical aerial photo analysis that delineated the extent of the drum staging area. The analytical results of the previous soil borings are illustrated in Figure 1-6, and these same results and the proposed locations for the new soil borings are illustrated in Figure 2-1.

Groundwater

The complete sampling and analysis plan is presented in Worksheet 18.

One round of pre-design groundwater samples will be collected from nine existing wells for multiple parameters. The specific parameters are listed in Worksheet 18.

Seven rounds of groundwater samples will be collected on a quarterly basis.

Round 1 will be baseline sampling prior to the pilot study.

Rounds 1, 3, 5, and 7 will be of the complete 20 wells.

Rounds 2, 4, and 6 will be of a 10-well subset of the 20 total wells. The 10 wells chosen for sampling may not be the same wells each round. The wells will be chosen to fill the particular data requirements needed at that time, based on the cumulative results of the project's sampling program up to that point. For example, if a well exhibits little or no response to the bioremediation, it may be removed from the sampling program or have its sampling frequency reduced, while a well that exhibits rapid response to the bioremediation may be sampled every round. Prior to Rounds 2, 4, and 6, the TtNUS PM will present a list of 10 recommended wells to the Navy PM for review and approval.

Rounds 6 and 7 will be post-study sampling.

Concentrations are expected to be low to medium.

There are no project action levels for soils and groundwater samples at the site. The MCLs were chosen for required groundwater reporting limits from the laboratory only. For this pilot study, the objective is to determine if the treatment alternative is effective by looking for decreasing concentrations trends and other indicators (changes in chemistry). Absolute action levels are not driving the pilot study, such as requiring concentrations to be reduced to levels below MCLs. However, reporting limits for VOCs from the laboratory will meet MCLs so the data can be used for the full-scale alternative.

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

Solid and aqueous data will be generated at a fixed-base laboratory upon receipt of the soil and groundwater samples generated in the field at Site 5. Soil samples will be collected from direct-push soil borings. Groundwater samples will be collected using the low-flow sampling procedure. Some groundwater data will be generated in the field using direct-reading meter and test kits (see Worksheets 18 and 19).

Data deliverables will be submitted by the laboratory as a hardcopy and in Portable Document Format (PDF). Laboratory reports will be fully validatable and contain raw data, summary forms for all sample and laboratory method blank data, and summary forms containing all method specific quality control

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5
Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study
Revision Number: 0
Revision Date: September 2008

SAP Worksheet #11 -- Project Quality Objectives/Systematic Planning Process Statements
[\(UFP-QAPP Manual Section 2.6.1\)](#)

(results, recoveries, relative percent differences, relative standard deviations, and percent differences). The deliverables will be received by the TtNUS Sample Management Coordinator. The Sample Management Coordinator will upload the data into the project database and distribute the data packages to the data validators. The data validation report will be delivered to the project manager and the finalized data will be maintained on a Simple Query Language (SQL) server. Hardcopies will be filed in the project file according to CLEAN contractual obligations.

MNA – Monitored Natural Attenuation
NELAP – National Environmental Laboratory Accreditation Program
NFESC – Navy Facilities Engineering Service Center
QA/QC – Quality Assurance/Quality Control
TCL – Target Compound List
TtNUS – Tetra Tech NUS, Inc.
VOC – Volatile Organic Compound

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #12 -- Measurement Performance Criteria Table note matrix in table entry[\(UFP-QAPP Manual Section 2.6.2\)](#)**12.1 Soil Measurement Performance Criteria Table – Field QC Samples**

QC Sample	Analytical Group¹	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Matrix spike/matrix spike duplicate (MS/MSD)	TCL VOC	One per Sample Delivery Group (SDG) or every 20 samples	Accuracy / Bias / Precision	See table in Worksheet 28 for specific %R limits, 30% RPD	A
Field Duplicates	TCL VOC	One every 10 samples	Precision	Values > 5X QL: within \pm 50% Values < 5X QL: absolute difference must be <2XQL	S & A
Cooler Temperature Indicator	TCL VOC	Each cooler	Accuracy / Representativeness	Between 2 and 6 °C	S

¹If information varies within an analytical group, separate by individual analyte.**No field or equipment blanks will be necessary for soil sampling because disposable or dedicated equipment will be used.**

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5
 Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #12 -- Measurement Performance Criteria Table note matrix in table entry
[\(UFP-QAPP Manual Section 2.6.2\)](#)

12.2 Groundwater Measurement Performance Criteria Table – Field QC Samples

QC Sample	Analytical Group ¹	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Trip Blank	TCL VOC	One per cooler of VOC samples shipped to laboratory	Bias / Contamination	No target analytes \geq QL; with the exception of common field/laboratory contaminants	S & A
MS/MSD	TCL VOC	One per Sample Delivery Group (SDG) or every 20 samples	Accuracy / Bias / Precision	Statistically derived %R limits, 30% RPD	A
Field Duplicates	TCL VOC	One every 10 samples	Precision	Values > 5X QL: within \pm 30% Values < 5X QL: absolute difference must be <QL	S & A
Field Blank	TCL VOC	One per source of rinsate water	Bias / Contamination	No target analytes \geq QL; with the exception of common field/laboratory contaminants	S
Equipment Blank	TCL VOC	One every 20 samples	Bias / Contamination	No target analytes \geq QL; with the exception of common field/laboratory contaminants	S
Cooler Temperature Indicator	TCL VOC	Each cooler	Accuracy / Representativeness	Between 2 and 6 °C.	S

¹If information varies within an analytical group, separate by individual analyte.

No field QC samples will be collected for the other groundwater parameters.

°C – Degrees Centigrade

%R – Percent Recovery

MS – Matrix Spike

MSD – Matrix Spike Duplicate

QL – Quantitation Limit

RPD – Relative Percent Difference

SDG – Sample Delivery Group

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

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

Secondary Data	Data Source (originating organization, report title and date)	Data Generator(s) (originating organization, data types, data generation / collection dates)	How Data Will Be Used	Limitations on Data Use
RI Analytical Data	Remedial Investigation Report for Site 5 – Fire Training Area	TtNUS, February 2002	Data will used to establish trend with current groundwater conditions	None, the data were fully validated.
Soil Sampling Analytical Data	Soil Investigation for Volatile Organic Compound, Soil to Groundwater Impact, Site 5 – Fire Training Area	TtNUS, March 2006	Data will used to determine source area.	None, the data were fully validated.
RI Addendum Analytical Data	Remedial Investigation Addendum for Site 5 – Fire Training Area Groundwater (OU 2)	TtNUS, September 2006	Data will used to establish trend with current groundwater conditions	None, the data were fully validated.

OU – Operating Unit
RI – Remedial Investigation
TtNUS – Tetra Tech NUS, Inc.

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #14 -- Summary of Project Tasks[\(UFP-QAPP Manual Section 2.8.1\)](#)**PRE-TREATABILITY STUDY FIELD INVESTIGATION (PHASE I INVESTIGATION)****14.1 SOIL**

Soil sampling and analysis will be conducted to determine the lateral and vertical extent of VOCs in the subsurface soils at the former drum storage area. The results of the previous soil investigations indicate that the location of the highest VOC concentrations in the soils coincides with the location of the highest VOC concentrations in the groundwater, suggesting that these impacted soils may continue to serve as a residual source of the groundwater plume. The existing data, however, are too widely spaced to fully evaluate their impact on the underlying groundwater, to confidently define the extent of the source area, or to calculate the volume of soil potentially requiring remediation. The analytical data obtained through this current investigation will be used to refine the extent of impacted soils, to determine whether remediation of these soils is required, and if so, to evaluate the potential remedial options and alternatives.

The soil boring investigation is a dynamic program designed to delineate the total extent of impacted soils. As discussed in Worksheet 11, the planned boring locations are based on the known present site conditions (the analytical results of previous borings) and historical site conditions (the historical aerial photo analysis that delineated the extent of the drum staging area). These boring locations, however, may be adjusted in the field or additional borings may be added, if the borehole conditions observed during the drilling program indicate that additional data are needed to define the extent of contamination. For example, if the field screening results for a boring located at the lateral extent of the planned program indicate that VOCs are present at that location, then an additional boring will be located and drilled at a distance of approximately 20 feet beyond that boring. This process will be repeated until the field screening process indicates that a region of uncontaminated soil has been reached.

As discussed in the following sections, continuous soil cores will be obtained from the ground surface to the top of bedrock (14.1.1). The entire core will be screened for VOCs with a photoionization detector (PID). The results of a previous soil boring program (TtNUS, March 2006, see Worksheet 13) indicated that the PID was a reliable indicator for the presence of VOCs, and there was good agreement between the relative levels of the PID readings and the concentrations of the VOCs in the soils. In addition, the sampling strategy (14.1.2) calls for the submittal of two samples from every boring, including those borings where no elevated PID readings are detected.”

14.1.1 Soil Boring Drilling

Soil samples will be acquired from 12 soil borings drilled at the former drum staging area. The locations and the most significant analytical results from previous soil borings as well as proposed borings are illustrated on Figure 2-1.

The boring locations will be staked in the field by TtNUS prior to the mobilization of the drilling subcontractor. The locations will be cleared for subsurface utilities by the drilling subcontractor through the PA One-Call program. In addition, NAS JRB personnel will clear the area for site-specific utility locations not available to the One-Call program.

The soil borings will be drilled using a direct-push technology (DPT, or “Geoprobe”) drilling rig. With a DPT rig, a probe assembly that is fitted with a 4-foot or 5-foot acetate sleeve is hydraulically advanced (or “pushed”) into the subsurface to obtain soil cores. The probe assembly is withdrawn, the acetate liner is cut open,

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #14 -- Summary of Project Tasks**[\(UFP-QAPP Manual Section 2.8.1\)](#)**

and the core is described and sampled. The sampling assembly (with a new acetate sleeve) is then reinserted into the open borehole to obtain a core from the next, deeper 4-foot or 5-foot interval of soil. If collapsing borehole conditions exist due to uncohesive soils, a discrete-interval (closed piston) sampler can be used to prevent the subsequent resampling of shallow soils. However, the TtNUS experience at this site during previous investigations indicates that the fine-grained, cohesive soils create a borehole that remains open when the sampling apparatus is removed. Therefore, the need for a discrete-interval sampler is not anticipated.

Each soil boring will be drilled to the depth of DPT refusal, which is expected to occur at the top of bedrock, and at a maximum depth of about 20 feet. Upon retrieval, the acetate liner for each cored interval will be cut open, the lithology of the cored interval will be described by the site geologist, the cored interval will be vertically screened for evidence of contamination with a Photoionization detector (PID), and any qualitative visual signs of potential contamination (such as soil staining) will be noted. Engineered controls will be employed, where possible, to minimize the effects of the wind on the PID readings (for example, the readings may be taken on the lee side of a field vehicle, or the wind will be screened by the sampling trowel.

The soil samples will be classified through the Unified Soil Classification System (USCS). The lithology, PID measurements, and all other pertinent observations will be recorded on the borehole log.

14.1.2 Soil Sampling and Analysis

An estimated total of 24 primary subsurface soil samples (excluding quality assurance/quality control [QA/QC] samples) will be submitted for VOC analysis. The sampling program is summarized in Worksheets 18 and 19, including the bottle ware, preservative, and holding time requirements.

The selection criteria for choosing the samples for analysis will include the soil's relative degree of contamination (as determined by the highest PID readings), visible staining, noticeable odors, and the vertical position of the soil sample relative to the top of bedrock. The decision pathway and guidelines for sample selection are summarized in Table 2-1.

The VOC soil samples will be obtained with EnCore™ sampling equipment. Field preservation of the samples will not be performed. To ensure that no loss of VOCs occurs through volatilization in the shallower core intervals (while the deeper intervals are being drilled), samples from each core will be taken immediately after the PID screening from the interval exhibiting the highest elevated readings. This sampling strategy may result in the sampling of intervals that are not ultimately submitted to the laboratory for analysis, because the highest PID readings within a boring or the samples with the highest field screening responses will not be known until the entire boring is drilled. The samples not submitted for laboratory analysis will either be returned to the boring, spread on the adjacent ground surface, or be disposed as investigation-derived waste (IDW). At the conclusion of a boring, soils that do not generate a PID response will be returned to the boring, or spread on the adjacent ground surface if the boring becomes filled. Soils that generate a PID response will be drummed for later disposal as IDW.

14.2 GROUNDWATER

The groundwater investigation will be performed in two separate phases or mobilizations. Phase I (the Pre – Treatability Study field investigation) will include installation of 5 monitoring wells at pre-selected locations, sampling of newly installed and certain existing wells for chemical and biological parameters, and performance of an aquifer pumping test. Phase II work (Pilot Test) will include installation of two additional monitoring wells, two injection wells, and two extraction

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #14 -- Summary of Project Tasks[\(UFP-QAPP Manual Section 2.8.1\)](#)

wells at locations to be determined based on the results of the Phase I work. A baseline sampling and analysis for chemical and biological parameters of all wells to be included in the pilot test will be performed immediately prior to the start of the pilot test.

During the Phase I investigation, field tests will be conducted to determine the amount of sodium bicarbonate or sodium carbonate that will be required to produce a sustainable pH (ranging between 6.2 to 8.0) to foster effective dechlorination. During the Phase I field activities, subsurface soil and rock samples will be collected during the installation of the monitoring wells to be used to conduct field buffering tests. The subsurface samples will be placed in sealed containers with the buffering solution and mixed or stirred. The materials will be allowed to come to equilibrium during the overnight period, and the pH will be measured (either by colored indicator or by field measurement) the next morning. The type and amount of solution and the resultant pH will be recorded, and the procedure will be repeated until results in the acceptable range of pH are attained.

14.2.1 Monitoring Well Installation

The existing monitoring well network spans the entire extent of the groundwater plume from its source area to its distal limit. The monitoring well density in the immediate vicinity of the source area, however, is not sufficient to adequately monitor the progress of the bioremediation pilot program, which is anticipated to be most active within about 50 feet of this area. Currently, the closest downgradient wells are at the 05MW09 cluster, which is located approximately 100 feet south of the 05MW01 cluster. The locations of the new monitoring wells to be installed are illustrated on Figure 2-2. The rationale for each location (relative to the site conceptual model) and the drilling, borehole evaluation, and well construction plans are discussed in the following paragraphs.

Well Locations

Monitoring well 05MW16S will be located upgradient of the source area and in the immediate vicinity of the planned injection wells. If the results of the soil investigation (which will be conducted prior to the well installation activities) suggest that the planned well location is still within the source area, then the location of the well will be moved further to the north.

Monitoring wells 05MW17S / 05MW17I and 05MW18S / 05MW18I will be located downgradient of the source area and downgradient of the planned extraction wells. These wells will be located between the source area and the currently impacted 05MW09 and 05MW10 well clusters. These wells will be used to monitor the quality of the groundwater immediately downgradient of the bioremediation circulation system.

Monitoring wells 05MW19S / 05MW19I are tentatively located west of the source area, and will detect or monitor any influences caused by the regional groundwater divide. This cluster will not be installed until the second phase of well installations (along with the injection and extraction wells), and its location may be revised based on the results of the aquifer pumping test.

Borehole Drilling

The monitoring well boreholes will be drilled by the air percussion drilling method. At each location, a 6-inch I.D. steel casing will be grouted to a depth of approximately 5 feet into bedrock. A 6-inch diameter borehole will be drilled out of the casing to the total depth of the borehole. For the shallow wells, the borehole will be drilled to a depth of 70 feet. For the intermediate wells, the borehole will be drilled to a depth of 150 feet.

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #14 -- Summary of Project Tasks[\(UFP-QAPP Manual Section 2.8.1\)](#)Borehole Evaluation

The initial borehole evaluation will be done by the TtNUS rig geologist, who will construct a boring log for each borehole. The geologist will describe the lithology of the rock cuttings at a maximum interval of every 5 feet, or at shorter intervals to be determined by the occurrence of significant lithologic changes. The geologist will also note the depths that fractures are encountered, the fracture's approximate yield, and the cumulative borehole yield with depth. In addition, the geologist will monitor the borehole for organic vapors with a PID, and will record the PID readings on the boring log.

The boreholes will be geophysically logged by a TtNUS geophysical subcontractor. The boreholes will be logged a minimum of 2 days after their drilling completion to allow the borehole parameters to return to ambient conditions. The borehole logs to be generated include the following:

- Caliper
- Natural gamma
- Single point resistance
- Fluid temperature
- Fluid conductivity
- Fluid velocity by heat pulse flow meter

Upon the completion of the geophysical logging program, the field copies of the boring logs and the geophysical logs will be transmitted (either by telefax or electronically in pdf format) to the Navy, EPA, and PADEP for review and to choose the borehole retrofitting plan. For each borehole, the potential zones to be screened will be identified, and the zone that ultimately best meets the project needs at that location will be chosen. A teleconference will be held within one week of the data's distribution to discuss the results and to formulate the retrofitting plan.

Well Construction

Each borehole will be retrofitted with a screened monitoring well that is constructed to monitor the groundwater from a specific vertical interval. The interval to be monitored in each borehole will generally be based upon the approximate depths that hydrogeologic and chemical data are needed to fill the data gaps at that location, and will specifically be based upon the evaluation criteria discussed above, including the boring logs, the geophysical logs, and the subsequent technical discussions.

The monitoring wells will be constructed with 2-inch-diameter, flush-joint and threaded polyvinyl chloride (PVC) well casing and 2-inch-diameter, Schedule 40, 0.020-inch slotted well screen that is fitted with a bottom cap. The space between the bottom of the borehole and the bottom of the screen (if any) will be sealed with bentonite to a depth of 5 feet below the bottom of the screen, and with No. 2 quartz sand from that depth to the bottom of the screen. The annular space between the well screen and the borehole will be packed with No. 2 quartz sand to a height of at least 3 feet above the top of the screen, and a bentonite seal with a minimum thickness of 3 feet will be emplaced above the sand pack. The remainder of the annular space from the bentonite seal to the ground surface will be grouted with a 5% bentonite/cement grout.

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

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

The monitoring wells will be developed with a submersible pump. The groundwater temperature, pH, conductivity, and turbidity will be monitored during development. The amount of water developed from the wells will be dependent on their yield and the time needed for the monitored parameters to stabilize. The wells will be developed until the parameters from three consecutive samples (taken a minimum of 5 minutes apart) fall within 10 percent of one another, or for a maximum of 2 hours, whichever comes first. If the well has not fully developed after 2 hours but has made significant progress, then development may continue for a maximum of 1 additional hour. If the well has shown little or no development after 2 hours, then no additional development will be attempted until the Navy and the regulatory agencies discuss alternate development strategies.

14.2.2 Investigation-Derived Waste (IDW)

The drilling-generated groundwater will be captured and disposed into the NAS JRB Willow Grove sanitary sewer manhole that is located at Site 5, southwest of the Marines Corps Building. While drilling, the borehole discharge will be diverted into a lined roll-off box. As the solids (drill cuttings) settle out, the overlying drilling water will be pumped from the top of the box and discharged to the sewer.

Well development water and purge water generated by well sampling will contain minimal solids, and will be captured and disposed offsite by the IDW subcontractor.

The drill cuttings will be sampled by a TtNUS IDW subcontractor for waste disposal characterization, and subsequently disposed by the subcontractor in a regulatory-approved facility.

14.2.3 Aquifer Pumping Test

The aquifer test will be used to determine the hydraulic conductivity of the aquifer. This parameter will be used to calculate the groundwater velocity, which is needed to determine the distance between the injection and extraction wells, or the length of the recirculation zone. This test will also aid in the system design by determining the probable pumping rates of the extraction wells. The well to be pumped for the aquifer test and the wells to be monitored are not chosen at this time, but will be determined on the results of the Phase I monitoring well installations. Aquifer test procedures are discussed in TtNUS SOP GH-2.3 (Appendix A of the SAP).

The test will be performed as a constant-rate test, using a pumping rate determined by the observations made during the Phase I field work. The test is anticipated to run for approximately 12 hours, but may be extended if required by conditions including, but not limited to, the failure to obtain steady-state aquifer conditions within the test area. The progress of the test will be closely monitored to assure that project objectives are achieved. If the desired results are not obtained by the end of the initial 12-hour period, the test will be extended for another 12-hour segment, at the end of which time the test will either be completed (if the desired results have been obtained), or extended for another 12-hour period. This procedure will be repeated, as necessary, until the test has been performed for up to a period of 48 hours.

Every effort will be made to schedule the test during a time period when no significant precipitation is anticipated. In the event of significant precipitation, the test may be shut down if field conditions indicate that continuing the test would lead to the acquisition of low quality or useless data. In either case, the TtNUS project manager will contact the Navy for approval prior to either extending the test beyond its planned duration or shutting down the test prior to its full duration.

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #14 -- Summary of Project Tasks[\(UFP-QAPP Manual Section 2.8.1\)](#)

The hydraulic head (drawdown) will be measured in a number of monitoring wells (to be identified after the Phase I field work) during the duration of each aquifer test by electronic pressure transducers (data loggers) or by a manual, water-level recording instrument.

Background or trend data will be collected for a period of one week preceding the test. Barometric pressure and precipitation readings will also be recorded during the period that the trend data are collected.

Water level measurements will be obtained from the pumping and observation wells immediately upon starting the pumping test. The wells with data loggers will record the measurements on the log-cycle recording option. The wells requiring manual water level readings will be measured at a lower frequency of 10-minute intervals for the first 3 hours of the test, and then at 30 minute intervals for the remainder of the test. If any manual measurements indicate that any particular well requires more frequent readings (such as unstable or fluctuating water levels), then it may be monitored at a more frequent interval, at the discretion of the field hydrogeologist.

Drawdown data from the pumping well and the observation wells will be plotted in the field on semi-log paper throughout the duration of the test. The field data plots will be used to evaluate drawdown trends, look for boundary conditions, project head-rises for the latter stages of the test, and to determine whether steady-state drawdown conditions have been attained. Field personnel will be in frequent communication with senior technical personnel to provide updates of the test progress.

Barometric pressure and precipitation will be recorded at 1-hour intervals throughout the test, or more frequently if a weather front approaches the area during the test period. These data are measured by the Base, and will be available for use in this test. Should the availability of this information change, these parameters will be measured in the field by Tetra Tech during the performance of the test.

Flow rates will be controlled through the use of a variable-speed submersible pump or the installation of a ball valve in the discharge line. Flow rates measurements will be obtained throughout the test with a totalizing flow meter installed in the discharge line, and field-verified through the use of a calibrated bucket and a stopwatch. The target flow rate will be maintained within a variance of 5 percent throughout the duration of the test. During the startup phase, the pumping rate will be constantly monitored until the flow is stabilized at the target rate. Following stabilization, flow measurements will be recorded at least every 10 minutes for the first 100 minutes of the test, at least every 20 minutes for the next 100 minutes, and at least hourly for the duration of the test. In each instance, the instantaneous flow rate and the cumulative gallons pumped will be recorded. All pumped groundwater will be discharged directly into the Base sewer system.

At the conclusion of the active pumping portion of the aquifer test, the pump will be shut off and recovery measurements will be obtained until the aquifer has recovered to within 90% of the original water levels or for a maximum period of 2 hours from the pumping well and the observation wells where significant rises in head were observed. The frequency of readings will be the same as those for the start of the test (at log-cycle frequencies).

The aquifer test data will be analyzed through both manual and automated (commercial software) methods. The drawdown data will be plotted on both semilog and log-log graph paper and analyzed using appropriate data analysis methods. Both time-drawdown and distance-drawdown methods will be considered, with the final selection of the analytical methods made based on a review of the data plots and the hydrogeologic conditions observed during the test. In addition to the manual calculations, a commercially-available software package will be used to interpret the drawdown data. The AquiferTest software package distributed by Waterloo Hydrogeologic, Inc., will be the primary software source, although other software packages may also be considered or used.

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #14 -- Summary of Project Tasks[\(UFP-QAPP Manual Section 2.8.1\)](#)

TtNUS will recommend to the Navy which wells should be monitored during the aquifer test based on the wells' geographic location, the known hydraulic properties (yield, specific capacity) of the existing wells, and the observed hydraulic properties of the wells to be installed during the Phase I field work. The results of this aquifer test will be incorporated with all Phase I data to be included in the Letter Addendum Work Plan for Phase II field work (see Worksheet 16) to be submitted to the Navy, EPA, and PADEP.

PILOT STUDY FIELD INVESTIGATION AND OPERATIONS (PHASE II INVESTIGATION)**14.3 INSTALLATION OF INJECTION, EXTRACTION, AND MONITORING WELLS**

The Phase II field work will begin with the installation of two additional monitoring wells and the installation of the groundwater recirculation system, which will consist of two injection wells and two extraction wells.

The two additional monitoring wells (well cluster 05MW19) will be installed in the identical manner prescribed in the Phase I monitoring wells. The tentative location for the new wells is illustrated in Figure 2-2, but as discussed, their actual location will be determined by the results and data requirements identified by the Phase I investigation.

The injection wells and extraction wells will be drilled to an estimated subsurface depth of 75 feet and 125 feet, respectively. The projected depths of the wells will be adjusted by the results of the Phase I investigation, and the actual final depths of the wells will be determined by both the field observations made during the drilling, and the post-drilling analysis (geophysical logging), of the respective boreholes. The tentative locations of the injection wells (05IW01 and 05IW02) and the extraction wells (05EW01 and 05EW02) are illustrated in Figure 4-1.

Borehole Drilling for Injection and Extraction Wells

The injection and extraction well boreholes will be drilled by the air percussion drilling method. At each location, an 8-inch I.D. steel casing will be grouted to a depth of approximately 5 feet into bedrock. An 8-inch diameter borehole will be drilled out of the casing to the total depth of the borehole. For the injection wells, the projected borehole depth is up to 75 feet. For the extraction wells, the projected borehole may be as deep as 125 feet. These projected depths may be adjusted after the results of the Phase I field work are interpreted and reviewed.

Borehole Evaluation

Consistent with the planned approach for the monitoring wells, the initial borehole evaluation will be done by the TtNUS rig geologist, who will construct a boring log for each borehole. The geologist will describe the lithology of the rock cuttings at a maximum interval of every 5 feet, or at shorter intervals to be determined by the occurrence of significant lithologic changes. The geologist will also note the depths that fractures are encountered, the fracture's approximate yield, and the cumulative borehole yield with depth. In addition, the geologist will monitor the borehole for organic vapors with a PID, and will record the PID readings on the boring log.

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #14 -- Summary of Project Tasks[\(UFP-QAPP Manual Section 2.8.1\)](#)

The injection and extraction wells are projected to be completed as open-bedrock wells. However, the boreholes will still be logged in order to maximize the technical information (including the depths and distributions of water entry and exit zones) obtained at each location. In addition, should the results of the Phase I field work or this geophysical logging indicate that particular zones should be cased-off or excluded from the recirculation system, these results will be used to develop the borehole retrofitting plans. The need to retrofit the injection or extraction wells is not likely but a decision will be made upon the collection of the field observations and geophysical data. The planned construction techniques and materials for the extraction and injection wells are illustrated in Figures 5-1 and 5-2, respectively. These figures include well casing and screen, in the event that retrofitting of the boreholes is required.

The boreholes will be geophysically logged by a TtNUS geophysical subcontractor. The boreholes will be logged a minimum of 2 days after their drilling completion to allow the borehole parameters to return to ambient conditions. The borehole logs to be generated include the following:

- Caliper
- Natural gamma
- Single point resistance
- Fluid temperature
- Fluid conductivity
- Fluid velocity by heat pulse flow meter

14.4 GROUNDWATER RECIRCULATION SYSTEM

To establish the recirculation system, groundwater will be pumped from the extraction wells, through a mixing system installed in a trailer-mounted unit, and returned to the subsurface via the injection wells. Sodium lactate and sodium carbonate or bicarbonate solutions will be proportionally injected into the groundwater piping and mixed by a static mixer. The dosages of sodium lactate and sodium carbonate or bicarbonate injected into the subsurface may be incrementally increased as data are analyzed during the pilot study. Metering pumps will initially inject sodium lactate and sodium carbonate or bicarbonate into the groundwater injection line at rates to be defined by the results of the Phase I investigation.

Both the sodium lactate and sodium carbonate injection rates may be adjusted depending on the ORP and pH values measured within the pilot study area. Also, a decision will be made whether to continue with sodium carbonate or to switch to sodium bicarbonate. The injection well will be monitored routinely for biofouling by observing the water level at the injection well. If a higher than normal water level is observed, the chemical feed rates may be reduced and/or the injection well will be flushed. Figure 4-4 shows a schematic of the treatment system.

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5
Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #14 -- Summary of Project Tasks

[\(UFP-QAPP Manual Section 2.8.1\)](#)

14.4.1 System Construction

The anticipated process equipment to be used for this pilot test is summarized in Appendix F. Groundwater will be extracted from the extraction wells by a Grundfos SQ (or equivalent) pump. The flow rate will be controlled with a manually adjusted globe or diaphragm valve. A paddle wheel flow meter or Rota meter will be used to measure the flow rate and adjust the control valve. Sodium lactate solution will be prepared in a 50-gallon tank and injected into the groundwater piping at a location upstream of a static mixer. Similarly, the sodium carbonate or bicarbonate solution will be prepared in a 150-gallon tank and injected into the groundwater piping at a location upstream of a static mixer. The chemicals and groundwater will be thoroughly blended using an inline static mixer. This mixing approach is used to minimize the introduction of oxygen to the groundwater. After mixing, the groundwater will be piped and delivered to the injection wells.

A 5-micron filter will be used during the initial operation of the recirculation system to capture any particles flushed from the extraction well that were not removed during the development of the well. The filter will be removed during normal operation.

The sodium lactate solution tank will likely provide more than 7 days of storage. The sodium carbonate or bicarbonate tank will provide more than 4 days of storage.

Pressure gauges will be provided to monitor pressure drop through the system.

14.4.2 System Startup and Operation

The milestone operating and sampling events for the pilot study are summarized in Figure 4-3. The details of these activities are described below. A copy of an example daily log sheet of operational activities is provided in Appendix G. The actual log sheet will be developed after the specific system components are determined.

Equipment and System Inspection

After all of the equipment has been installed, the mechanical equipment (pumps and mixers) will be tested briefly to ensure operation.

The extraction well pumps will be tested by performing the following steps. Open the valve at the injection well, close the valves at the chemical injection points, close the control valves, and open the shut-off valve at the control valves. Activate the pump and slowly open the control valves while observing the flow meter. Gradually open the control valves until the predetermine pumping rate from the aquifer is reached while noting the number of turns on the control valves. Check the system for leaks. Then shut off the pump, and close the control valves.

The chemical feed pumps will be tested by performing the following steps. Disconnect the discharge hose and direct the discharge back into the appropriate feed tank. Operate the pumps long enough to confirm operation.

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #14 -- Summary of Project Tasks[\(UFP-QAPP Manual Section 2.8.1\)](#)Startup Operation

The systems operation details cannot be fully planned until the pumping rates are determined. However, the following actions generally describe the procedures to be performed. The recirculation system will be operated initially with no chemical addition until the aquifer within the pilot study attains steady-state water level conditions. The steady-state conditions will be determined by routinely measuring the water levels until they have stabilized. After the water levels have stabilized and steady-state conditions are achieved, the sodium carbonate chemical feed pump will be activated. The groundwater recirculation system will then be operated with sodium carbonate injections for several weeks (an estimated 2 weeks) to bring the pH of the area groundwater into a range suitable for microorganism growth. The levels in the chemical feed tanks will be monitored and recorded to confirm the feed rate. The water levels will be monitored daily in all Site 5 wells.

Chemical Addition Operation

After the groundwater pH levels have been brought to within a suitable range (6.0 – 8.0) within the injection well area, the sodium lactate chemical feed pump will be activated. Also, a decision will be made at this time whether to continue with sodium carbonate, or to switch to sodium bicarbonate. This decision will be based on the pH of the pilot study area and the amount of sodium bicarbonate that was needed to adjust the pH. Care must and will be taken to minimize aeration of the water when the tanks are filled with water and mixed with the chemicals. The agitation rate will be kept as low as possible and the tanks will be covered to minimize the amount of oxygen in the makeup water. The levels of the chemical feed tank will be noted and recorded daily.

The groundwater recirculation system will be operated with sodium lactate and sodium carbonate or bicarbonate injections for several weeks to maintain the pH of the pilot study area within a range suitable for microorganism growth, to create anaerobic conditions, and to monitor for TCE and 1,1,1-TCA daughter products. During the chemical addition operation, the levels in the chemical feed tanks will be monitored and recorded to confirm the feed rate. Water levels will be monitored in all wells.

Long-Term Operation

After the chemical addition is started, the pilot system will be visited 2 to 3 times a week, or at another frequency identified as appropriate for this specific system. Each time the site is visited, the following tasks will be performed and noted on a daily sheet which is included in Appendix G.

Approximately three months after the start of the chemical addition, the biological inoculum (Dhc and/or Dhb) will be added if identified as appropriate. Dhc and/or Dhb will be added if either no or only very low concentrations of TCE and 1,1,1-TCA daughter products are detected. If the addition of Dhc and/or Dhb is not required, then the operating instructions to be followed for the remainder of the pilot study will be the same as that previously described for the chemical addition operations.

If Dhc and/or Dhb inoculum is used, it will be added while under minimal exposure to oxygen. The inoculum is provided in special sealed containers. The container has a fitting for compressed inert gas (nitrogen or argon) and a vent valve to release gas from the container. A hose from the inert gas regulator is attached to the container and inert gas is purged through the inoculum in the container at 3 psig for 3 to 5 minutes. The injection wells will also be purged with the inert gas. After the container has been degassed, the inoculum can be injected.

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #14 -- Summary of Project Tasks[\(UFP-QAPP Manual Section 2.8.1\)](#)

After Dhc and/or Dhb is added, the recirculation system will continue to be visited 2 to 3 times a week, or on an as needed basis that is based on the continuing acquisition and interpretation of the groundwater data and on the pilot system performance. To minimize the potential of injection well clogging, the sodium lactate feed will be stopped for 4 to 6 hours on one day each week to flush the immediate vicinity of the injection well. Household bleach may be added to each well to reduce clogging if necessary.

The recirculation system will be operated for approximately 8 months. Chemical feed rates and the frequency and duration of chemical feeds may be modified over the course of the study based on the observations of monitoring well water levels and the results of the periodic groundwater analyses.

14.4.3 Shutdown and Restart

The pilot system is primarily a manually operated system, but is provided with some automation. To prevent the potential spilling of contaminated groundwater onto the ground surface, the system automatically shuts down if the water level in either injection well becomes too high. The chemical feed pumps will also shut down automatically if the extraction well pump overload protection device trips, which will prevent the overfeeding the chemicals.

Care must be taken following any shutdown because the individual pump switches may be "on", but power has been interrupted further up the line. When the power is restored or the system controls reset, the pumps may begin operating.

14.5 SAMPLE COLLECTION DOCUMENTATION, HANDLING, TRACKING, AND CUSTODY PROCEDURES

The following sections outline the procedures that will be used to document project activities and sample collection, handling, tracking, and custody procedures during performance monitoring tasks. Detailed and accurate documentation is necessary in order to ensure data integrity, authenticity, and defensibility.

Sample Collection Documentation

Samples will be collected following procedures outlined in Appendix A. The equipment used to collect the sample will be noted in the logbook, along with date and time of sampling, sampler's name, sample description, depth at which the sample was collected, and the volume and number of containers collected. QC sample information will be appropriately recorded. Measurements made will be recorded. All instruments used to make measurements will be identified, along with the date of calibration.

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #14 -- Summary of Project Tasks[\(UFP-QAPP Manual Section 2.8.1\)](#)

Standard log sheets will be used to record data and will include:

- Daily Activity Log
- Equipment Calibration Log
- Boring Log Sheet
- Groundwater Level Measurement Sheet
- Low Flow Purge Data Sheet
- Groundwater Sample Log sheet
- Soil Sample Log Sheet
- QC Sampling Log
- Well Inspection Log
- COC record

Log sheets will include entries in every blank, with appropriate use of the abbreviations NA (not applicable) and NR (not recorded). All "NR" entries should be accompanied by an explanation. All entries will be recorded in waterproof ink and signed and dated by the person making the entry. No erasures will be made. If an incorrect entry is made, the information will be crossed out with a single strike mark, the correct entry recorded, and the change initialed and dated by the person making the correction.

14.6 DATA MANAGEMENT TASKS

This section describes how project information will be managed, organized, and maintained for efficient use by project personnel. The information management process is outlined from data generation to ultimate storage.

14.6.1 Project Documentation and Records

A summary of project documentation and records to be generated and stored in the project files is provided in Worksheet 29.

14.6.2 Data Package Deliverables

Certain field measurements (i.e., photo-ionization detection, etc.) are made primarily for health and safety monitoring. Additional field measurements may include readings such as pH, temperature, and specific conductance to monitor ambient conditions prior to sample collection. These data will be recorded on the appropriate log sheets.

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #14 -- Summary of Project Tasks[\(UFP-QAPP Manual Section 2.8.1\)](#)

For the Site 5 pilot study sampling events, the fixed-base laboratories will provide Contract Laboratory Program (CLP) equivalent data packages for TCL volatiles analyses. The laboratory will provide abbreviated packages for the additional environmental indicators and MNA parameters. Additionally electronic deliverables, formatted according to the requirements stated in the laboratory subcontracts, will be provided by the laboratories for all analytical data. Worksheet No. 30 summarizes the analytical requirements.

14.6.3 Data Reporting Formats

Field data will be recorded on log sheets or in the project logbook. The laboratory will provide CLP equivalent data reporting forms 1 through 15 for the required metals and organic analyses presented in the previous paragraph.

14.6.4 Data Handling and Management

The data-handling procedures to be followed by the laboratories will meet the requirements in the laboratory subcontracts. All analytical and field data will be maintained in the project files. The project files will contain hard copies of the COC forms, sample log forms, sample location maps, and documentation of QA of data manipulation.

14.6.5 Data Tracking and Control

A "cradle-to-grave" sample tracking system will be used from the beginning to the end of the sampling event. Before field mobilization, the FOL will coordinate/initiate the sample tracking process. Sample jar labels will be hand-written in the field.

The labels will be reviewed for adherence to SAP requirements as well as for accuracy. The PM will coordinate with the analytical laboratories to ensure that they are aware of the number and type of samples and analyses.

When field sampling is underway, the FOL forwards the chain of custody (COC) forms to the PM or designee and the laboratories for each day that samples are collected. The PM or designee will confirm that the COC forms provide the information required by the SAP.

This will allow for early detection of errors made in the field so that adjustments can be made while the field team is mobilized. After successful completion of all requested analyses, the laboratories will submit an electronic deliverable for every sample delivery group (SDG). When all electronic deliverables have been received from the laboratories, the PM or designee will ensure that the laboratories performed all the requested analyses. Ideally, discrepancies can be noted early enough so that all samples can be analyzed within the prescribed holding times.

Sample Information

Data from field measurements will be recorded using the appropriate log sheets.

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #14 -- Summary of Project Tasks[\(UFP-QAPP Manual Section 2.8.1\)](#)

Reduction of field data entails the summarization and presentation of these data in tabular form. The reduction of laboratory data entails the manipulation of raw data instrument output into reportable results. Field data (e.g., photo-ionization detection) will be verified on a daily basis by the FOL. Laboratory data will be verified by the group supervisor and then by the laboratory's QC/Documentation Department.

For field data, the FOL will coordinate with the geographical information system (GIS) lead to ensure that all survey technical specifications are consistent with the underlying coordinate system in the GIS.

Electronic data arriving from the laboratories will pass through to the data validation manager (DVM) for database compilation and validation. The DVM will compile all the formatted laboratory electronic deliverables into a working project database. Data that are to be validated will be printed as data packages, which include the samples as part of each SDG and the appropriate analytical fraction. The data packages will be distributed to the appropriate data validators. The data validators will enter all data qualifiers and qualifier codes into the database and print out a hard copy and return it to the DVM. The DVM will check the data qualifiers and qualifier codes in the project database and print the final validated data for incorporation into the data validation letter. When all samples and analyses have been accounted for and validated, the PM will ensure that the analytical data are incorporated into the project database.

Project Data Compilation

The analytical laboratories will generate a PDF file of the analytical data packages, as well as electronic database deliverables. The electronic database will be checked against the PDF file provided by the laboratories and updated as required, based on data qualifier flags applied during the data validation process. The data generated during the implementation of the QAPP will be incorporated into the NAS JRB Willow Grove database and GIS. All data, such as units of measure and chemical nomenclature, will be manipulated to maintain consistency with the project database.

Geographical Information System

Data management systems consist of a relational database and GIS that are being used to manage environmental information pertaining to NAS JRB Willow Grove. The relational database stores chemical, geological, hydrogeologic, and other environmental data collected during environmental investigations. The GIS is built from the relational database and contains subsets of the larger data pool. Using the GIS, environmental data can be posted on base mapping to provide a graphical representation of the information.

Upon compilation of sample, chemical, biological, and positional data, the data will be compiled and incorporated into the NAS JRB Willow Grove GIS. The GIS system can be used to generate various maps for NAS JRB Willow Grove data including site location maps, sample location maps, and contaminant tag maps, as needed. The GIS software that is used will be documented in performance monitoring reports.

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #14 -- Summary of Project Tasks[\(UFP-QAPP Manual Section 2.8.1\)](#)**14.7 DATA REVIEW**

The internal data verification requirements for this project include the maintenance and periodic review of field documentation (i.e., site logbooks, instrument calibration logs, chain of custody forms (COC), field summary reports, and field modification records) and laboratory analytical data packages. After receipt of analytical laboratory results, TtNUS will perform data validation according to the requirements outlined in the Region 3 Modifications to the National Functional Guidelines for Organic Review (September 1994) and method-specific requirements to ensure that the analytical results meet the project quality objectives.

After the data are validated, a list of nonconformities will be generated. Nonconformities require data qualifiers, which are used to alert the data user to inaccurate or imprecise data. For situations in which several quality control criteria are out of specification with regard to the limits specified in the DoD QSM (January, 2006), the data validator may make professional judgments and/or comments on the validity of the overall data package. In situations where the validity of an entire data package is in question, it may be necessary for the sample(s) to be reanalyzed. The reviewer will then prepare a technical memorandum presenting changes in the data, if necessary, and the rationale for making such changes.

The net result is a data package that has been carefully reviewed for its adherence to prescribed requirements and is suitable for its intended use. Data validation therefore plays a major role in determining the confidence with which key technical evaluations may be made.

Data validation reports for all parameters will be generated according to the procedures described in Standard Operating Procedure (SOP) DV-02. The final data validation report will include a technical memorandum, qualified analytical results, results reported by the laboratory, and documentation to support data qualification. All data will be flagged by an appropriate qualifying symbol.

The data and field records will also be reviewed by project personnel to ensure that the samples represent the intended sampling conditions and populations. Data qualified during validation will be reviewed to assess the impact of the qualifiers on the attainment of project objectives.

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #15 -- Reference Limits and Evaluation Table[\(UFP-QAPP Manual Section 2.8.1\)](#)**SOIL LABORATORY LIMITS**

ORGANIC COMPOUNDS	CAS	Laboratory Method Detection Limit (MDL) (ug/kg)	Laboratory Quantitation Limit (QL) (ug/kg)
VOLATILES			
1,1,1-Trichloroethane	71-55-6	0.3	2
1,1,2,2-Tetrachloroethane	79-34-5	0.3	2
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	0.5	2
1,1,2-Trichloroethane	79-00-5	0.6	2
1,1-Dichloroethane	75-34-3	0.3	2
1,1-Dichloroethene	75-35-4	0.5	2
1,2,3-Trichlorobenzene	87-61-6	0.3	5
1,2,4-Trichlorobenzene	120-82-1	0.4	5
1,2-Dibromo-3-chloropropane	96-12-8	1	4
1,2-Dibromoethane	106-93-4	0.4	2
1,2-Dichlorobenzene	95-50-1	0.4	2
1,2-Dichloroethane	107-06-2	0.3	2
1,2-Dichloropropane	78-87-5	0.4	2
1,3-Dichlorobenzene	541-73-1	0.3	2
1,4-Dichlorobenzene	106-46-7	0.3	2
2-Butanone	78-93-3	6.1	20
2-Hexanone	591-78-6	1.8	10
4-Methyl-2-pentanone	108-10-1	2.1	10
Acetone	67-64-1	5.2	20
Benzene	71-43-2	0.4	2

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #15 -- Reference Limits and Evaluation Table[\(UFP-QAPP Manual Section 2.8.1\)](#)**SOIL LABORATORY LIMITS**

ORGANIC COMPOUNDS	CAS	Laboratory MDL (ug/kg)	Laboratory QL (ug/kg)
VOLATILES			
Bromochloromethane	74-97-5	0.5	2
Bromodichloromethane	75-27-4	0.4	2
Bromoform	75-25-2	0.6	2
Bromomethane	74-83-9	1.2	4
Carbon disulfide	75-15-0	0.7	4
Carbon tetrachloride	56-23-5	0.3	2
Chlorobenzene	108-90-7	0.3	2
Chloroethane	75-00-3	0.4	2
Chloroform	67-66-3	0.5	2
Chloromethane	74-87-3	0.3	2
cis-1,2-Dichloroethene	156-59-2	0.4	2
cis-1,3-Dichloropropene	10061-01-5	0.3	2
Cyclohexane	108-94-1	0.3	2
Dibromochloromethane	124-48-1	0.3	2
Dichlorodifluoromethane	75-71-8	0.3	2
Ethylbenzene	100-41-4	0.2	2
Isopropylbenzene	98-82-8	0.2	2
m,p-Xylene	179601-23-1	0.5	4
Methyl acetate	79-20-9	0.7	4
Methyl tert-butyl ether	1634-04-4	0.5	2
Methylcyclohexane	108-87-2	0.4	2

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #15 -- Reference Limits and Evaluation Table[\(UFP-QAPP Manual Section 2.8.1\)](#)**SOIL LABORATORY LIMITS**

ORGANIC COMPOUNDS	CAS	Laboratory MDL (ug/kg)	Laboratory QL (ug/kg)
VOLATILES			
Methylene chloride	75-09-2	1.3	5
o-Xylene	95-47-6	0.3	2
Styrene	100-42-5	0.3	2
Tetrachloroethene	127-18-4	0.2	2
Toluene	108-88-3	0.6	2
trans-1,2-Dichloroethene	156-60-5	0.4	2
trans-1,3-Dichloropropene	10061-02-6	0.3	2
Trichloroethene	79-01-6	0.3	2
Trichlorofluoromethane	75-69-4	0.3	2
Vinyl chloride	75-01-4	0.4	2

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #15 -- Reference Limits and Evaluation Table[\(UFP-QAPP Manual Section 2.8.1\)](#)**GROUNDWATER REFERENCE LIMITS AND LABORATORY LIMITS**

ORGANIC COMPOUNDS (SW-846 8260B)	CAS	Project Action Level Federal MCLs¹ (µg/L)	Laboratory MDL (µg/L)	Laboratory QL (µg/L)
VOLATILES				
1,1,1-Trichloroethane	71-55-6	200	0.2	1
1,1,2,2-Tetrachloroethane	79-34-5	na	0.3	1
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	na	0.4	2
1,1,2-Trichloroethane	79-00-5	5	0.4	1
1,1-Dichloroethane	75-34-3	na	0.3	1
1,1-Dichloroethene	75-35-4	7	0.3	1
1,2,3-Trichlorobenzene	87-61-6	na	0.6	2
1,2,4-Trichlorobenzene	120-82-1	70	0.3	2
1,2-Dibromo-3-chloropropane	96-12-8	na	2	7
1,2-Dibromoethane	106-93-4	na	0.3	1
1,2-Dichlorobenzene	95-50-1	600	0.2	1
1,2-Dichloroethane	107-06-2	5	0.2	1
1,2-Dichloropropane	78-87-5	na	0.3	1
1,3-Dichlorobenzene	541-73-1	na	0.2	1
1,4-Dichlorobenzene	106-46-7	75	0.3	1
2-Butanone	78-93-3	na	3	10
2-Hexanone	591-78-6	na	2	10
4-Methyl-2-pentanone	108-10-1	na	2	10
Acetone	67-64-1	na	3	10
Benzene	71-43-2	5	0.2	1

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #15 -- Reference Limits and Evaluation Table[\(UFP-QAPP Manual Section 2.8.1\)](#)**GROUNDWATER REFERENCE LIMITS AND LABORATORY LIMITS**

ORGANIC COMPOUNDS (SW-846 8260B)	CAS	Project Action Level Federal MCLs¹ (µg/L)	Laboratory MDL (µg/L)	Laboratory QL (µg/L)
VOLATILES				
Bromochloromethane	74-97-5	na	0.4	2
Bromodichloromethane	75-27-4	na	0.3	1
Bromoform	75-25-2	na	0.4	2
Bromomethane	74-83-9	na	0.6	2
Carbon disulfide	75-15-0	na	0.3	1
Carbon tetrachloride	56-23-5	5	0.4	1
Chlorobenzene	108-90-7	100	0.3	1
Chloroethane	75-00-3	na	0.3	1
Chloroform	67-66-3	na	0.2	1
Chloromethane	74-87-3	na	0.3	1
cis-1,2-Dichloroethene	156-59-2	70	0.3	1
cis-1,3-Dichloropropene	10061-01-5	na	0.3	1
Cyclohexane	108-94-1	na	0.3	1
Dibromochloromethane	124-48-1	na	0.3	1
Dichlorodifluoromethane	75-71-8	na	0.2	1
Ethylbenzene	100-41-4	700	0.3	1
Isopropylbenzene	98-82-8	na	0.3	1
m,p-Xylene	179601-23-1	10	0.4	2
Methyl acetate	79-20-9	na	0.8	3
Methyl tert-butyl ether	1634-04-4	na	0.2	1
Methylcyclohexane	108-87-2	na	0.3	1

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #15 -- Reference Limits and Evaluation Table[\(UFP-QAPP Manual Section 2.8.1\)](#)**GROUNDWATER REFERENCE LIMITS AND LABORATORY LIMITS**

ORGANIC COMPOUNDS (SW-846 8260B)	CAS	Project Action Level Federal MCLs¹ (µg/L)	Laboratory MDL (µg/L)	Laboratory QL (µg/L)
VOLATILES				
Methylene chloride	75-09-2	na	0.3	1
o-Xylene	95-47-6	10	0.3	1
Styrene	100-42-5	100	0.2	1
Tetrachloroethene	127-18-4	5	0.4	1
Toluene	108-88-3	1000	0.2	1
trans-1,2-Dichloroethene	156-60-5	100	0.2	1
trans-1,3-Dichloropropene	10061-02-6	na	0.2	1
Trichloroethene	79-01-6	5	0.3	1
Trichlorofluoromethane	75-69-4	na	0.3	1
Vinyl chloride	75-01-4	2	0.6	2

INORGANIC ANALYTES (SW-846 6010B)	CAS	Project Action Level (µg/L)	Laboratory MDL (µg/L)	Laboratory QL (µg/L)
Iron (dissolved)	7439-89-6	na	17	70
Manganese (dissolved)	7439-96-5	na	0.3	6
Sodium (Total)	7440-23-5	na	30	560

OTHER ANALYTES	CAS	Project Action Level	Laboratory MDL	Laboratory QL
Total Organic Carbon (EPA 415.1) (mg/L)	---	na	0.25	5
Sulfide (EPA 376.1) (mg/L)	18496-25-8	na	0.3	1

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #15 -- Reference Limits and Evaluation Table[\(UFP-QAPP Manual Section 2.8.1\)](#)**GROUNDWATER REFERENCE LIMITS AND LABORATORY LIMITS**

OTHER ANALYTES	CAS	Project Action Level	Laboratory MDL	Laboratory QL
Anions (EPA 300.0) (mg/L)				
Chloride	16887-00-6	na	0.1	1
Nitrate	14797-55-8	na	0.01	0.1
Nitrite	14797-65-0	na	0.01	0.1
Sulfate	14808-79-8	na	0.1	1
Sulfide	18496-25-8	na	0.003	0.02
Orthophosphate	---	na		
Metabolic Acids (Laboratory SOP) (mg/L)				
Lactic	50-21-5	na	0.067	0.1
Pyruvic	127-17-3	na	0.067	0.07
Acetic	64-19-7	na	0.041	0.07
Propionic	79-09-4	na	0.052	0.07
Butyric	107-92-6	na	0.059	0.07
Dissolved Gases (RSK SOPs 147/175) (ug/L)				
Methane	74-82-8	na	0.0171	0.1
Ethane	74-84-0	na	0.0035	0.025
Ethene	74-85-1	na	0.0046	0.025
Acetylene	74-86-2	na	0.073	0.5
PCR/Enzymes (Laboratory SOP) (gene copies)				
TCE reductase	---	na	500	1000
BAV1 VC reductase	---	na	500	1000
VC reductase	---	na	500	1000

¹ National Primary Drinking Water Maximum Contaminant Levels (MCLs), June 2003. <http://www.epa.gov/safewater/contaminants/index.html>

NA – Not applicable

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #16 -- Project Schedule / Timeline Table (optional format)[\(UFP-QAPP Manual Section 2.8.2\)](#)

Activities	Organization	Dates (MM/DD/YY)		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
Project Plans	TtNUS	7/21/07	8/08	Final SAP	8/08
Soil sampling	TtNUS	April 2008	April 2008		
EVENT 1 - Preliminary Groundwater Sampling	TtNUS	May 2008	July 2008	Letter Addendum Work Plan and possible SAP amendment	9/08
EVENT 2 (Baseline) Groundwater Sampling	TtNUS	October 2008	October 2008		
EVENT 3 (1 mo. after start) Groundwater Sampling	TtNUS	November 2008	November 2008		
EVENT 4 (3 mo. after start, Innoculum Baseline) Groundwater Sampling	TtNUS	January 2008	January 2008		
EVENT 5 (4.5 mo. after start, 6 weeks after innoculum injection) Groundwater Sampling	TtNUS	March 2009	March 2009		
EVENT 6 (6 mo. after start, 12 weeks after innoculum injection) Groundwater Sampling	TtNUS	April 2009	April 2009		
EVENT 7 (Post study, 9 mo. after start, 24 weeks after innoculum injection) Groundwater Sampling	TtNUS	July 2009	July 2009		

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

Activities	Organization	Dates (MM/DD/YY)		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
EVENT 8 (Post study, 12 mo. after start, 40 weeks after innoculum injection) Groundwater Sampling	TtNUS	October 2009	October 2009	Internal Draft Report Draft Report Final Report	3/1/10 4/22/10 6/30/10

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5
Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #17 -- Sampling Design and Rationale

[\(UFP-QAPP Manual Section 3.1.1\)](#)

17.1 SAMPLING PROCEDURES

This section details the field sampling procedures to be used for the collection of pilot study groundwater samples. TtNUS SOPs can be found in Appendix A of the SAP.

17.1.1 Groundwater Sampling

The groundwater sampling and analysis program is presented in Worksheet 18, which includes the wells to be sampled and the parameters to be analyzed for each sampling round. The existing site wells to be sampled were chosen because of their proximity to the source area and the recirculation system. The new monitoring wells will be located in the immediate proximity of the source area and recirculation system, where the effects of the bioremediation are expected to be most pronounced. The rationale for each new monitoring well location is explained in detail in Worksheet 14, Section 14.2.1. The groundwater sampling schedule is illustrated in Figure 4-3 and Worksheet 16, and follows the approximate schedule described below. The continuing evaluation of the system's performance may result in the adjustment of this schedule.

Round 1	Phase I (Pre-Treatability Study Investigation)
Round 2	Baseline - Immediately prior to groundwater recirculation and substrate injection
Round 3	4 weeks after the startup of groundwater recirculation and substrate injection
Round 4	12 weeks after the startup of groundwater recirculation and substrate injection (innoculum baseline round)
Round 5	6 weeks after innoculum injection (approximately 4.5 months after startup of recirculation and substrate injection)
Round 6	32 weeks after innoculum injection (approximately 8 months after startup of recirculation and substrate injection). SYSTEM SHUTDOWN DECISION ROUND
Round 7	44 weeks after innoculum injection (approximately 11.0 months after startup of recirculation and substrate injection. POST SHUTDOWN MONITORING
Round 8	56 weeks after innoculum injection (approximately 13.0 months after startup of recirculation and substrate injection) POST SHUTDOWN MONITORING

Groundwater samples will be obtained by the low-flow purging and sampling method following EPA Region 3 guidelines (Bulletin QAD023 - June 16, 1999). The new monitoring wells will be sampled a minimum of 2 weeks after their development. The microbial samples will be obtained through the laboratories specifically prescribed sampling technique included as Appendix B.

Low-flow sampling in wells with more than one water bearing fracture present in the open or screened interval will adhere to a purging and sampling method using a low flow submersible type pump to purge at least one well volume prior to sampling the well with the pump set a the lowest setting.

In instances where the monitoring well open interval has only one discreet water-bearing zone, low-flow purging and sampling using EPA Directive QAD023-June 16, 1999 will be performed, with the following additional tasks:

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

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

- TtNUS will determine the appropriate location of the pump within the vertical interval of the well screen based on the review of the drilling logs and geophysical logs. The pump will be positioned at the same depth as the most significant (highest yielding) fracture within the screen interval.
- A groundwater sampling form (log) will be completed to record the stabilization parameters, the volume purged, etc.
- The technical team will determine the appropriate location of the pump within the vertical interval of the well intake based on the drilling and geophysical logs and packer testing results.

A complete round of synoptic water levels will be collected from the existing and newly installed wells prior to purging and sampling. Groundwater samples will be field analyzed using test methods for parameters summarized in Worksheet 18 and 19. After the data are collected and analyzed, it will be interpreted to evaluate groundwater recirculation and substrate injection effectiveness.

17.1.2 PCR Sample Collection

Samples will be collected for PCR analysis according to directions provided by Microbial Insights, Inc. The PCR sample is collected by pumping water from the sampling pump discharge through a filter provided by the laboratory. One to two liters of water will be pumped through the filter and the volume that is pumped through the filter will be measured and recorded. The instructions and sample COC form are included in Appendix B.

17.1.3 Soil Sample Collection

Twenty-four soil samples will be collected at 12 locations using direct-push techniques during the Phase 1 (Pre-Treatability Study) Investigation. The locations of the soil borings are illustrated in Figures 1-6 and 2-1. The soil boring locations were chosen using the analytical results of previous soil borings and using the results of historical aerial photo analysis that delineated the extent of the source area (see Worksheet 11). The soil samples will be collected for volatile analysis using Encore samplers. The sampling procedures are discussed in detail in Worksheet 14. The decision matrix for selecting which samples are to be submitted to the laboratory (based on the PID response to the soil and the vertical position of the sample within the boring) is included as Figure 4-4. The TtNUS SOPs for soil sample collection are included in Appendix A.

17.1.4 QA/QC Samples

To assure that data obtained during the investigation are accurate, various quality assurance/quality control (QA/QC) requirements have been established for fieldwork, laboratory analysis of the collected samples, and validation of the analytical results obtained from the laboratory.

Field QC samples expected to be collected during the investigation are field duplicates, laboratory matrix spikes and matrix spike duplicates, (MS/MSD), trip blanks, and rinsate blanks. Worksheet 20 summarizes the frequency and type of field QA/QC samples to be collected for this sampling program. These requirements do not apply to the field parameters, natural attenuation parameters, and PCR analyses.

Data validation procedures are discussed in Worksheets 34 through 36. Full data validation will be performed on volatile organic compound samples only.

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5
 Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #18 -- Sampling Locations and Methods/SOP Requirements Table
 (UFP-QAPP Manual Section 3.1.1)

Location	Sample Designation	Sampling SOP	Fixed Base Laboratory							Process Test					Field Test						
			TCL VOCs	Dissolved Gases ⁽¹⁾	Iron and Manganese (Dissolved)	Sodium (Total)	Sulfide	Miscellaneous Anions ⁽²⁾	TOC	Metabolic Acids ⁽³⁾	PCR and genes ⁽⁴⁾	Water Level	Temperature	pH	Specific Conductivity	Oxidation / Reduction Potential	Dissolved Oxygen	DO	Dissolved CO ₂	Alkalinity as (CaCO ₃)	Iron (Ferrous)
Event 1 - Pre Treatability Study Sample Round																					
05SB60	05SB60-XXXX ⁽⁵⁾	SA-2.5 and	•																		
	05SB60-XXXX		•																		
05SB61	05SB61-XXXX	SA-1.3	•																		
	05SB61-XXXX		•																		
05SB62	05SB62-XXXX		•																		
	05SB62-XXXX		•																		
05SB63	05SB63-XXXX		•																		
	05SB63-XXXX		•																		
05SB64	05SB64-XXXX		•																		
	05SB64-XXXX+Duplicate		•																		
05SB65	05SB65-XXXX		•																		
	05SB65-XXXX		•																		
05SB66	05SB66-XXXX		•																		
	05SB66-XXXX		•																		
05SB67	05SB67-XXXX		•																		
	05SB67-XXXX		•																		
05SB68	05SB68-XXXX		•																		
	05SB68-XXXX		•																		
05SB69	05SB69-XXXX		•																		
	05SB69-XXXX+Duplicate		•																		
05SB70	05SB70-XXXX		•																		
	05SB70-XXXX		•																		

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5
 Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

Location	Sample Designation	Sampling SOP	Fixed Base Laboratory								Process Test					Field Test					
			TCL VOCs	Dissolved Gases ⁽¹⁾	Iron and Manganese (Dissolved)	Sodium (Total)	Sulfide	Miscellaneous Anions ⁽²⁾	TOC	Metabolic Acids ⁽³⁾	PCR and genes ⁽⁴⁾	Water Level	Temperature	pH	Specific Conductivity	Oxidation / Reduction Potential	Dissolved Oxygen	DO	Dissolved CO ₂	Alkalinity as (CaCO ₃)	Iron (Ferrous)
05SB71	05SB71-XXXX	SA-1.1	•																		
	05SB71-XXXX+Duplicate		•																		
05MW01S	05MW01S-GW-02		•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•		
05MW01SI	05MW01SI-GW-02		•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•		
05MW16S	05MW16S-GW-01		•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•		
05MW17S	05MW17S-GW-01		•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•		
05MW17I	05MW17I-GW-01		•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•		
05MW18S	05MW18S-GW-01		•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•		
05MW18I	05MW18I-GW-01		•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•		
05MW19S	05MW19S-GW-01		•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•		
05MW19I	05MW19I-GW-01 + Duplicate*	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•			
Event 2 - Baseline Samples																					
05MW01S	05MW01S-GW-02	SA-1.1	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•
05MW01SI	05MW01SI-GW-02		•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•
05MW01I	05MW01I-GW-02		•																		
05MW10I	05MW10I-GW-02		•																		
05MW16S	05MW16S-GW-02		•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•
05MW17S	05MW17S-GW-02		•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•
05MW17I	05MW17I-GW-02		•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•
05MW18S	05MW18S-GW-02		•																		
05MW18I	05MW18I-GW-02		•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•
05MW19S	05MW19S-GW-02 + Duplicate*		•																		
05MW19I	05MW19I-GW-02		•																		
05IW01	05IW01-GW-02		•	•								•	•	•	•	•	•	•	•		
05IW01	05IW01-GW-02		•	•								•	•	•	•	•	•	•	•		

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5
 Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

Location	Sample Designation	Sampling SOP	Fixed Base Laboratory								Process Test					Field Test						
			TCL VOCs	Dissolved Gases ⁽¹⁾	Iron and Manganese (Dissolved)	Sodium (Total)	Sulfide	Miscellaneous Anions ⁽²⁾	TOC	Metabolic Acids ⁽³⁾	PCR and genes ⁽⁴⁾	Water Level	Temperature	pH	Specific Conductivity	Oxidation / Reduction Potential	Dissolved Oxygen	DO	Dissolved CO ₂	Alkalinity as (CaCO ₃)	Iron (Ferrous)	Hydrogen Sulfide
05EW01	05EW01-GW-02		•	•							•	•	•	•	•	•	•	•				
05EW01	05EW01-GW-02 + Duplicate*		•	•							•	•	•	•	•	•	•	•				
System Check (Twice a week for length of Study at the discretion of the tech lead and field technician)																						
05MW01S	05MW01S-YYYYMMDD	SA-1.1									•	•	•	•	•	•	•	•				
05MW01SI	05MW01SI-YYYYMMDD											•	•	•	•	•	•	•	•			
05MW01I	05MW01I-YYYYMMDD																					
05MW10I	05MW10I-YYYYMMDD																					
05MW16S	05MW16S-YYYYMMDD											•	•	•	•	•	•	•	•			
05MW17S	05MW17S-YYYYMMDD																					
05MW17I	05MW17I-YYYYMMDD																					
05MW18S	05MW18S-YYYYMMDD																					
05MW18I	05MW18I-YYYYMMDD																					
05MW19S	05MW19S-YYYYMMDD																					
05MW19I	05MW19I-YYYYMMDD																					
05IW01	05IW01-YYYYMMDD											•	•	•	•	•	•	•	•			
05IW01	05IW01-YYYYMMDD											•	•	•	•	•	•	•	•			
05EW01	05EW01-YYYYMMDD											•	•	•	•	•	•	•	•			
05EW01	05EW01-YYYYMMDD											•	•	•	•	•	•	•	•			
Event 3 - 4 weeks after chem feed start (1 month after Pilot study start)																						
05MW01S	05MW01S-GW-03	SA-1.1	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	
05MW01SI	05MW01SI-GW-03		•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•
05MW01I	05MW01I-GW-03																					
05MW10I	05MW10I-GW-03																					
05MW16S	05MW16S-GW-03		•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•
05MW17S	05MW17S-GW-03		•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•
05MW17I	05MW17I-GW-03		•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5
 Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

Location	Sample Designation	Sampling SOP	Fixed Base Laboratory								Process Test						Field Test				
			TCL VOCs	Dissolved Gases ⁽¹⁾	Iron and Manganese (Dissolved)	Sodium (Total)	Sulfide	Miscellaneous Anions ⁽²⁾	TOC	Metabolic Acids ⁽³⁾	PCR and genes ⁽⁴⁾	Water Level	Temperature	pH	Specific Conductivity	Oxidation / Reduction Potential	Dissolved Oxygen	DO	Dissolved CO ₂	Alkalinity as (CaCO ₃)	Iron (Ferrous)
05MW18S	05MW18S-GW-03	SA-1.1																			
05MW18I	05MW18I-GW-03		•	•	•	•	•	•	•		•	•	•	•	•	•	•	•	•	•	•
05MW19S	05MW19S-GW-03																				
05MW19I	05MW19I-GW-03																				
05IW01	05IW01-GW-03		•	•							•	•	•	•	•	•	•	•	•		
05IW01	05IW01-GW-03		•	•							•	•	•	•	•	•	•	•	•		
05EW01	05EW01-GW-03		•	•							•	•	•	•	•	•	•	•	•		
05EW01	05EW01-GW-03 + Duplicate*		•	•							•	•	•	•	•	•	•	•	•		
Event 4 - 12 weeks after chem feed start / Innoculum Baseline (3 months after Pilot study start)																					
05MW01S	05MW01S-GW-04	SA-1.1	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•
05MW01SI	05MW01SI-GW-04		•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•	•
05MW01I	05MW01I-GW-04																				
05MW10I	05MW10I-GW-04																				
05MW16S	05MW16S-GW-04		•	•	•	•	•	•	•	•		•	•	•	•	•	•	•	•	•	•
05MW17S	05MW17S-GW-04		•	•	•	•	•	•	•	•		•	•	•	•	•	•	•	•	•	•
05MW17I	05MW17I-GW-04		•	•	•	•	•	•	•	•		•	•	•	•	•	•	•	•	•	•
05MW18S	05MW18S-GW-04		•	•	•	•	•	•	•	•		•	•	•	•	•	•	•	•	•	•
05MW18I	05MW18I-GW-04		•	•	•	•	•	•	•	•		•	•	•	•	•	•	•	•	•	•
05MW19S	05MW19S-GW-04		•									•	•	•	•	•	•	•	•		
05MW19I	05MW19I-GW-04		•									•	•	•	•	•	•	•	•		
05IW01	05IW01-GW-04 + Duplicate*		•	•							•	•	•	•	•	•	•	•	•		
05IW01	05IW01-GW-04		•	•							•	•	•	•	•	•	•	•	•		
05EW01	05EW01-GW-04		•	•							•	•	•	•	•	•	•	•	•		
05EW01	05EW01-GW-04		•	•							•	•	•	•	•	•	•	•	•		
Event 5 - 6 weeks after Innoculum injection (4.5 months after Pilot study start)																					

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5
 Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

Location	Sample Designation	Sampling SOP	Fixed Base Laboratory									Process Test					Field Test					
			TCL VOCs	Dissolved Gases ⁽¹⁾	Iron and Manganese (Dissolved)	Sodium (Total)	Sulfide	Miscellaneous Anions ⁽²⁾	TOC	Metabolic Acids ⁽³⁾	PCR and genes ⁽⁴⁾	Water Level	Temperature	pH	Specific Conductivity	Oxidation / Reduction Potential	Dissolved Oxygen	DO	Dissolved CO ₂	Alkalinity as (CaCO ₃)	Iron (Ferrous)	Hydrogen Sulfide
05MW01S	05MW01S-GW-05	SA-1.1	•	•	•	•	•	•	•	•		•	•	•	•	•	•	•	•	•	•	
05MW01SI	05MW01SI-GW-05		•	•	•	•	•	•	•	•		•	•	•	•	•	•	•	•	•	•	•
05MW01I	05MW01I-GW-05																					
05MW10I	05MW10I-GW-05																					
05MW16S	05MW16S-GW-05		•	•	•	•	•	•	•	•		•	•	•	•	•	•	•	•	•	•	•
05MW17S	05MW17S-GW-05		•	•	•	•	•	•	•	•		•	•	•	•	•	•	•	•	•	•	•
05MW17I	05MW17I-GW-05		•	•	•	•	•	•	•	•		•	•	•	•	•	•	•	•	•	•	•
05MW18S	05MW18S-GW-05																					
05MW18I	05MW18I-GW-05		•	•	•	•	•	•	•	•		•	•	•	•	•	•	•	•	•	•	•
05MW19S	05MW19S-GW-05																					
05MW19I	05MW19I-GW-05																					
05IW01	05IW01-GW-05 + Duplicate*		•	•								•	•	•	•	•	•	•	•			
05IW01	05IW01-GW-05		•	•								•	•	•	•	•	•	•	•			
05EW01	05EW01-GW-05		•	•								•	•	•	•	•	•	•	•			
05EW01	05EW01-GW-05 + Duplicate*	•	•								•	•	•	•	•	•	•	•				
Event 6 - 12 weeks after Inoculum injection (6.0 months after Pilot study start)																						
05MW01S	05MW01S-GW-06	SA-1.1	•	•	•	•	•	•	•	•		•	•	•	•	•	•	•	•	•	•	
05MW01SI	05MW01SI-GW-06		•	•	•	•	•	•	•	•		•	•	•	•	•	•	•	•	•	•	•
05MW01I	05MW01I-GW-06																					
05MW10I	05MW10I-GW-06																					
05MW16S	05MW16S-GW-06		•	•	•	•	•	•	•	•		•	•	•	•	•	•	•	•	•	•	•
05MW17S	05MW17S-GW-06		•	•	•	•	•	•	•	•		•	•	•	•	•	•	•	•	•	•	•
05MW17I	05MW17I-GW-06		•	•	•	•	•	•	•	•		•	•	•	•	•	•	•	•	•	•	•
05MW18S	05MW18S-GW-06																					
05MW18I	05MW18I-GW-06		•	•	•	•	•	•	•	•		•	•	•	•	•	•	•	•	•	•	•

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5
 Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

Location	Sample Designation	Sampling SOP	Fixed Base Laboratory								Process Test						Field Test					
			TCL VOCs	Dissolved Gases ⁽¹⁾	Iron and Manganese (Dissolved)	Sodium (Total)	Sulfide	Miscellaneous Anions ⁽²⁾	TOC	Metabolic Acids ⁽³⁾	PCR and genes ⁽⁴⁾	Water Level	Temperature	pH	Specific Conductivity	Oxidation / Reduction Potential	Dissolved Oxygen	DO	Dissolved CO ₂	Alkalinity as (CaCO ₃)	Iron (Ferrous)	Hydrogen Sulfide
05MW19S	05MW19S-GW-06	SA-1.1																				
05MW19I	05MW19I-GW-06																					
05IW01	05IW01-GW-06		•	•							•	•	•	•	•	•	•	•	•			
05IW01	05IW01-GW-06		•	•							•	•	•	•	•	•	•	•	•			
05EW01	05EW01-GW-06		•	•							•	•	•	•	•	•	•	•	•			
05EW01	05EW01-GW-06 + Duplicate*		•	•							•	•	•	•	•	•	•	•	•			
Event 7 - 24 weeks after Inoculum injection (9.0 months after Pilot study start)																						
05MW01S	05MW01S-GW-07	SA-1.1	•	•	•	•	•	•	•		•	•	•	•	•	•	•	•	•	•	•	
05MW01SI	05MW01SI-GW-07		•	•	•	•	•	•	•	•		•	•	•	•	•	•	•	•	•	•	•
05MW01I	05MW01I-GW-07		•									•	•	•	•	•	•	•	•			
05MW10I	05MW10I-GW-07		•									•	•	•	•	•	•	•	•			
05MW16S	05MW16S-GW-07		•	•	•	•	•	•	•	•		•	•	•	•	•	•	•	•	•	•	•
05MW17S	05MW17S-GW-07		•	•	•	•	•	•	•	•		•	•	•	•	•	•	•	•	•	•	•
05MW17I	05MW17I-GW-07		•	•	•	•	•	•	•	•		•	•	•	•	•	•	•	•	•	•	•
05MW18S	05MW18S-GW-07		•									•	•	•	•	•	•	•	•	•		
05MW18I	05MW18I-GW-07		•	•	•	•	•	•	•	•		•	•	•	•	•	•	•	•	•	•	•
05MW19S	05MW19S-GW-07 + Duplicate*		•									•	•	•	•	•	•	•	•	•		
05MW19I	05MW19I-GW-07		•									•	•	•	•	•	•	•	•	•		
05IW01	05IW01-GW-07		•	•								•	•	•	•	•	•	•	•	•		
05IW01	05IW01-GW-07		•	•								•	•	•	•	•	•	•	•	•		
05EW01	05EW01-GW-07		•	•								•	•	•	•	•	•	•	•	•		
05EW01	05EW01-GW-07 + Duplicate*		•	•								•	•	•	•	•	•	•	•	•		
Event 8 - 40 weeks after Inoculum injection (12.0 months after Pilot study start)																						
05MW01S	05MW01S-GW-08	SA-1.1	•	•	•	•	•	•	•	•		•	•	•	•	•	•	•	•	•	•	
05MW01SI	05MW01SI-GW-08		•	•	•	•	•	•	•	•	•		•	•	•	•	•	•	•	•	•	•

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5
 Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

Location	Sample Designation	Sampling SOP	Fixed Base Laboratory								Process Test					Field Test						
			TCL VOCs	Dissolved Gases ⁽¹⁾	Iron and Manganese (Dissolved)	Sodium (Total)	Sulfide	Miscellaneous Anions ⁽²⁾	TOC	Metabolic Acids ⁽³⁾	PCR and genes ⁽⁴⁾	Water Level	Temperature	pH	Specific Conductivity	Oxidation / Reduction Potential	Dissolved Oxygen	DO	Dissolved CO ₂	Alkalinity as (CaCO ₃)	Iron (Ferrous)	Hydrogen Sulfide
05MW011	05MW011-GW-08	SA-1.1	•								•	•	•	•	•	•	•	•				
05MW10I	05MW10I-GW-08		•								•	•	•	•	•	•	•	•				
05MW16S	05MW16S-GW-08		•	•	•	•	•	•	•		•	•	•	•	•	•	•	•	•	•	•	•
05MW17S	05MW17S-GW-08		•	•	•	•	•	•	•		•	•	•	•	•	•	•	•	•	•	•	•
05MW17I	05MW17I-GW-08		•	•	•	•	•	•	•		•	•	•	•	•	•	•	•	•	•	•	•
05MW18S	05MW18S-GW-08		•								•	•	•	•	•	•	•	•	•			
05MW18I	05MW18I-GW-08		•	•	•	•	•	•	•		•	•	•	•	•	•	•	•	•	•	•	•
05MW19S	05MW19S-GW-08 + Duplicate*		•								•	•	•	•	•	•	•	•	•			
05MW19I	05MW19I-GW-08		•								•	•	•	•	•	•	•	•	•			
05IW01	05IW01-GW-08		•	•						•	•	•	•	•	•	•	•	•	•			
05IW01	05IW01-GW-08		•	•							•	•	•	•	•	•	•	•	•			
05EW01	05EW01-GW-08		•	•							•	•	•	•	•	•	•	•	•			
05EW01	05EW01-GW-08 + Duplicate*		•	•							•	•	•	•	•	•	•	•	•			

1 Dissolved gases include ethane, ethene, methane, and acetylene.

2 Miscellaneous anions include sulfate, sulfide, nitrate, nitrite, chloride, phosphate.

3 Metabolic Acids include lactic, pyruvic, acetic, propionic, and butyric.

4 Functional genes include TCE reductase, BAV1 VC reductase, and VC reductase.

5 Soil sample depths will be determined in the field. Sample designation will include top and bottom depths. For example, a 0-1 foot depth will be recorded as 0001.

PCR = Polymerase chain reaction.

VOC = Volatile organic compounds.

*Field QC samples will be collected for VOC analysis only as follows: Field duplicates are collected every 10 environmental samples. Trip blanks will be shipped in each cooler containing VOC samples. Matrix spikes (MS) and matrix spike duplicates (MSD) will be determined in the field. MS/MSD samples will be collected at a rate of 1 per 20 environmental samples. Rinsate blanks will be collected at a rate of 1 per 20 environmental samples per sampling equipment.

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

IDW SAMPLING

Sampling Location / ID Number	Matrix	Depth	Analytical Group*	Number of Samples*	Sampling SOP Reference
IDW-SO-01	Soil	NA	Hazardous Waste Characterization including the suite of TCLP parameters and characteristics identified in 40 CFR Part 261.	1	
IDW-GW-01	Groundwater	NA	Hazardous Waste Characterization including the suite of TCLP parameters and characteristics identified in 40 CFR Part 261.	1	

* The hazardous waste characterization analyses will be handled by the IDW subcontractor. The number and matrices of IDW samples will be determined by the IDW subcontractor.

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5
 Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study
 Revision Number: 0
 Revision Date: September 2008

SAP Worksheet #19 -- Analytical SOP Requirements Table
 (UFP-QAPP Manual Section 3.1.1)

Matrix	Analytical Group	Analytical and Preparation Method / SOP Reference ¹	Containers (number, size, and type)	Sample volume ² (units)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time ³ (preparation / analysis)
Soil	TCL VOC	SW-846 5035/8260B ALSI SOP-02-5035 ALSI SOP-02-8260B	3 – 5 gram Encore samplers	15 grams	Cool to 4°C	48 hours to preparation; 14 days to analysis
Groundwater	TCL VOC	SW-846 8260B	3 – 40 mL glass vials with Teflon septum	120 mL	Cool to 4°C with HCl to pH ≤ 2; no headspace	14 days to analysis
	Total Sodium	SW-846 6010B	1 – 500 mL HDPE bottle	500 mL	Cool to 4°C with HNO ₃ to pH ≤ 2	180 days to analysis
	Dissolved Manganese and Iron	SW-846 6010B	1 – 500 mL HDPE bottle	500 mL	Cool to 4°C with HNO ₃ to pH ≤ 2	180 days to analysis
	TOC	EPA 415.1 Microseeps SOP – WC21	1 – 100 mL Polypropylene bottle	100 mL	Cool to 4°C, H ₂ SO ₄ or H ₃ PO ₄ to pH<2	28 days to analysis
	Sulfide	EPA 376.1 ALSI SOP-04-S	1 – .5L Polypropylene bottle	500 mL	Cool to 4°C with NaOH to pH>9; Zn acetate	7 days to analysis
	Anions: Nitrate, nitrite, sulfate, and chloride	EPA 300.0 ALSI SOP-04-ANION2	1 – 1L Polypropylene bottle	1 L	Cool to 4°C	Nitrate/Nitrite: 48 hours to analysis 28 days to analysis for others
	Anions: orthophosphate	SM 4500-PE ALSI SOP-04-OP	1 – 250 mL Polypropylene bottle	100 mL	Cool to 4°C	48 hours to analysis
	Methane, Ethane, Ethene, and Acetylene	RSK SOPs 147/175 Microseeps SOP - AM20GAX	2 – 40 mL clear glass vials	80 mL	Cool to 4°C with Na ₃ PO ₄ to pH>9	14 days to analysis

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5
 Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #19 -- Analytical SOP Requirements Table

(UFP-QAPP Manual Section 3.1.1)

Matrix	Analytical Group	Analytical and Preparation Method / SOP Reference ¹	Containers (number, size, and type)	Sample volume ² (units)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time ³ (preparation / analysis)
Groundwater	Metabolic Acids: Lactic, Pyruvic, Acetic, Propionic, Butyric	Microseeps SOP - AM23G	2 – 40 mL amber glass vials	80 mL	Cool to 4°C with Benzalkonium chloride (BAK)	14 days to analysis
	PCR/Enzymes: TCE Reductase, BAV1 VC Reductase, and VC Reductase	Microbial Insights SOP - qPCR	Laboratory filters (preferred)—1 per sample	1 L	Cool to 4°C	Ship day of collection; 24-48 hours to analysis

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

² Provide the minimum sample volume or mass requirement if it differs from the container volume.

³ Maximum holding time is calculated from the time the sample is collected to the time the sample is prepared/extracted.

°C – Degrees Centigrade

L – Liter

PCR – Polymerase Chain Reaction

HCl – Hydrochloric Acid

mL – Milliliter

HDPE – High Density Polyethylene

Na₃PO₄ – Trisodium phosphate

HNO₃ – Nitric Acid

NaOH – Sodium Hydroxide

H₃PO₄ – Phosphoric Acid

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #19 -- Analytical SOP Requirements Table[\(UFP-QAPP Manual Section 3.1.1\)](#)**Groundwater Field Analyses⁽¹⁾ Biological Activity Indicators Field Analytical SOP Requirements Table**

Parameter	Method/ Reference	Sample Volume, Container, & Preservation
Ferrous Iron	HACH IR-18C	Follow test kit instructions. Analyze immediately at well head. Filter if turbid.
Hydrogen Sulfide	HACH HS-C	Follow test kit instructions. Do not aerate or agitate. Avoid agitation and analyze immediately at well head.
Temperature	Direct-reading meter	100 to 250 ml in glass or plastic container. Analyze at well head.
DO	CHEMetrics – K-7501, K-7512 vacuum vials	Follow test kit instructions. Analyze at well head.
pH	Direct-reading meter	100 to 250 ml in glass or plastic container. Analyze at well head.
Specific Conductivity	Direct-reading meter	100 to 250 ml in glass or plastic container. Analyze at well head.
ORP	Direct-reading meter	10 to 250 ml in glass container filling from the bottom. Do not aerate or agitate. Analyze at well head with flow-through cell.
Alkalinity	CHEMetrics ampoules kits	Follow test kit instructions.

¹ Table adapted from overview of the Technical Guidelines for Evaluating Natural Attenuation of Chlorinated Solvents in Groundwater (U.S. EPA, 1998).

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

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

Matrix	Analytical Group	No. of Sampling Locations ²	No. of Field Duplicates	No. of MS/MSDs ¹	No. of Field Blanks	No. of Equip. Blanks	No. of VOA Trip Blanks	No. of PT Samples ³	Total No. of Samples to Lab
Soil	TCL VOC	24	2	2	NA	NA	NA	0	28
Groundwater	TCL VOC	150	15	8	1	8	35	0	217
	Metals	49	0	0	0	0	NA	0	49
	TOC	49	0	0	0	0	NA	0	49
	Sulfide	49	0	0	0	0	NA	0	49
	Anions	49	0	0	0	0	NA	0	49
	Dissolved Gases	49	0	0	0	0	NA	0	49
	Metabolic Acids	49	0	0	0	0	NA	0	49
	PCR/Enzymes	16	0	0	0	0	NA	0	16

¹ Although the MS/MSD is not typically considered a field QC it is included here because location determination is often established in the field.

² If samples will be collected at different depths at the same location, count each discrete sampling depth as a separate sampling location or station.

³ The number of Batch or Project-specific proficiency testing (PT) samples are optional but highly recommended.

No field QC Samples will be collected for the field analyses.

MS –Matrix Spike

PCR – Polymerase Chain Reaction

PT – Proficiency Testing

SOP – Standard Operating Procedure

TCL VOC – Target Analyte List Volatile Organic Compound

TOC – Total Organic Carbon

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5
 Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

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

Reference Number	Title, Revision Date and / or Number*	Originating Organization of Sampling SOP	Equipment Type	Modified for Project Work? (Y/N)	Comments
SA-2.5	Direct Push Technology (Geoprobe®/Hydropunch™) 09/2003 Rev. 3	TtNUS	Sampling Procedures, Methods	N	
SA-1.3	Soil Sampling 04/2008 Rev. 9	TtNUS	Sampling Procedures, Methods	N	
SA-1.1	Groundwater Sample Acquisition and Onsite Water Quality Testing 04/2008 Rev. 7	TtNUS	Sampling Procedures, Methods	N	
SA-1.6	Natural Attenuation Parameter Collection 09/2003 Rev. 1	TtNUS	Sampling Procedures, Methods	N	
CT-04	Sample Nomenclature 09/2003 Rev. 1	TtNUS	NA	Y	See Sample Designations in Worksheet 18
SA-6.1	Non-radiological Sample Handling 02/2004 Rev. 3	TtNUS	Sample Bottle ware, Packaging Material, Shipping Materials	N	
SA-6.3	Field Documentation 09/2003 Rev. 2	TtNUS	Field Logbook, Field Sample Forms, Boring Logs	N	
SA-7.1	Decontamination of Field Equipment 04/2008 Rev. 5	TtNUS	Decontamination Equipment (scrub brushes, phosphate free detergent, de-ionized water)	N	
NA	Photo-Ionization Detector	Manufacturer Instructions	Calibration and operation	N	

*The TtNUS SOPs are currently being reviewed and updated. The procedures included in this SAP have not changed significantly nor are they expected affect the quality of the data being collected.

NA – Not Applicable
 TtNUS – Tetra Tech NUS

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #22 -- Field Equipment Calibration, Maintenance, Testing, and Inspection Table

(UFP-QAPP Manual Section 3.1.2.4)

Field Equipment	Activity ¹	Frequency	Acceptance Criteria	Corrective Action	Resp. Person	SOP Reference ²	Comments
Photo-Ionization Detector	Calibrate with gas in accordance with manufacturer specifications; Visual Inspection	Daily	Manufacturer's Guidance	Replace	FOL	NA	
Multi-parameter Water Quality Meter	Calibrate in accordance with manufacturer specifications; Visual Inspection	Daily	Manufacturer's Guidance	Replace	FOL	SA-1.1	
Bladder Pump	Visual Inspection	Daily	Equipment Inspection Sheet Criteria	Replace	FOL	SA-1.1	
Redi Flo™ Submersible Pump	Visual Inspection	Daily	Equipment Inspection Sheet Criteria	Replace	FOL	SA-1.1	

¹ Activities may include: calibration, verification, testing, maintenance.

² Specify the appropriate reference letter or number from the Project Sampling SOP References table (Worksheet #21).

FOL – Field Operations Leader

SOP – Standard Operating Procedure

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

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

Lab SOP Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? ¹ (Y/N)
ALSI SOP-02-8260B	Volatile Organics by GC/MS: Capillary Column Technique Rev. 9, 8/17/06	Definitive	Organic/GC/MS	GC/MS	Analytical Laboratory Services, Inc. of Middletown, PA	N
ALSI SOP-02-5035	Closed System Purge and Trap and Extraction for Volatile Organics in Soil and Waste Samples	Definitive	Organic/GC/MS	NA	Analytical Laboratory Services, Inc. of Middletown, PA	N
ALSI SOP-09-3015	Microwave Assisted Digestion of Aqueous Samples and Extracts for Total Metals Analysis by ICP or GFAA Spectroscopy rev 7 2/20/06	Definitive	Inorganic/Metals	NA/ Digestion	Analytical Laboratory Services, Inc. of Middletown, PA	N
ALSI SOP-03-6010B	Analysis of Total Metals by Inductively Coupled Plasma Using the TJA Trace ICP rev 12, 2/13/07	Definitive	Inorganic/Metals	ICP-AES	Analytical Laboratory Services, Inc. of Middletown, PA	N
ALSI SOP-04-S	The Determination of Sulfide in Water and Wastewater Rev 4 11/22/06	Definitive	Wet Chemistry	NA	Analytical Laboratory Services, Inc. of Middletown, PA	N
ALSI SOP-04-ANION2	Determination of Inorganic Anions by Ion Chromatography	Definitive	Wet Chemistry	IC	Analytical Laboratory Services, Inc. of Middletown, PA	N
ALSI SOP-04-OP	Orthophosphate	Definitive	Wet Chemistry	Spectrophotometer	Analytical Laboratory Services, Inc. of Middletown, PA	N
Microseeps SOP - AM23G	SOP for the Analyses of Low Level Volatile Fatty Acids by Ion Chromatography, March 3, 2005	Definitive	Semivolatiles	Dionex IC DX-500	Microseeps, Inc. of Pittsburgh, PA	N

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

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

Lab SOP Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? ¹ (Y/N)
Microseeps SOP – WC21	SOP for the Determination of Organic Carbon in Water Samples, March 1, 2005	Definitive	Wet Chemistry	Thermo Scientific HiPerTOC	Microseeps, Inc. of Pittsburgh, PA	N
Microseeps SOP - AM20GAX	SOP for the Analysis of Biodegradation Indicator Gases, September 15, 2006	Definitive	Risk	GC/FID/TCD	Microseeps, Inc. of Pittsburgh, PA	N
Microbial Insights SOP - qPCR-2006	PCR/Enzymes ²	Definitive	qPCR	ABI 7300	Microbial Insights, Inc. of Rockford, TN	N

- 1 If yes, then specify the modification that has been made. Note that any analytical SOP modification made relative to project specific needs must be reviewed and approved by the Navy QAO.
- 2 *The qPCR-2006 SOP from Microbial Insights is not available to Tetra Tech NUS due to proprietary rights. Quality Assurance information about the method was provided by Microbial Insights and is summarized in Tables 23, 24, and 28.

GC/FID/TCD – Gas Chromatograph/Flame Ionization Detector/Thermal Conductivity Detector

GC/MS – Gas Chromatograph/Mass Spectrometer

GFAA – Graphite Furnace Atomic Absorption

IC – Ion Chromatograph

ICP-AES – Inductively Couple Plasma-Atomic Emission Spectroscopy

NA – Not Applicable

SOP – Standard Operating Procedure

TBD - To Be Determined

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #24 -- Analytical Instrument Calibration Table[\(UFP-QAPP Manual Section 3.2.2\)](#)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA ²	SOP Reference ¹
GC/MS	Minimum five point calibration for all analytes	Instrument receipt, instrument change (new trap, column, etc.), when continuing calibration does not meet criteria	Relative Standard Deviation (RSD) for each Calibration Check Compound (CCC) < 30%, minimum mean response factor (RF) for each System Performance Check Compound (SPCC) as noted in 7.3.5.4 of method 8260B or 8270C. If RSD for an analyte is > 15% apply linear ($r^2 > 0.99$) or quadratic method for quantitation	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data.	Analyst/ Supervisor	ALSI SOP-02-8260B
ICP-AES	4-5 point calibration plus blank per manufacturer's guidelines	At the beginning of each day or if quality control is out of criteria.	4-5 point calibration plus blank per manufacturer's guidelines; analytes run at their calibration levels must fall within 90-110% of True Values	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards	Analyst/ Supervisor	ALSI SOP-03-6010B
Ion Chromatograph (IC) for Anions	2 6-point calibrations	1/month or as needed	Correlation coefficient ≥ 0.995 , passing second source	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards	Analyst/ Supervisor	ALSI SOP-04-ANION2
Spectrophotometer	7-point curve	Every 3 months or when quality control fails	Correlation coefficient ≥ 0.995	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards.	Analyst/ Supervisor	ALSI SOP-04-OP

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #24 -- Analytical Instrument Calibration Table[\(UFP-QAPP Manual Section 3.2.2\)](#)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA²	SOP Reference¹
Dionex IC DX-500 for Volatile Fatty Acids (VFA)	7-point Initial Calibration	When major revision to the method is performed, maintenance is required, or when continuing calibration fails criterion	Correlation coefficient ≥ 0.995 ; Initial Calibration Verification (ICV) within 90-110%	Re-prepare and reanalyze standards.	Analyst/ Supervisor	Microseeps SOP - AM23G
Dionex IC DX-500 for VFA	Continuing Calibration	Per 20 samples	Mid-point standard recovery within 70-130%	Re-prepare and reanalyze standards, if still outside criterion, recalibrate.	Analyst/ Supervisor	Microseeps SOP - AM23G
Thermo Scientific HiPerTOC	5-point curve Initial Calibration	When major revision to the method is performed, maintenance is required, or when continuing calibration fails criterion	Correlation coefficient ≥ 0.995 ; ICV within 90-110%	Re-prepare and reanalyze standards.	Analyst/ Supervisor	Microseeps SOP - 9060
Thermo Scientific HiPerTOC	Continuing Calibration	Per 15 samples	Mid-point standard recovery within 90-110%	Re-prepare and reanalyze standards, if still outside criterion, recalibrate.	Analyst/ Supervisor	Microseeps SOP - WC21
GC for Dissolved Gases	Minimum of 5-point Initial Calibration per detector	When major revision to the method is performed, maintenance is required, or when continuing calibration fails criterion	Correlation coefficient ≥ 0.995 ; ICV within 80-120%	Re-prepare and reanalyze standards.	Analyst/ Supervisor	Microseeps SOP - AM20GAX

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #24 -- Analytical Instrument Calibration Table

(UFP-QAPP Manual Section 3.2.2)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA²	SOP Reference¹
GC for Dissolved Gases	Continuing Calibration	Per 20 samples	Mid-point standard recovery within 80-120%	Re-prepare and reanalyze standards, if still outside criterion, recalibrate.	Analyst/ Supervisor	Microseeps SOP - AM20GAX
ABI 7300 (qPCR)	Initial Assay Calibration (standard curve)	Once per assay	Standard curve R2 >0.95	Rerun and/or optimize assay	Analyst/ Supervisor	Microbial Insights SOP - 7300ABI
ABI 7300 (qPCR)	Continuing Calibration Verification (CCV)	Primary – Semi-annual Secondary – every plate (assay)	Primary: Standard curve R2 >0.95 Replicate within 1 Threshold Cycle (CT) Secondary: CT value within 2 units of same point on standard curve	Rerun assay / check reagents. Non conformance report—call service engineer with ABI	Laboratory Manager	Microbial Insights SOP - 7300ABI

¹ Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).² Name or title of responsible person may be used.

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5
 Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #25 -- Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table
 (UFP-QAPP Manual Section 3.2.3)

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person ²	SOP Reference ¹
GC/MS	Check pressure and gas supply daily. Bake out trap and column, manual tune, if Bromofluorobenzene (BFB) not in criteria, change septa as needed, cut column as needed, change trap as needed.	Volatile Organic Compound Analysis	Initial Calibration, Continuing Calibration	Initial Cal: Instrument receipt, instrument change (new trap, column, etc.), when CCC do not meet criteria Cont. Cal: At beginning of each 12 hour shift immediately after BFB tune.	Initial Cal: RSD for each CCC < 30%, min. mean RF for each SPCC as noted in 7.3.5.4 of method 8260B. If RSD for an analyte is > 15% apply linear or quadratic method for quantitation Cont. Cal: %D for each CCC < 20%, min. RF for each SPCC as noted in 7.3.5.4 of method 8260B.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data. Record maintenance activities in MS05-02 Maintenance Log 02-0160.	Analyst/Supervisor	ALSI SOP-02-8260B
ICP-AES	Clean torch assembly and spray chamber when discolored or when degradation in data quality is observed. Clean nebulizer, check argon, and replace peristaltic pump tubing as needed.	Metals Analysis	ICV, CCV	Init. Cal: At the beginning of each day or if QC is out of criteria. ICV: Immediately after instrument calibration CCV: After every 10 samples and at end of analytical sequence	Init. Cal: 4-5 point calibration plus blank with ≥ 0.995 correction coefficient ICV and CCV: 90-110% of true value for ICP	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected metals. Record maintenance activities in Thermo Trace Maintenance Logbook 03-0102.	Analyst/Supervisor	ALSI SOP-03-6010B

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #25 -- Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table[\(UFP-QAPP Manual Section 3.2.3\)](#)

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person ²	SOP Reference ¹
Ion Chromatograph	See "Maintenance" in Appendix B	Anion Analysis	CCV	After end of analytical sequence	90-110% of true value	Re-run CCV, Re-prepare CCV, Re-calibrate, perform necessary equipment maintenance	Analyst/Supervisor	ALSI SOP-04-ANION2
Spectrophotometer	7-point curve	Orthophosphate analysis	CCV	After every 10 samples and after end of analytical sequence	CCV: 90-110% of true value	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected samples. Record maintenance activities.	Analyst/Supervisor	ALSI SOP-04-OP
Dionex IC DX-500	Check column performance, check detector response	Calibration procedures in Worksheet 24	Check for leaks, all tubing for wear and discoloration, gas cylinder, pump pistons	Leak check daily; tubing check weekly, gas cylinder daily, pump pistons quarterly	Calibration criterion in Worksheet 24	Repair and replace as needed	Analyst/Supervisor	Microseeps SOP - AM23G

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5
 Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study
 Revision Number: 0
 Revision Date: September 2008

SAP Worksheet #25 -- Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table
 (UFP-QAPP Manual Section 3.2.3)

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person ²	SOP Reference ¹
Thermo Scientific HiPerTOC	Check valves and clean as necessary, clean reactor, check tubing for leaks, prepare reagents	Calibration procedures in Worksheet 24	Check gas cylinder, check waste container	Daily	Calibration criterion in Worksheet 24	Maintenance and replacement activities as needed	Analyst/ Supervisor	Microseeps SOP – WC21
GC/FID/TCD for Dissolved Gases	Clean Flame Ionization Detector (FID) as needed, replace Thermal Conductivity Detector (TCD) filaments as needed, bake out columns daily and replace as needed	Calibration procedures in Worksheet 24	Check septa, gas cylinder, and injector body	Daily	Calibration criterion in Worksheet 24	Replace septa, gas cylinder, and injector body as needed Recalibrate as needed.	Analyst/ Supervisor	Microseeps SOP - AM20GAX
ABI 7300 (PCR)	Dye calibration Contamination Check	Dye calibration plates Water control plate	ABI Analyst/ Supervisor	Semi-annual Monthly	Curve within specified range (see ABI 7300 manual) No contaminant curves	Re-calibrate dyes Clean wells following SOP and repeat contamination check	ABI Service Engineer Analyst/ Supervisor	MISOP-7300ABI

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

² Name or title of responsible person may be used.

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #26 -- Sample Handling System

[\(UFP-QAPP Manual Appendix A\)](#)

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): TBD/TtNUS
Sample Packaging (Personnel/Organization): TBD/TtNUS
Coordination of Shipment (Personnel/Organization): TBD/TtNUS
Type of Shipment/Carrier: Overnight courier service (Federal Express)
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Sample custodians / ALSI / Microseeps / Microbial Insights
Sample Custody and Storage (Personnel/Organization): Sample custodians / ALSI/Microseeps / Microbial Insights
Sample Preparation (Personnel/Organization): Preparation Laboratory Staff / ALSI / Microseeps / Microbial Insights
Sample Determinative Analysis (Personnel/Organization): GC/MS, ICP, GC/FID, Spectrophotometer, PCR / ALSI / Microseeps / Microbial Insights
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): 30 days from submittal of final laboratory report for ALSI and Microbial Insights, for Microseeps – 30 days from sample receipt
Sample Extract/Digestate Storage (No. of days from extraction/digestion): 30 days from submittal of final laboratory report for ALSI and Microbial Insights, for Microseeps – 30 days from sample receipt
Biological Sample Storage (No. of days from sample collection): Not Applicable
SAMPLE DISPOSAL
Personnel/Organization: Sample custodians/ALSI/Microseeps/Microbial Insights
Number of Days from Analysis: 30 days from submittal of final laboratory report for ALSI and Microbial Insights, for Microseeps – 30 days from sample receipt

ALSI – Analytical Laboratory Services, Inc.
ICP – Inductively Couple Plasma
TtNUS – Tetra Tech NUS, Inc.

GC/MS – Gas Chromatograph/Mass Spectrometer
PCR – Polymerase Chain Reaction

GC/FID – Gas Chromatograph/Flame Ionization Detector
TBD – To Be Determined

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5
Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #27 – Sample Custody Requirements Table

[\(UFP-QAPP Manual Section 3.3.3\)](#)

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

Following sample collection into the appropriate bottle ware, all samples will be immediately placed on ice in a cooler. The glass sample containers will be enclosed in bubble-wrap in order to protect the bottle ware during shipment. The cooler will be secured using duct or clear packaging tape along with a signed custody seal. Sample coolers will be delivered to a local courier location for priority overnight delivery to the selected laboratory for analysis. Samples will be preserved as appropriate based on the analytical method. Laboratories will provide pre-preserved sample containers for sample collection. Samples will be maintained at 4 degrees centigrade (°C) until delivery to the laboratories. Proper custody procedures will be followed throughout all phases of sample collection and handling. COC protocols will be used throughout sample handling to establish the evidentiary integrity of sample containers. These protocols will be used to demonstrate that the samples were handled and transferred in a manner that would eliminate possible tampering. Samples for the laboratory will be packaged and shipped in accordance with TtNUS SOP SA-6.1.

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

ALSI-19-Rec/Han (Standard Operating Procedure for Sample Receipt/Sample Handling)
ALSI-19-Disp (Standard Operating Procedure for Disposal of Samples, Extracts, Digestates, and Leachates)
Microseeps-SOP-S2 (Standard Operating Procedure for Sample Receiving)
Microseeps-ADM-14 (Standard Operating Procedure for Waste Disposal)

Sample Identification Procedures:

Sample nomenclature will be conducted in general accordance with the procedures outlined in TtNUS SOP CT-04 (Sample Nomenclature). Sample nomenclature put forth for this field event has been selected based on historical usage. The sample nomenclature for each tracking number includes the site being investigated, sample media identifier, and sample location number. The standard sample matrix and type codes used for this field event are as follows: Duplicate samples will be submitted to the laboratory as blind duplicates. Therefore duplicate codes will be reflective of the standard sample matrix code followed by a "DUP" tag and sequentially listed. Due to the blind nature of the duplicate samples, no sample depth or date will be listed with the duplicated sample. An example of a duplicate sample would be "05DUP001". The QA/QC type codes used for this field event are as follows: TB for Trip Blanks, FB for field blanks, and RB for rinsate blanks. Field QC blanks will be labeled sequentially followed by the date (i.e., TB20070430, FB20070501). Samples to be used for matrix spikes and matrix spike duplicates will be labeled MS/MSD on the bottle label and noted on the COC, as required in the laboratory QA Plan; however, "MS/MSD" will not be part of the unique sample identifier in order to maintain consistency with the project database. Additional information regarding protocol for sample labeling is contained in TtNUS SOP SA-6.3 and LAB SOP.

Chain-of-custody Procedures:

After recovery, each samples will be maintained in the sampler's custody until formally transferred to another party (e.g., Federal Express). For all samples recovered, custody records will document the date and time of sample collection, the sampler's name, and the names of all others who subsequently held custody of the sample. Specifications for chemical analyses will also be documented on the custody record. Attached SOP SA-6.3 (Field Documentation) provides further details on the COC procedure. COC requirements are also documented with instructions contained in each shipment for the laboratory. (ALSI-19-COC [Standard Operating Procedure for Chain of Custody Entry], Microseeps-SOP-S2)

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5
 Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #28 -- Laboratory QC Samples Table
 (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
Method Blank	One every 12 hours prior to sample analysis	No target compounds $\geq \frac{1}{2}$ QL except common lab contaminants which should be < RL	Re-clean, retest, re-extract, reanalyze, and/or qualify data.	Analyst, Laboratory Supervisor and Data Validator	Bias / Contamination	No target compounds $\geq \frac{1}{2}$ QL except common lab contaminants which should be < RL.
Surrogates	3 per sample	4-Bromofluorobenzene 51-128% Dibromofluoromethane 62-123% Toluene-d8 59-131%	(1) Re-prep and reanalyze for confirmation of matrix interference when appropriate.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias	70-130%
Laboratory Control Sample (LCS)	One per batch of 20 or less	See attached QC limit table in Appendix B.	(1) Evaluate and reanalyze if possible (2) If an MS/MSD was performed in the same 12 hour clock and acceptable narrate. (3) If the LCS recoveries are high but the sample results are <QL narrate otherwise re- prep and reanalyze.	Analyst, Laboratory Supervisor and Data Validator	Precision / Accuracy / Bias	70-130%
Internal Standards (IS)	3 per sample	Retention time + 30 seconds; EICP area within -50% to +100% of last calibration verification (12 hours) for each IS.	Inspect mass spectrometer or GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning.	Analyst, Laboratory Supervisor and Data Validator	Precision / Accuracy / Bias	Retention time + 30 seconds; EICP area within -50% to +100% of last calibration verification (12 hours) for each IS.
Matrix spike / Matrix spike duplicate (MS/MSD)	One per SDG or every 20 samples	See attached QC limit table in Appendix B. MS/MSD limits are the same as LCS limits. RPD < 30%	(1) CA will not be taken for samples when recoveries are outside limits and surrogate and LCS criteria are met. (2) If both the LCS and MS/MSD are unacceptable re- prep the samples and QC.	Analyst, Laboratory Supervisor and Data Validator	Precision / Accuracy / Bias	70-130% RPD < 30%

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5
 Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study
 Revision Number: 0
 Revision Date: September 2008

SAP Worksheet #28 -- Laboratory QC Samples Table
[\(UFP-QAPP Manual Section 3.4\)](#)

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Trip Blank	One per cooler of VOC samples shipped to laboratory	No target analytes \geq QL; with the exception of common field/laboratory contaminants	No corrective action by laboratory.	Data Validator	Bias / Contamination	No target analytes \geq QL; with the exception of common field/laboratory contaminants.
Method Blank	One every 12 hours prior to sample analysis	No target compounds \geq $\frac{1}{2}$ QL except common lab contaminants which should be $<$ RL	Re-clean, retest, re-extract, reanalyze, and/or qualify data.	Analyst, Laboratory Supervisor and Data Validator	Bias / Contamination	No target compounds \geq $\frac{1}{2}$ QL except common lab contaminants which should be $<$ RL.
Surrogates	3 per sample	4-Bromofluorobenzene 78-116% Dibromofluoromethane 79-114% Toluene-d8 76-127%	(1) Re-prep and reanalyze for confirmation of matrix interference when appropriate.	Analyst, Laboratory Supervisor and Data Validator	Accuracy / Bias	70-130%
LCS	One per batch of 20 or less	See attached QC limit table in Appendix B.	(1) Evaluate and reanalyze if possible (2) If an MS/MSD was performed in the same 12 hour clock and acceptable narrate. (3) If the LCS recoveries are high but the sample results are $<$ QL narrate otherwise re-prep and reanalyze.	Analyst, Laboratory Supervisor and Data Validator	Precision / Accuracy / Bias	70-130%
IS	3 per sample	Retention time + 30 seconds; EICP area within -50% to +100% of last calibration verification (12 hours) for each IS.	Inspect mass spectrometer or GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning.	Analyst, Laboratory Supervisor and Data Validator	Precision / Accuracy / Bias	Retention time + 30 seconds; EICP area within -50% to +100% of last calibration verification (12 hours) for each IS.

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #28 -- Laboratory QC Samples Table[\(UFP-QAPP Manual Section 3.4\)](#)

Matrix	Groundwater					
Analytical Group	TCL VOC					
Analytical Method/ SOP Reference	SW-846 8260B/ ALSI SOP-02-8260B					
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	One per SDG or every 20 samples	See attached QC limit table in Appendix B. MS/MSD limits are the same as LCS limits. RPD < 30%	(1) CA will not be taken for samples when recoveries are outside limits and surrogate and LCS criteria are met. (2) If both the LCS and MS/MSD are unacceptable re- prep the samples and QC.	Analyst, Laboratory Supervisor and Data Validator	Precision / Accuracy / Bias	70-130% RPD < 30%

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5
 Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

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

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Matrix	Groundwater					
Analytical Group	Metals (dissolved iron and manganese, and total sodium)					
Analytical Method/SOP Reference	SW-846 6010B/ ALSI SOP-03-6010B					
Method Blank	One per digestion batch of 20 or fewer samples	Less than 1/2 PQL	1) Investigate source of contamination. Re-digest and reanalyze all associated samples if sample concentration \geq QL and $<10x$ the blank concentration.	Laboratory Supervisor	Bias / Contamination	Less than 1/2 PQL
LCS	One per digestion batch of 20 or fewer samples	Recovery within $\pm 20\%$ of true value, unless vendor-supplied or statistical limits have been established.	1) Investigate source of problem. 2) Re-digest and reanalyze all associated samples.	Laboratory Supervisor	Accuracy / Bias / Contamination	Recovery within $\pm 20\%$ of true value, unless vendor-supplied or statistical limits have been established.
Duplicate Sample	One per digestion batch of 20 or fewer samples	RPD $\leq 20\%$ for duplicate spikes.	Flag results.	Analyst, Laboratory Supervisor	Precision	RPD $\leq 20\%$ for duplicate spikes
MS	One per digestion batch of 20 or fewer samples	Recovery $\pm 25\%$ of true value, if sample $< 4x$ spike added.	Flag results.	Analyst, Laboratory Supervisor	Accuracy / Bias	Recovery $\pm 25\%$ of true value, if sample $< 4x$ spike added.
ICP Serial Dilution	One per digestion batch	If original sample result is at least 50x IDL, 5X dilution must agree within $\pm 10\%$ of the original result.	Flag result or dilute and reanalyzed sample to eliminate interference.	Analyst, Laboratory Supervisor	Accuracy / Bias	If original sample result is at least 50x IDL, 5-fold dilution must agree within $\pm 10\%$ of the original result.

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5
 Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #28 -- Laboratory QC Samples Table
[\(UFP-QAPP Manual Section 3.4\)](#)

Matrix	Groundwater					
Analytical Group	TOC					
Analytical Method/ SOP Reference	EPA 415.1/ Microseeps SOP – WC21					
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	One per prep batch	< concentration of lowest standard	(1) Investigate source of contamination. (2) Prepare and analyze new method blank	Analyst, Laboratory Supervisor	Bias Contamination	No analyte detected >PQL
LCS	One per 20 client samples	70-130 %R	Recalibrate and/or reanalyze other samples.	Analyst, Laboratory Supervisor	Accuracy / Bias	70-130 %R
MSD	One per 10 client samples	RPD \leq 20 for samples >5X the PQL	If MS is out and other QC is in, note matrix interference in narrative.	Analyst, Laboratory Supervisor	Precision	RPD \leq 20 for samples >5X the PQL
MS	One per 10 client samples	70-130% R	If MS is out and other QC is in, note matrix interference in narrative.	Analyst, Laboratory Supervisor	Accuracy / Bias	70-130% R

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #28 -- Laboratory QC Samples Table[\(UFP-QAPP Manual Section 3.4\)](#)

Matrix	Groundwater					
Analytical Group	Sulfide					
Analytical Method/ SOP Reference	EPA 376.1/ALSI SOP- 04-S					
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	One per 10 client samples, beginning and end of run.	< RL of 1 ug/L	Reanalyze all detectable samples. If volume not available, narrate.	Analyst, Laboratory Supervisor	Bias/Contamination	< RL of 1 ug/L
LCS	One per 10 client samples, beginning and end of run.	90-110 %R	Do not analyze samples until LCS recovery is acceptable. If end LCS is unacceptable, reanalyze samples.	Analyst, Laboratory Supervisor	Accuracy / Bias	90-110 %R
Laboratory Duplicate	One per 10 client samples	RPD ≤20%	Report in narrative.	Analyst, Laboratory Supervisor	Precision	RPD ≤20%

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #28 -- Laboratory QC Samples Table[\(UFP-QAPP Manual Section 3.4\)](#)

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	One per 10 client samples, beginning and end of run.	< 1/2 RL	(1) Identify and correct problem. (2) Reanalyze blank and detectable samples since unacceptable blank.	Analyst, Laboratory Supervisor	Bias Contamination	No analyte detected >1/2 RL
Second Source Standard	One per 20 client samples, beginning of run.	90-110 %R	(1) Reanalyze once, if unacceptable, reanalyze all associated samples.	Analyst, Laboratory Supervisor	Accuracy / Bias	90-110 %R
MS	One per 10 client samples	80-120 %R	If all other QC in, report in narrative.	Analyst, Laboratory Supervisor	Accuracy / Bias	80-120 %R
MSD	One per 20 client samples	RPD \leq 20%	Report in narrative	Analyst, Laboratory Supervisor	Accuracy / Bias / Precision	RPD \leq 20%
Laboratory Duplicate	One per 10 client samples	RPD \leq 10%	Reanalyze, if volume not available, report in narrative	Analyst, Laboratory Supervisor	Precision	RPD \leq 10%

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #28 -- Laboratory QC Samples Table[\(UFP-QAPP Manual Section 3.4\)](#)

Matrix	Groundwater					
Analytical Group	Orthophosphate					
Analytical Method/ SOP Reference	SM 4500-PE/ ALSI SOP-04-OP					
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	One per 10 client samples, beginning and end of run.	< 0.02 mg/L or 1/10 RL	(1) Investigate source of contamination. (2) Reanalyze blank and associated samples.	Analyst, Laboratory Supervisor	Bias Contamination	No analyte detected > 0.02 mg/L or 1/10 RL
MS	One per 10 client samples	90-110 %R	(1) If all other QC in, reanalyze spike once. (2) If reanalysis fails or is not possible, report in narrative.	Analyst, Laboratory Supervisor	Accuracy / Bias	90-110 %R
MSD	One per 10 client samples	RPD ≤ 10%	(1) If all other QC in, reanalyze MSD once. (2) If reanalysis fails or is not possible, report in narrative.	Analyst, Laboratory Supervisor	Accuracy / Bias Precision	RPD ≤ 10%

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #28 -- Laboratory QC Samples Table[\(UFP-QAPP Manual Section 3.4\)](#)

Matrix	Groundwater					
Analytical Group	Dissolved Gases (ethene, ethane, methane, acetylene)					
Analytical Method/ SOP Reference	RSK 14/175/ Microseeps SOP - AM20GAX					
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	One per prep batch	< PQL	(1) Investigate source of contamination. (2) Re-prepare and analyze blank.	Analyst, Laboratory Supervisor	Bias Contamination	No analyte detected > PQL
LCS	One per prep batch	75-125 %R	(1) Re-prepare and analyze. (2) If new LCS fails, Investigate problem. (3) Recalibrate and run LCS and samples.	Analyst, Laboratory Supervisor	Accuracy / Bias	75-125 %R
LCS Duplicate	One per prep batch	RPD ≤ 20%	Note in narrative.	Analyst, Laboratory Supervisor	Precision	RPD ≤ 20%

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #28 -- Laboratory QC Samples Table[\(UFP-QAPP Manual Section 3.4\)](#)

Matrix	Groundwater					
Analytical Group	Metabolic Acids (lactic, pyruvic, acetic, propionic, butyric)					
Analytical Method/ SOP Reference	Microseeps SOP - AM23G					
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	One per prep batch	No analyte detected > lowest analyte in lowest standard	(1) Prepare and analyze new blank.	Analyst, Laboratory Supervisor	Bias Contamination	No analyte detected > lowest analyte in lowest standard
LCS	One per 20 client samples	70-130 %R	(1) Prepare and analyze new LCS. (2) If it fails again, re-prepare and analyze associated samples. (3) Report in narrative.	Analyst, Laboratory Supervisor	Accuracy / Bias	70-130 %R
MS	One per analytical batch	70-130 %R	If all other QC is in, report in narrative.	Analyst, Laboratory Supervisor	Accuracy / Bias	70-130 %R
MSD	One per analytical batch	RPD \leq 30 %	If all other QC is in, report in narrative.	Analyst, Laboratory Supervisor	Accuracy / Bias Precision	RPD \leq 30 %

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5
 Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study
 Revision Number: 0
 Revision Date: September 2008

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

Matrix	Groundwater					
Analytical Group	PCR/Enzymes					
Analytical Method/ SOP Reference	Microbial Insights SOP – qPCR-2006					
QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Assay Negative Control (Blank)	1 per analytical assay plate	Values for positive samples are set above any fluorescence for the negative control	Rerun assay; may have to re-optimize assay	Analyst; Laboratory Supervisor	Bias / Contamination	Values for positive samples are set above any fluorescence for the negative control
DNA extraction negative control	1 per analytical batch	CT ≤ Assay Negative Control	Rerun assay or re-extract samples if problem persists	Analyst; Laboratory Supervisor	Bias / Contamination	CT ≤ Assay Negative Control
Positive Control	1 per analytical assay plate	CT value within 2 units of same point on standard curve	Rerun assay / check reagents	Analyst; Laboratory Supervisor	Bias / Contamination	CT value within 2 units of same point on standard curve
Laboratory Duplicate	All field samples	CT value within 2 units of other duplicate	Rerun assay; if still not within 2 CT units, flag J (estimate)	Analyst; Laboratory Supervisor	Precision	CT value within 2 units of other duplicate

- %R – Percent Recovery
- CT – Threshold Cycle
- EICP – Extracted Ion Current Profile
- GC – Gas Chromatograph
- ICP – Inductively Coupled Plasma
- IDL – Instrument Detection Limit
- IS – Internal Standard
- LCS – Laboratory Control Sample
- QC – Quality Control
- QL – Quantitation Limit
- IS – Internal Standard
- LCS – Laboratory Control Sample
- mg/L – Milligrams per Liter
- MS – Matrix Spike
- MSD – Matrix Spike Duplicate
- PCR – Polymerase Chain Reaction
- PQL – Practical Quantitation Limit
- RL – Reporting Limit
- RPD – Relative Percent Difference

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

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

Document	Where Maintained
<u>Field Documents</u> Field Logbook Field Sample Forms COC Records Air bills Sampling Instrument Calibration Logs Sampling Notes Drilling Logs Photographs Field Task Modification Forms This SAP Health and Safety Plan	Field documents will be maintained in the project file located in the TtNUS King of Prussia office.
<u>Laboratory Documents</u> Sample receipt, custody, and tracking record Standards traceability logs Equipment calibration logs Sample preparation logs Analysis Run logs Equipment maintenance, testing, and inspection logs Corrective action forms Reported field sample results Reported results for standards, QC checks, and QC samples Sample storage and disposal records Telephone logs Extraction/clean-up records Raw data Data Completeness checklist	Laboratory documents will be included in the hardcopy and PDF deliverables from the laboratory. Laboratory data deliverables will be maintained in the TtNUS King of Prussia project file and in long-term data package storage at a third-party professional document storage firm. Electronic data results will be maintained in a database on a password protected SQL server.
<u>Assessment Findings</u> Field Sampling Audit Checklist (if conducted) Analytical Audit Checklist (if conducted) Data Validation Memoranda (includes tabulated data summary forms)	All assessment documents will be maintained in the TtNUS King of Prussia project file.
<u>Reports</u> Interim Pilot Study Report Pilot Study Report	All reports for the Site 5 Bioremediation Pilot Study will be stored in hardcopy in the TtNUS King of Prussia project file and electronically in the server library.

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #30 -- Analytical Services Table[\(UFP-QAPP Manual Section 3.5.2.3\)](#)

Matrix	Analytical Group	Concentration Level	Sample Location/ID Numbers	Analytical SOP	Data Package Turnaround Time	Laboratory/ Organization (Name and Address, Contact Person and Telephone Number)	Backup Laboratory/Organization ¹ (Name and Address, Contact Person and Telephone Number)
Soil	TCL VOC	Low	See Worksheet 18.	ALSI	Requested: 21 days Actual: 21 days	Scott Brunk Analytical Laboratory Services, Inc. (ALSI) 34 Dogwood Lane Middletown, PA 17057 Phone: (717) 944-5541 ext. 3150 Fax: (717) 944-8972 Alternate contact: Christopher Pugliano (717) 944-5541 ext. 3150	TBD
Groundwater	TCL VOC	Low	See Worksheet 18.	ALSI	Requested: 21 days Actual: 21 days		
Groundwater	Metals	Low	See Worksheet 18.	ALSI	Requested: 21 days Actual: 21 days		
Groundwater	Sulfide	Low	See Worksheet 18.	ALSI	Requested: 21 days Actual: 21 days		
Groundwater	Anions	Low	See Worksheet 18.	ALSI	Requested: 21 days Actual: 21 days		
Groundwater	TOC	Low	See Worksheet 18.	Microseeps	Requested: 21 days Actual: 21 days	Rachel Whitby Microseeps, Inc. 220 William Pitt Way Pittsburgh, PA 15238 Phone: (412)826-2389 Fax: (412) 826-5251 Alternate contact: Debbie Hallo	TBD
Groundwater	Dissolved Gases	Low	See Worksheet 18.	Microseeps	Requested: 21 days Actual: 21 days		
Groundwater	Metabolic Acids	Low	See Worksheet 18.	Microseeps	Requested: 21 days Actual: 21 days		

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

Matrix	Analytical Group	Concentration Level	Sample Location/ID Numbers	Analytical SOP	Data Package Turnaround Time	Laboratory/ Organization (Name and Address, Contact Person and Telephone Number)	Backup Laboratory/Organization ¹ (Name and Address, Contact Person and Telephone Number)
Groundwater	PCR/Enzymes	Low	See Worksheet 18.	Microbial Insights	Requested: 21 days Actual: 21 days	Cheryl Davis Microbial Insights 2340 Stock Creek Blvd. Rockford, TN 37853 Phone: (865) 573-8188 ext. 104 Fax: (865) 573-8133 Alternate contact: Greg Davis gdavis@microbe.com	TBD

A backup laboratory is not expected for this project. If circumstances arise where the contracted laboratories cannot perform the analysis, other laboratories will be procured at that time.

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #31 -- Planned Project Assessments Table[\(UFP-QAPP Manual Section 4.1.1\)](#)

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment (title and organizational affiliation)	Person(s) Responsible for Responding to Assessment Findings (title and organizational affiliation)	Person(s) Responsible for Identifying and Implementing Corrective Actions (CA) (title and organizational affiliation)	Person(s) Responsible for Monitoring Effectiveness of CA (title and organizational affiliation)
Field Sampling Systems Audit	1 per contract year	Internal	TtNUS	TBD	TtNUS PM	TtNUS Auditor and TtNUS QAM	TtNUS QAM
Laboratory Systems Audit	Every 18 months	External	NFESC	TBD	Laboratory QA Manager	Laboratory QA Manager	Laboratory QA Manager
Health and Safety	1 per contract year	Internal	TtNUS	TBD	TtNUS PM	TtNUS Auditor and TtNUS Health and Safety Manager	TtNUS Health and Safety Manager

NFESC – Navy Facilities Engineering Service Center

PM – Project Manager

QA – Quality Assurance

TBD – To Be Determined

TtNUS – Tetra Tech NUS

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

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

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (name, title, organization)	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (name, title, organization)	Timeframe for Response
Field Sampling System Audit	Audit checklist and written audit finding summary	TtNUS PM, TtNUS FOL, and TtNUS Program Manager	Dependent on findings, if major, a stop work maybe issued immediately, however, if minor, within 1 week of audit	Written memo	TtNUS QAM, TtNUS Auditor, TtNUS Program Manager	Within 48 hours of notification
Laboratory Systems Audit	Written audit report	Laboratory QA Manager	Not specified by NFESC	Letter	NFESC	Specified by NFESC
Health and Safety Audit	Audit checklist and written audit finding summary	TtNUS PM, TtNUS FOL, and TtNUS Program Manager	Dependent on findings, if major, a stop work maybe issued immediately, however, if minor, within 1 week of audit	Written memo	TtNUS Health and Safety Manager, TtNUS Auditor, TtNUS Program Manager	Within 48 hours of notification

NFESC – Navy Facilities Engineering Service Center

QA – Quality Assurance

TtNUS – Tetra Tech NUS

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #33 -- QA Management Reports Table[\(UFP QAPP Manual Section 4.2\)](#)

Type of Report	Frequency (daily, weekly monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (title and organizational affiliation)	Report Recipient(s) (title and organizational affiliation)
Data validation report	Per SDG	Within two weeks after receiving the data from the laboratory	TtNUS DVM or designated data validators	TtNUS PM, project file
Major analysis problem identification (Internal Memorandum)	When persistent analysis problems are detected	Immediately	TtNUS QAM	TtNUS PM, QAM, Program Manager, project file
Project monthly progress report	Monthly for duration of the project	Monthly	TtNUS PM	Navy, project file
Field progress reports	Daily, oral, during the course of sampling	Everyday that field sampling is occurring	TtNUS FOL	TtNUS PM
Laboratory QA report	When significant plan deviations results from unanticipated circumstances	Immediately	Subcontracted laboratories will contact the TtNUS QAM or project chemist. The project chemist and TtNUS PM will discuss a solution. If necessary, the Navy RPM will be included in the decision process.	TtNUS PM, QAM, project chemist, project file

FOL – Field Operations Leader
PM – Project Manager
QA – Quality Assurance
QAM – Quality Assurance Manager
SDG – Sample Delivery Group
TtNUS – Tetra Tech NUS

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

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

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
Sample Tables (Worksheet 18)	Proposed samples verified to have been collected.	Internal	TtNUS FOL or designee
Chain of custody	COC records will be reviewed internally by the Project Manager or designee and compared against sample table (Worksheet 18) listing the proposed samples to verify that all planned samples have been collected.	Internal	TtNUS PM or designee
Sample Coordinates	Sample locations have been verified to be correct and in accordance with the SAP (overlay maps proposed locations against actual locations).	Internal	TtNUS FOL, PM, or designee
Electronic Data	Verify that all sample results are included in the Electronic Data Deliverable from the laboratory. Verify the samples against the Chain-of-Custody. Upload the data into the SQL database for the data validators.	Internal	Laboratory Project Manager and TtNUS Sample Management Coordinator
Analytical Data Package	The analytical data will be verified for completeness by the laboratory performing the work. The laboratory shall complete an appropriate form documenting the organization and complete contents of each data package.	External	Scott Brunk, ALSI Rachel Whitby, Microseeps Cheryl Davis, Microbial Insights
Data Package	Verify that the data package contains all the elements required by CLP format and the scope of work, this occurs as part of the data validation process.	Internal	TtNUS Data Validator
Sample Log Sheets	Log sheets completed as samples are collected in the field are verified for completeness and are maintained at the project office.	Internal	TtNUS PM or designee

FOL – Field Operations Leader

PM – Project Manager

SAP – Sampling and Analysis Plan

TtNUS – Tetra Tech NUS

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5
 Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #35 – Validation (Steps IIa and IIb) Process Table

(UFP-QAPP Manual Section 5.2.2) (Figure 37 UFP-QAPP Manual) (Table 9 UFP-QAPP Manual)

Step IIa / IIb ¹	Validation Input	Description	Responsible for Validation (name, organization)
IIa	Field SOPs	Ensure that the sampling SOPs were followed.	TtNUS FOL
IIa	Analytical SOPs	Ensure that all laboratory analytical SOPs were followed.	Scott Brunk, ALSI Rachel Whitby, Microseeps Cheryl Davis, Microbial Insights
IIa	Data package	Validator will verify that elements of the data package that are required for validation are present and if not, the lab will be contacted and the missing information will be requested. Validation will be performed as per Worksheet 36.	TtNUS Data Validator
IIa	Field logs/sample coordinates	Verify that the sampling plan was implemented and carried out as written and any deviations are documented.	TtNUS PM
IIa	Electronic Data	Verify all data have been transferred correctly and completely to the final SQL database.	TtNUS PM, Data Validator, or designee
IIa / IIb	SAP Worksheet 12, Field SOPs/Field Logs, COCs	Verify that deviations have been documented and MPCs have been achieved for field QC samples.	TtNUS FOL, PM, Data Validator or designee
IIa / IIb	SAP Worksheet 28, Laboratory SOPs, Data Package	Verify that deviations have been documented and MPCs have been achieved for laboratory data including environmental sample results and laboratory QC.	TtNUS Data Validator

¹ IIa=compliance with methods, procedures, and contracts [see Table 10, page 117, UFP-QAPP manual, V.1, March 2005.]

IIb=comparison with measurement performance criteria in the SAP [see Table 11, page 118, UFP-QAPP manual, V.1, March 2005]

FOL – Field Operations Leader
 MPC – Measurement Performance Criteria
 PM – Project Manager
 SAP – Sampling and Analysis Plan
 SOP – Standard Operating Procedure
 SQL – Structured Query Language
 TtNUS – Tetra Tech NUS

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #36 –Analytical Data Validation (Steps IIa and IIb) Summary Table[\(UFP-QAPP Manual Section 5.2.2.1\)](#)

Step IIa / IIb	Matrix	Analytical Group	Validation Criteria	Data Validator (title and organizational affiliation)
IIa and IIb	Soil	Volatiles	TtNUS SOP-DV02 and SAP Worksheets 12 and 28.	TtNUS Data Validator
IIa and IIb	Groundwater	Volatiles	TtNUS SOP-DV02 and SAP Worksheets 12 and 28.	TtNUS Data Validator

*Only VOC data will undergo Step IIb validation for the Site 5 Bioremediation Pilot Study. The indicator parameters and field test kit parameters will undergo Verification Step I and Validation Step IIa unless analysis errors are identified. A full laboratory data package will be provided for the indicator parameters in the event that a Step IIb validation will be performed.

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5
Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study
Revision Number: 0
Revision Date: September 2008

SAP Worksheet #37 -- Usability Assessment
[\(UFP-QAPP Manual Section 5.2.3\)](#)

A data usability assessment will be conducted by the planning team, including the TtNUS PM, project chemist, and technical staff. If significant data usability limitations are encountered the planning team will be expanded to include the Navy RPM and Navy technical staff.

The data validation procedure (Worksheets 35 and 36) will be used to help determine which data are usable. Qualifiers will be applied to each value based on the results of the data validation. Rejected values (qualified with "R") and blank qualified values ("B") will be eliminated from further consideration. Estimated and biased values (J [estimated], K [biased high], and L [biased low]) will be used as the reported value. The quantitation limits from the data will be evaluated for sensitivity to the project action levels. Limitations on the use of the data due to lack of project-required sensitivity will be discussed. In addition, the data will be reviewed to evaluate if samples were collected from the intended locations and are representative of site conditions.

After data validation and an overall review of data quality indicators, the data will be reconciled with measurement performance criteria (MPCs) to determine whether sufficient data of acceptable quality are available for decision making. A series of checks will be performed to estimate several of the data set characteristics. Simple summary statistics for target analytes may be presented, such as the maximum concentration, minimum concentration, number of samples exhibiting no detectable analyte, the number of samples exhibiting detectable analytes, and the proportion of samples with detectable and undetectable analytes. Rejected values and significant deviations from planning documents, if any, will be identified so the planning team can assess their impacts to the attainment of project objectives. Project assumptions will also be evaluated to determine their validity. If assumptions are shown to be invalid the team will assess the impact of the invalid assumption and take actions necessary to mitigate the impact. In extreme cases, a revision of DQOs may be necessary.

The quantitative bias and precision data quality indicators will be reviewed to determine whether any significant bias or significant imprecision exist in the data. A significant bias is a bias greater than +/- 30 percent (corresponding to consistent analyte recoveries of 70 to 130 percent in LCSs, MSs, or surrogate compound concentrations). Laboratory precision and field precision (based on RPD values from duplicate samples) will be compared to ensure that laboratory precision is not significantly worse (i.e., exhibits greater RPD values) than field precision. At the discretion of the project manager correlations may also be assessed among various parameters as a cross-check to ensure that results appear to be reasonable. Although bias and precision indicators can be assessed quantitatively, evaluations will involve professional judgment which will consider site conditions, the normal analytical performance for the analytes in question, and other factors that affect the precision and agreements among analyte concentrations. The intent will be to identify any deviations or anomalies that adversely affect the ability to attain project objectives and to document how these characteristics were handled by the team to complete the project.

RPD and Percent Recovery values will be computed as follows:

$$RPD = \frac{|Amount\ in\ Sample\ 1 - Amount\ in\ Sample\ 2|}{0.5 (Amount\ in\ Sample\ 1 + Amount\ in\ Sample\ 2)} \times 100$$

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5

Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study

Revision Number: 0

Revision Date: September 2008

SAP Worksheet #37 -- Usability Assessment[\(UFP-QAPP Manual Section 5.2.3\)](#)

The %R for a spiked sample will be calculated by using the following formula:

$$\%R = \frac{\text{Amount in Spiked Sample} - \text{Amount in Sample}}{\text{Known Amount Added}} \times 100 \%$$

The %R calculation for LCSs and surrogate spikes will be as follows:

$$\%R = \frac{\text{Experimental Concentration}}{\text{Certified or Known Concentration}} \times 100 \%$$

Potential data outliers will also be investigated to determine whether they represent unanticipated site conditions or they are true outliers. No statistical outlier will be removed from a data set unless a physical reason can be assigned to the datum to demonstrate that it is not representative of the intended population.

The comparability among data sets will be evaluated to ensure it is satisfactory. For example, if comparability problems are anticipated because of poor analyte precision or bias this will be noted so that subsequent data generators or project planners can take these potential data deficiencies into account. Comparability and representativeness assessments will be based on professional judgment with consideration of the quantitative quality indicators such as precision, accuracy, and completeness of data sets.

Data Limitations and Actions from Usability Assessment

After all data evaluations are completed, any limitations on the use of data will be known to the planning team and the limitations will be considered during decision making. If necessary, investigation objectives may be revised in anticipation of additional data collection in order to meet project quality objectives for the site. The data usability assessments for each stage of the bioremediation pilot study will be summarized in the final report for that phase.

Project-Specific SAP

Site Name/Project Name: NAS JRB Willow Grove Site 5
Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study
Revision Number: 0
Revision Date: September 2008

REFERENCES

TtNUS, 2002. Remedial Investigation Report for Site 5 – Fire Training Area. February.

TtNUS, 2004. Draft Feasibility Study for Site 5 – Fire Training Area Groundwater (OU 2). October.

TtNUS, 2006a. Soil Investigation for Volatile Organic Compounds, Soil to Groundwater Impact, Site 5 – Fire Training Area. March.

TtNUS, 2006b. Remedial Investigation Addendum for Site 5 – Fire Training Area Groundwater (OU 2). September.

U.S. DOD, 2006. Department of Defense Quality Systems Manual. January.

U.S. DOD, U.S. DOE, USEPA, 2005. Interagency Data Quality Task Force Uniform Federal Policy for Quality Assurance Project Plans. March

USEPA, 1986. Test Methods for Evaluating Solid Waste; Physical/Chemical Methods (SW-846), 3rd Edition (including Update III). Office of Solid Waste and Emergency Response, Washington, DC.

USEPA, 1993. Region III Modifications to the Laboratory Data Validation Functional Guidelines for Evaluating Inorganics Analyses. April.

USEPA, 1994. Region III Modifications to National Functional Guidelines for Organic Data Review. September.

USEPA, 2006. Guidance on Systematic Planning using the Data Quality Objectives Process. EPA QA/G-4, EPA/240/B-06/001. USEPA Office of Environmental Information, Washington DC. February.

TABLES

**TABLE 1-1
WELL CONSTRUCTION DETAILS
SITE 5 - FIRE TRAINING AREA
NAS JRB WILLOW GROVE, PENNSYLVANIA**

Previous Designation	Well Depth (feet bgs)	Screen Interval (feet bgs)
05MW01S	32	12 - 32
05MW01SI	84.5	74.5 - 84.5
05MW01I	135	125 - 135
05MW02S	30	10 - 30
05MW03S	31	11 - 31
05MW03I	128	118 - 128
05MW04S	30	10 - 30
05MW04I	84.5	74.5 - 84.5
05MW05S	40	20 - 40
05MW05I	209.5	189.5 - 209.5
05MW06S	37.5	17.5 - 37.5
05MW06I	84	74 - 84
05MW07S	26	6 - 26
05MW07I	84	74 - 84
05MW08S	36	26 - 36
05MW08SI	65	55 - 65
05MW08I	99	89 - 99
05MW09S	32	27 - 32
05MW09SI	74	59 - 74
05MW09I	106	96 - 106
05MW10S	32	22 - 32
05MW10SI	94	79 - 94
05MW10I	126	116 - 126
05MW11S	25	20 - 25
05MW11I	50	40 - 50
05MW11D	149	139 - 149
05MW12S	70	50 - 70
05MW12I	113.5	103.5 - 113.5
05MW13I	142	127 - 142
05MW14S	66	36 - 66
05MW14I	150	130 - 150
05MW15S	45	30 - 45
05MW15I	150	140 - 150
*03MW8S	68	38 - 68
*03MW8D	173	163 - 173

TOC = Top of casing bgs = Below ground surface
 * = Wells installed as part of the Site 3 RI, but also applicable to Site 5.

Table 1-2
Page 2 of 8

DATA SUMMARY OF ANALYTICAL RESULTS
MONITORING WELL SAMPLES
NAS JRB WILLOW GROVE SITE 5 - FORMER FIRE TRAINING AREA

Sample ID:	05-MW01-I	05-MW01-S	05-MW01-S-D	05-MW01-SI	05-MW02-S	05-MW03-I	05-MW03-I-D	05-MW03-S	05-MW04-I	05-MW04-S	05-MW05-I
Sample Date:	08/17/05	08/18/05	08/18/05	08/18/05	08/17/05	08/16/05	08/16/05	08/16/05	08/16/05	08/16/05	08/10/05
Dup Of:			05-MW01-S				05-MW03-I				
SEMIVOLATILES											
	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L
1,4-Dioxane	2.1 U	13	12	2.1 U	2.1 U	2.1 U	2.1 U	2.1 U	2.1 U	2.1 U	2.1 U
VOLATILES											
	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L
1,1,1-Trichloroethane	0.45 J	930	960	15	0.16 U	1.3	1.3	0.16 U	25	0.16 U	0.16 U
1,1,2-Trichloroethane	0.11 U	23	22	0.66 J	0.11 U	0.11 U	0.11 U	0.11 U	0.7 J	0.11 U	0.11 U
1,1-Dichloroethane	0.85 J	480	500	26	0.17 U	5.7	5.7	0.17 U	24	0.17 U	0.17 U
1,1-Dichloroethene	0.41 J	180	190	12	0.19 U	1.8	1.8	0.19 U	16	0.19 U	2.4
1,2-Dichlorobenzene	0.08 U	1.7	1.8	0.08 U	0.08 U	0.08 U	0.08 U	0.08 U	0.08 U	0.08 U	0.08 U
1,2-Dichloroethane	0.13 U	4.1	4.1	3.8	0.13 U	0.13 U	0.13 U	0.13 U	0.4 J	0.13 U	0.13 U
1,2-Dichloroethene (cis)	0.09 U	270	270	5.5	0.61 J	1.4	1.4	0.09 U	5.7	0.09 U	0.09 U
1,2-Dichloroethene (trans)	0.1 U	0.89 J	0.93 J	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U	0.1 U
1,4-Dichlorobenzene	0.12 U	7.6	8.2	0.12 U	0.12 U	0.12 U	0.12 U	0.12 U	0.12 U	0.12 U	0.12 U
Acetone	1.6 UR	1.6 UR	1.6 UR	1.6 UR	1.6 UR	1.6 UR	1.6 UR	1.6 UR	1.6 UR	1.6 UR	1.6 UR
Benzene	0.15 U	8.5	8.8	0.15 U	0.15 U	0.15 U	0.15 U	0.15 U	0.15 U	0.15 U	0.15 U
Chloroethane	0.46 U	0.63 J	0.62 J	0.46 U	0.46 U	0.46 U	0.46 U	0.46 U	0.46 U	0.46 U	0.46 U
Chloroform	0.16 U	0.33 J	0.33 J	0.16 U	0.16 U	0.16 U	0.16 U	0.16 U	0.16 U	0.16 U	0.16 U
m-p-xylenes	0.24 U	0.24 U	0.24 U	0.24 U	0.24 U	0.24 U	0.24 U	0.24 U	0.24 U	0.24 U	0.24 U
Methyl Cyclohexane	0.14 U	2.3	2	0.14 U	0.14 U	0.14 U	0.14 U	0.14 U	0.14 U	0.14 U	0.14 U
Methylene Chloride	0.78 J	1.2 B	1.1 B	0.42 U	0.42 U	0.42 U	0.42 U	0.42 U	0.42 U	0.42 U	0.42 U
Tetrachloroethene	0.12 UJ	50 J	49 J	2.5	1.7	1.2	1.1	0.12 U	1.5	0.12 U	0.12 U
Toluene	0.11 U	0.11 U	0.11 U	0.11 U	0.11 U	0.11 U	0.11 U	0.11 U	0.11 U	0.11 U	0.11 U
Trichloroethene	0.36 J	470	490	10	0.12 U	1.4	1.4	0.12 U	14	0.12 U	1 J
Xylene (Total)	0.37 U	0.37 U	0.37 U	0.37 U	0.37 U	0.37 U	0.37 U	0.37 U	0.37 U	0.37 U	0.37 U
MISCELLANEOUS											
Chloride (mg/L)	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Nitrate (mg/L)	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Sulfate (mg/L)	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Oxidation Reduction Potential (mV)	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Total Organic Carbon (mg/L)	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Ethane (ug/L)	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Ethene (ug/L)	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Methane (ug/L)	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Dissolved Oxygen (mg/L)	3.83	0.99	NA	5.12	9.14	8.92	NA	9.35	6.14	3.32	NA
pH	7.22	5.98	NA	4.83	4.2	4.52	NA	5.37	6.14	4.17	6.25
Specific Conductivity (mS/cm)	0.305	0.218	NA	0.196	0.127	0.165	NA	0.126	0.16	0.112	0.367
Turbidity (NTU)	6	0	NA	2	0	8.3	NA	0	55	5.9	30

Table 1-2
Page 3 of 8

DATA SUMMARY OF ANALYTICAL RESULTS
MONITORING WELL SAMPLES
NAS JRB WILLOW GROVE SITE 5 - FORMER FIRE TRAINING AREA

Sample ID:	05-MW05-S	05-MW06-I	05-MW06-S	05-MW07-I	05-MW07-S	05-MW08-I	05-MW08-S	05-MW08-SI	05-MW08-SI-D	05-MW09-I	05-MW09-S
Sample Date:	08/10/05	08/12/05	08/12/05	08/09/05	08/09/05	08/18/05	08/12/05	08/12/05	08/12/05	08/17/05	08/17/05
Dup Of:									05-MW08-SI		
SEMIVOLATILES	ug/L	ug/L	ug/L	ug/L							
1,4-Dioxane	2.1 U	2 U	2.1 U	2.1 U	2.1 U	2.1 U	2.1 U	2.1 U	2 U	2.1 U	2.1 U
VOLATILES	ug/L	ug/L	ug/L	ug/L							
1,1,1-Trichloroethane	0.16 U	0.16 U	9.4	8.2							
1,1,2-Trichloroethane	0.11 U	0.11 U	0.11 U	0.11 U							
1,1-Dichloroethane	0.17 U	0.17 U	14	21							
1,1-Dichloroethene	0.19 U	0.19 U	0.19 U	3	0.19 U	0.19 U	0.19 U	0.19 U	0.19 U	13	15
1,2-Dichlorobenzene	0.08 U	0.08 U	0.08 U	0.08 U							
1,2-Dichloroethane	0.13 U	0.13 U	0.78 J	0.4 J							
1,2-Dichloroethene (cis)	0.09 U	0.09 U	2.3	0.8 J							
1,2-Dichloroethene (trans)	0.1 U	0.1 U	0.1 U	0.1 U							
1,4-Dichlorobenzene	0.12 U	0.12 U	0.12 U	0.12 U							
Acetone	1.6 U	1.6 UR	1.6 UR	1.6 U	1.6 U	1.6 UR	1.6 UR	1.6 UR	1.6 UR	1.6 UR	1.6 UR
Benzene	0.15 U	0.15 U	0.15 U	0.83 J							
Chloroethane	0.46 U	0.46 U	0.46 U	0.46 U							
Chloroform	0.16 U	0.16 U	0.16 U	0.16 U							
M+p-xylenes	0.24 UJ	0.24 U	0.24 U	0.24 UJ	0.24 UJ	0.24 U	0.24 U	0.24 U	0.24 U	0.24 U	0.24 U
Methyl Cyclohexane	0.14 U	0.14 U	0.14 U	0.14 U							
Methylene Chloride	0.42 U	0.42 U	1	0.65 J							
Tetrachloroethene	0.12 U	0.12 U	0.77 J	0.12 U	0.12 U	0.12 UJ	0.12 U	0.12 U	0.12 U	1 J	1.5 J
Toluene	0.11 U	0.11 U	0.11 U	0.11 U							
Trichloroethene	0.12 U	0.12 U	0.12 U	1.6 J	0.12 U	0.12 U	0.12 U	0.12 U	0.12 U	7.3	6.2
Xylene (Total)	0.37 U	0.37 U	0.37 U	0.37 U							
MISCELLANEOUS											
Chloride (mg/L)	NA	NA	NA	NA							
Nitrate (mg/L)	NA	NA	NA	NA							
Sulfate (mg/L)	NA	NA	NA	NA							
Oxidation Reduction Potential (mV)	NA	NA	NA	NA							
Total Organic Carbon (mg/L)	NA	NA	NA	NA							
Ethane (ug/L)	NA	NA	NA	NA							
Ethene (ug/L)	NA	NA	NA	NA							
Methane (ug/L)	NA	NA	NA	NA							
Dissolved Oxygen (mg/L)	NA	5.2	2.54	2.87	NA	3.82	4.48	0.98	NA	1.73	5.73
pH	4.67	6.32	5.32	6.77	4.48	7.07	5.67	6.16	NA	6.63	4.19
Specific Conductivity (mS/cm)	0.124	0.362	0.141	0.316	0.284	0.267	0.24	0.278	NA	0.317	0.161
Turbidity (NTU)	8.7	4.2	10	6.3	0	6	6.2	18	NA	0	5.4

Table 1-2
Page 4 of 8

DATA SUMMARY OF ANALYTICAL RESULTS
MONITORING WELL SAMPLES
NAS JRB WILLOW GROVE SITE 5 - FORMER FIRE TRAINING AREA

Sample ID:	05-MW09-SI	05-MW10-I	05-MW10-S	05-MW10-SI	05-MW11-D	05-MW11-I	05-MW11-S	05-MW12-I	05-MW12S	05-MW13I	05-MW14I
Sample Date:	08/17/05	08/09/05	08/09/05	08/09/05	08/11/05	08/11/05	08/11/05	08/17/05	08/25/05	08/24/05	08/25/05
Dup Of:											
SEMIVOLATILES	ug/L										
1,4-Dioxane	2.1 U	2 U	2.1 U	2.1 U	2 U	2 U	2.1 U	2.1 U	2 U	2.1 U	2.1 U
VOLATILES	ug/L										
1,1,1-Trichloroethane	16	2.7	22	73	0.16 U	0.16 U	0.16 U	0.16 U	2.3 J	28	20
1,1,2-Trichloroethane	0.64 J	0.11 U	0.11 U	2.6	0.11 U	0.11 U	0.11 U	0.11 U	0.8 J	0.11 U	0.11 U
1,1-Dichloroethane	19	0.17 U	11 J	51	0.17 U	0.17 U	0.17 U	0.17 U	0.79 J	29	13
1,1-Dichloroethene	25	4.6	17	79	0.19 U	0.19 U	0.19 U	0.19 U	3 J	29	24
1,2-Dichlorobenzene	0.08 U										
1,2-Dichloroethane	0.89 J	0.13 U									
1,2-Dichloroethene (cis)	1.9	0.51 J	1.3	14	0.09 U	0.09 U	0.09 U	0.6 J	7.2 J	4.8	2
1,2-Dichloroethene (trans)	0.1 U										
1,4-Dichlorobenzene	0.12 U										
Acetone	1.6 UR	1.6 U	1.6 U	1.6 U	1.6 UR	1.6 UR	1.6 UR	1.6 UR	1.6 U	1.6 UR	1.6 U
Benzene	0.39 J	0.15 U	0.15 U	1.1	0.15 U						
Chloroethane	0.46 U										
Chloroform	0.16 U										
M+p-xylenes	0.24 U										
Methyl Cyclohexane	0.14 U										
Methylene Chloride	1	0.42 U									
Tetrachloroethene	1.5 J	0.12 U	1.5	4	0.12 U	0.12 U	0.12 U	1.1	1.2 J	1	0.12 U
Toluene	0.11 U	0.65 J	0.11 U	0.11 U							
Trichloroethene	13	2.3 J	6.2 J	52	0.12 U	0.12 U	0.12 U	0.12 U	3.7 J	13	7.9
Xylene (Total)	0.37 U	0.61 J	0.37 U	0.37 U							
MISCELLANEOUS											
Chloride (mg/L)	NA	3.52	2.88 L	3.72							
Nitrate (mg/L)	NA	0.613	0.621	0.481							
Sulfate (mg/L)	NA	29	29	28							
Oxidation Reduction Potential (mV)	NA	390	390	400							
Total Organic Carbon (mg/L)	NA	0.4 U	0.4 U	0.419							
Ethane (ug/L)	NA	5 U	5 U	5 U							
Ethene (ug/L)	NA	5 U	5 U	5 U							
Methane (ug/L)	NA	5 U	5 U	5 U							
Dissolved Oxygen (mg/L)	5.42	3.09	NA	NA	NA	NA	NA	3.3	3.19	1.53	1.16
pH	4.7	7.21	4.3	5.12	5.85	4.68	4.69	5.5	5.66	6.97	6.84
Specific Conductivity (mS/cm)	0.265	0.322	0.221	0.37	0.343	1.84	0.182	0.146	0.168	0.385	0.346
Turbidity (NTU)	3.9	3.2	10	9.4	1.9	0	0	110	19	19	9.7

Table 1-2
Page 5 of 8

DATA SUMMARY OF ANALYTICAL RESULTS
MONITORING WELL SAMPLES
NAS JRB WILLOW GROVE SITE 5 - FORMER FIRE TRAINING AREA

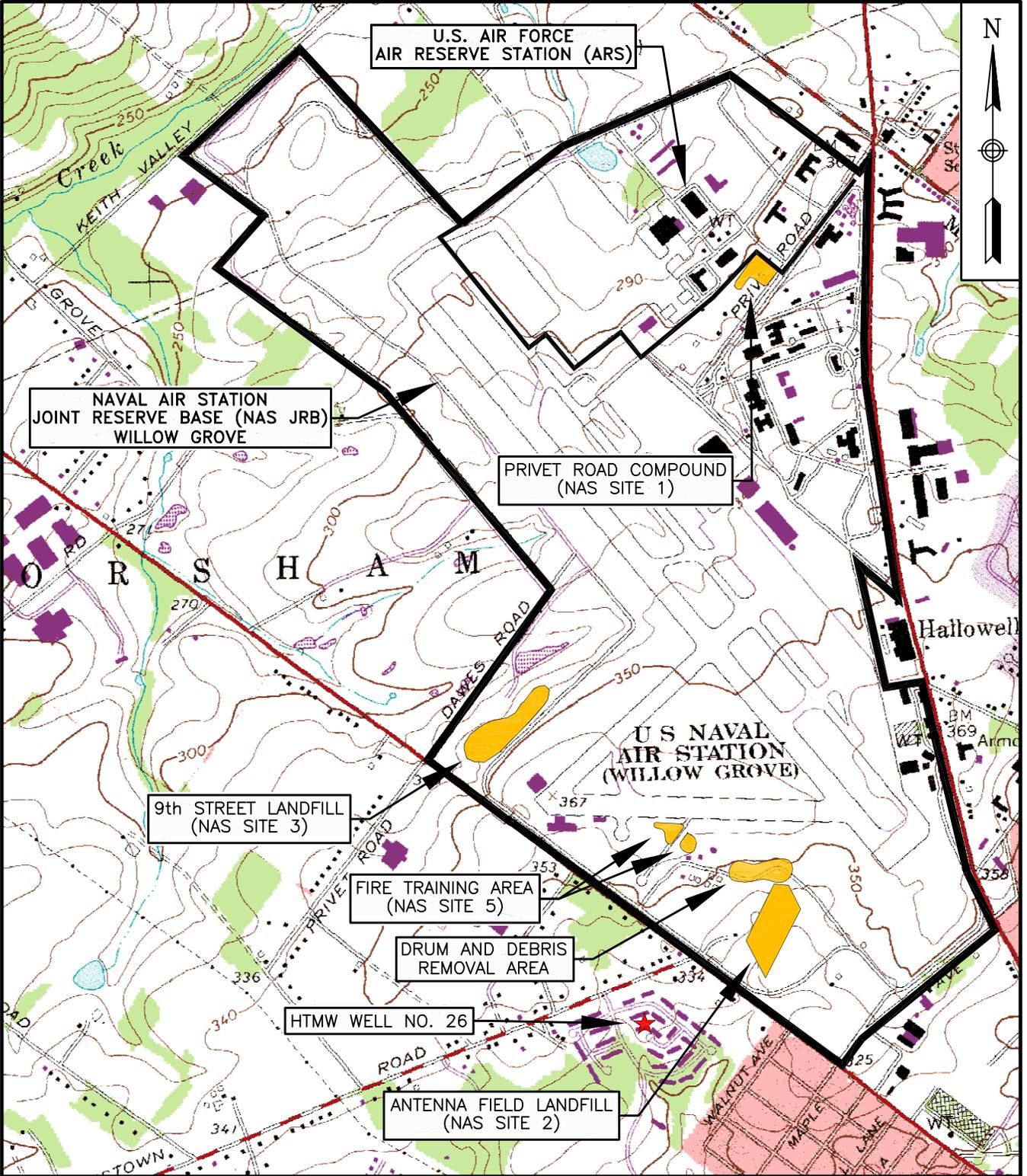
Sample ID:	05-MW14S	05-MW15I	05-MW15S	05MW14I-D	03-MW08-D	03-MW08-S
Sample Date:	08/24/05	08/24/05	08/24/05	08/25/05	01/30/06	01/30/06
Dup Of:				05MW14I		
SEMIVOLATILES						
	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L
1,4-Dioxane	2 U	2 U	2 U	2 U	NA	NA
VOLATILES						
	ug/L	ug/L	ug/L	ug/L		
1,1,1-Trichloroethane	0.16 UJ	2.4	0.16 U	20	3	ND
1,1,2-Trichloroethane	0.11 UJ	0.11 U	0.11 U	0.11 U	NA	NA
1,1-Dichloroethane	0.17 UJ	1.4	0.17 U	14	5.9	ND
1,1-Dichloroethene	0.19 UJ	1.8	0.19 U	23	2.5	ND
1,2-Dichlorobenzene	0.08 UJ	0.08 U	0.08 U	0.08 U	NA	NA
1,2-Dichloroethane	0.13 UJ	0.13 U	0.13 U	0.13 U	NA	NA
1,2-Dichloroethene (cis)	0.09 UJ	0.09 U	0.09 U	2	0.9 J	ND
1,2-Dichloroethene (trans)	0.1 UJ	0.1 U	0.1 U	0.1 U	NA	NA
1,4-Dichlorobenzene	0.12 UJ	0.12 U	0.12 U	0.12 U	NA	NA
Acetone	3.3 J	1.6 UR	1.6 UR	1.6 U	ND	ND
Benzene	0.15 UJ	0.15 U	0.15 U	0.15 U	NA	NA
Chloroethane	0.46 UJ	0.46 U	0.46 U	0.46 U	NA	NA
Chloroform	0.16 UJ	0.16 U	0.16 U	0.16 U	ND	ND
m+p-xylenes	0.24 UJ	0.24 U	0.24 U	0.24 U	NA	NA
Methyl Cyclohexane	0.14 UJ	0.14 U	0.14 U	0.14 U	NA	NA
Methylene Chloride	0.42 UJ	0.42 U	0.42 U	0.42 UJ	ND	ND
Tetrachloroethene	0.12 UJ	0.12 U	0.12 U	0.12 U	0.78 J	ND
Toluene	0.11 UJ	0.11 U	0.11 U	0.11 U	0.19 J	ND
Trichloroethene	0.12 UJ	1	0.12 U	7.5	1.5	ND
Xylene (Total)	0.37 UJ	0.37 U	0.37 U	0.37 U	NA	NA
MISCELLANEOUS						
Chloride (mg/L)	2.97 L	13 L	2.64 L	NA	NA	NA
Nitrate (mg/L)	0.783	0.791	0.678	NA	NA	NA
Sulfate (mg/L)	43	38	36	NA	NA	NA
Oxidation Reduction Potential (mV)	400	400	410	NA	NA	NA
Total Organic Carbon (mg/L)	0.4 U	0.621	0.4 U	NA	NA	NA
Ethane (ug/L)	5 U	5 U	5 U	NA	NA	NA
Ethene (ug/L)	5 U	5 U	5 U	NA	NA	NA
Methane (ug/L)	5 U	5 U	5 U	NA	NA	NA
Dissolved Oxygen (mg/L)	1.02	2.76	4.56	NA	NA	NA
pH	6.82	6.02	6.25	NA	NA	NA
Specific Conductivity (mS/cm)	0.413	0.343	0.247	NA	NA	NA
Turbidity (NTU)	550	3.6	14	NA	NA	NA

Table 1-2
Page 6 of 8

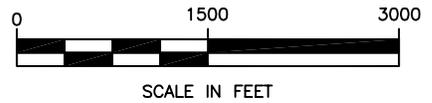
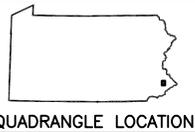
DATA SUMMARY OF ANALYTICAL RESULTS
COMPLETE DATA SET OF NATURAL ATTENUATION PARAMETERS - 2000 AND 2005
NAS JRB WILLOW GROVE SITE 5 - FIRE TRAINING AREA

Sample ID:	BACKGROUND													
	05MW02S	05MW01S	05MW01S1	05MW011	05MW03S	05MW031	05MW04S	05MW041	05MW05S	05MW051	05MW06S	05MW061	05MW07S	
Sample Date:	09/18/00	09/25/00	09/25/00	09/25/00	09/18/00	09/18/00	09/21/00	09/21/00	09/21/00	09/21/00	09/21/00	09/13/00	09/13/00	09/14/00
MISCELLANEOUS	Preliminary Screening													
PARAMETERS	Parameters	mg/L												
Chloride	>2X Background	1.3	14.4	4.5	2.5	1.2	1.8	1.7	2.6	2.9	9.3	2.3	2.9	NA
Ferrous Iron	>1mg/L	0.088	0.0045 B	0.05 U	0.0045 B	0.0045 B	0.0045 B	0.0045 B	0.33	0.078	0.63	0.05 U	0.05 U	NA
Nitrate	<1mg/L	0.74	0.5 U	0.78	1.1	0.94	0.63	0.79	0.75	0.53	0.18 B	1.3	1.1	NA
Sulfate	<20mg/L	29.7	15	19	17.2	27.7	27.4	31.8	23	16.9	27.3	32	10.5	NA
Sulfide	>1mg/L	1 U	1 U	3.8	1 U	1 U	1 U	1 U	1 U	1 U	2.4	1 U	1 U	1 U
Total Alkalinity	>2X Background	10	73.6	109	125	6.4	51.2	7.8	41.6	58.4	163	18.2	189	NA
Total Organic Carbon	>20mg/L	0.48 B	2.6	0.79 B	1 U	0.18 B	0.31 B	1 U	0.32 B	0.38 B	0.8 B	0.55 B	0.4 B	NA
Oxidation Reduction Potential	<50 mV or <-100mV	NS												
Carbon Dioxide	>2X Background	37.1	128.4	71.9	6.3	23.2	45.6	38.2	55.1	76.2	0.4 U	95.6	8.8	119.1
Oxygen	<0.5 mg/L	4.62	1.09	0.75	3.05	5.3	2.92	5.07	4	2.31	1.06	2.46	2.12	0.64
Hydrogen	>1 nM	3.6	6.4	8.8	8	4.1	3.4	7.1	7	>50	18.2	>50	>50	>50
		ug/L												
Ethane	>100 ug/L	0.01	0.03	0.01 U										
Ethene	>10 ug/L	0.01	0.05	0.02	0.01 U									
Methane	>500 ug/L	0.02 U	15.8	0.3	0.02 U	161.9	0.02 U							
Notes:														
	- Bolded means concentration meets the preliminary screening parameters.													
	- NA means not applicable.													
	- NS means not sampled for this analyte.													

FIGURES

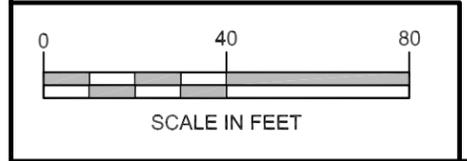
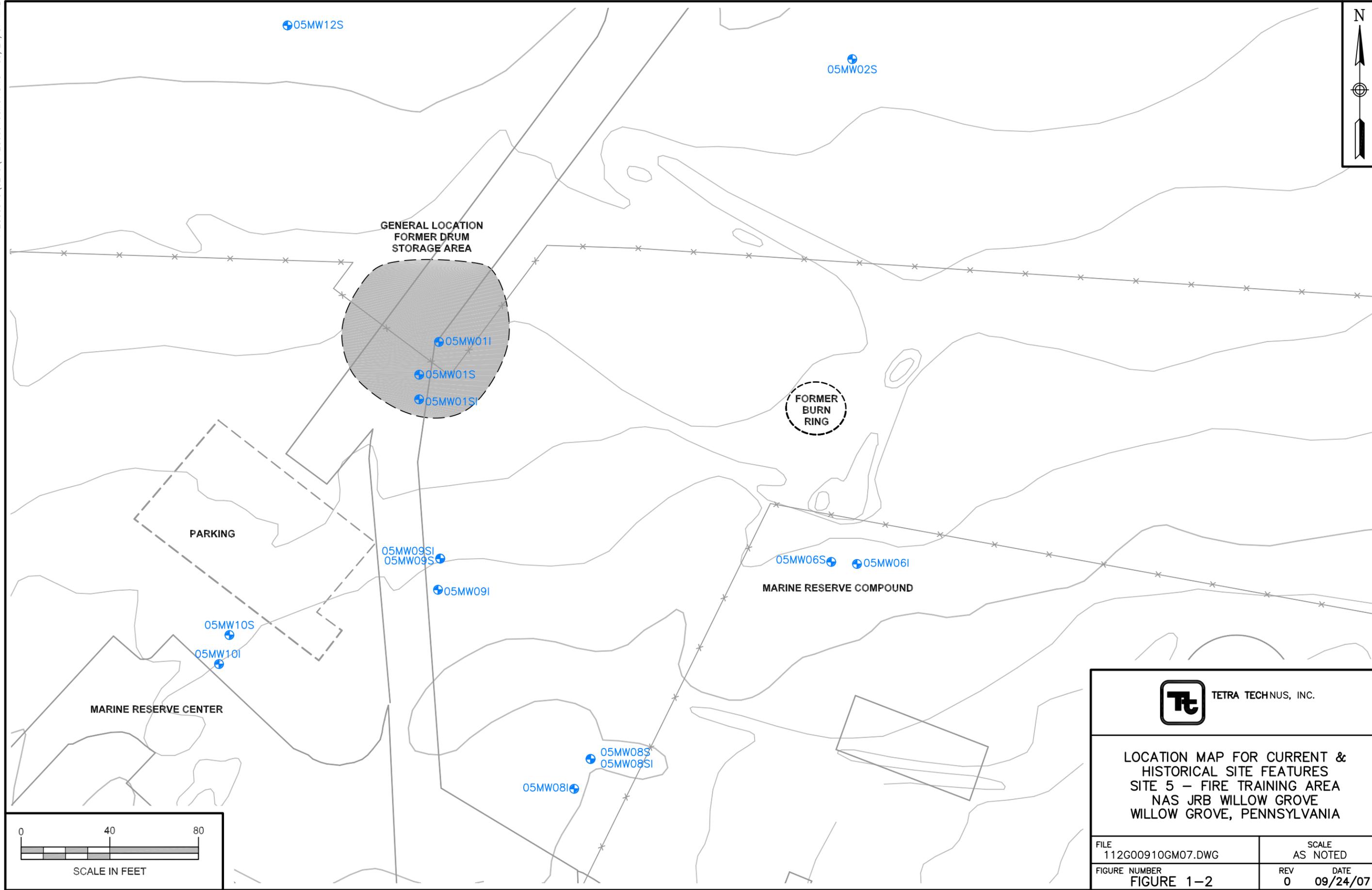


BASE MAP IS A PORTION OF THE AMBLER, PA U.S.G.S. 7.5 MINUTE QUADRANGLE MAP, DATED 1963, PHOTOREVISED IN 1983.

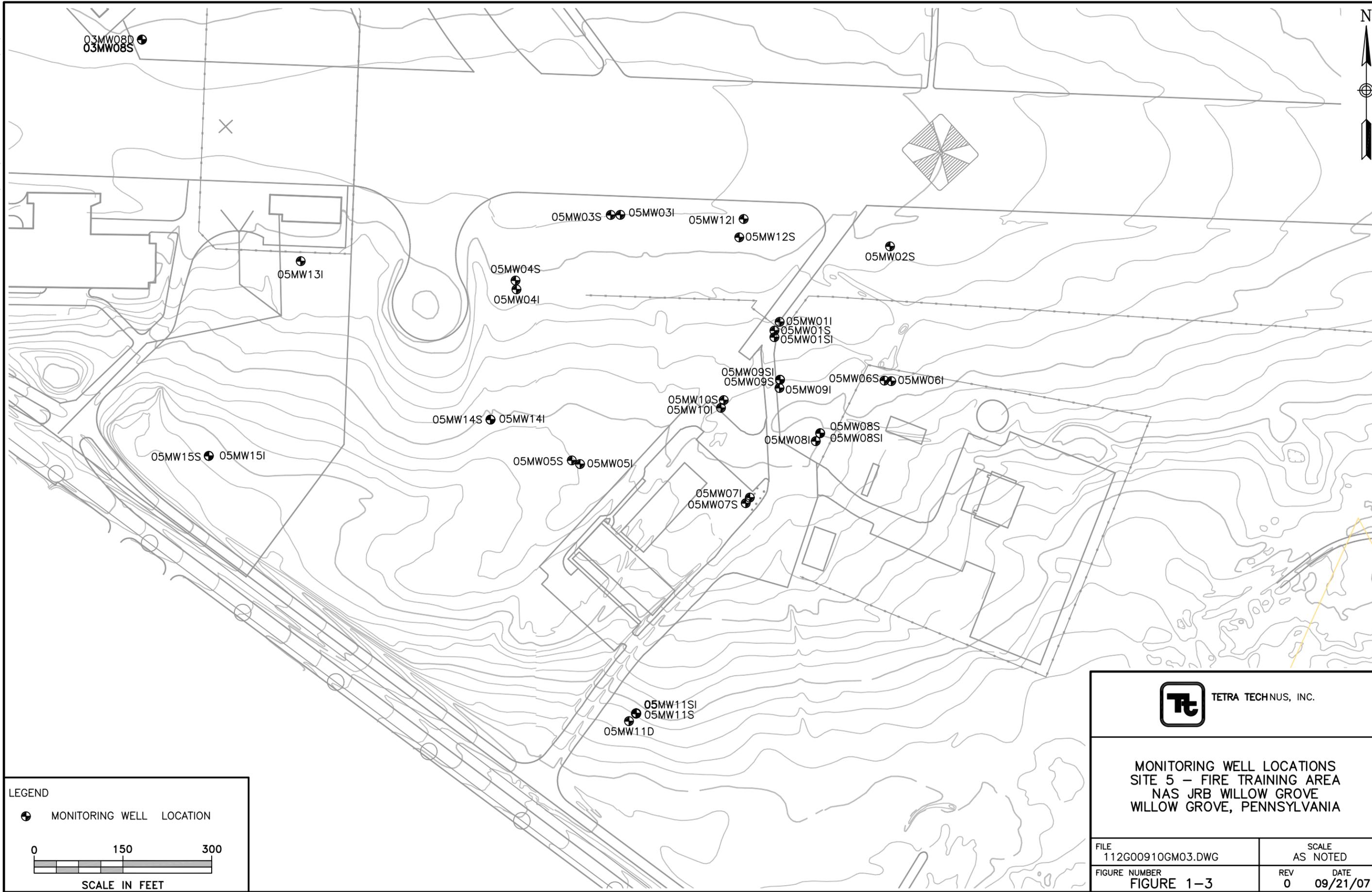


LOCATION OF RI SITES
 SITE 5 – FIRE TRAINING AREA
 NAS JRB WILLOW GROVE
 WILLOW GROVE, PENNSYLVANIA

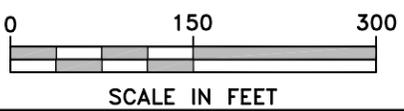
SCALE AS NOTED	
FILE 112G00910CM01	
REV 0	DATE 09/12/07
FIGURE NUMBER FIGURE 1-1	



 TETRA TECHNUS, INC.	
LOCATION MAP FOR CURRENT & HISTORICAL SITE FEATURES SITE 5 – FIRE TRAINING AREA NAS JRB WILLOW GROVE WILLOW GROVE, PENNSYLVANIA	
FILE 112G00910GM07.DWG	SCALE AS NOTED
FIGURE NUMBER FIGURE 1-2	REV DATE 0 09/24/07



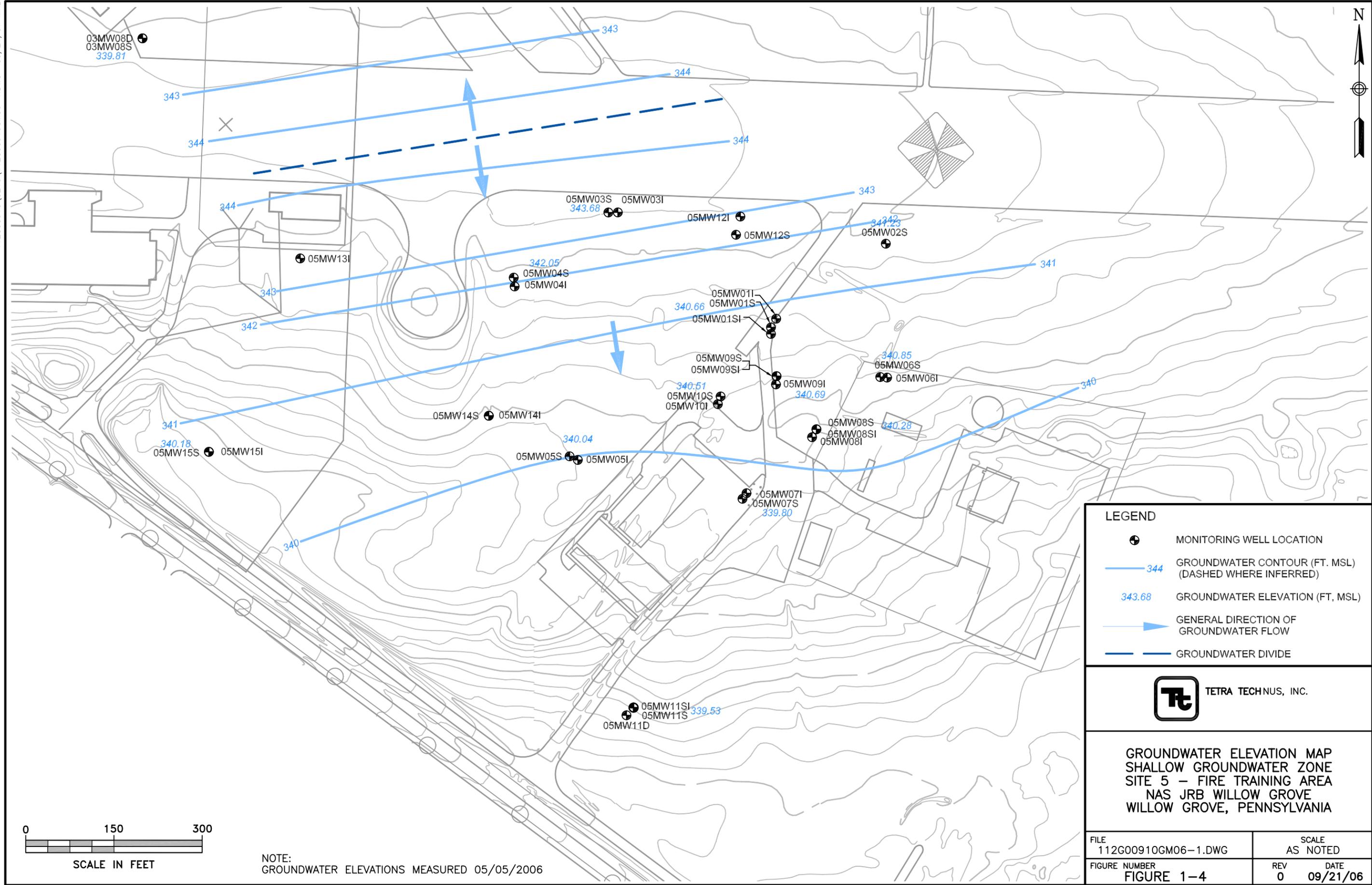
LEGEND
● MONITORING WELL LOCATION



MONITORING WELL LOCATIONS
SITE 5 - FIRE TRAINING AREA
NAS JRB WILLOW GROVE
WILLOW GROVE, PENNSYLVANIA

FILE
112G00910GM03.DWG
FIGURE NUMBER
FIGURE 1-3

SCALE
AS NOTED
REV 0
DATE 09/21/07

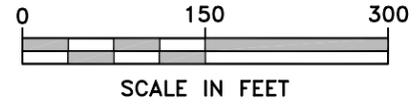


LEGEND

- MONITORING WELL LOCATION
- 344 GROUNDWATER CONTOUR (FT. MSL) (DASHED WHERE INFERRED)
- 343.68 GROUNDWATER ELEVATION (FT. MSL)
- GENERAL DIRECTION OF GROUNDWATER FLOW
- GROUNDWATER DIVIDE

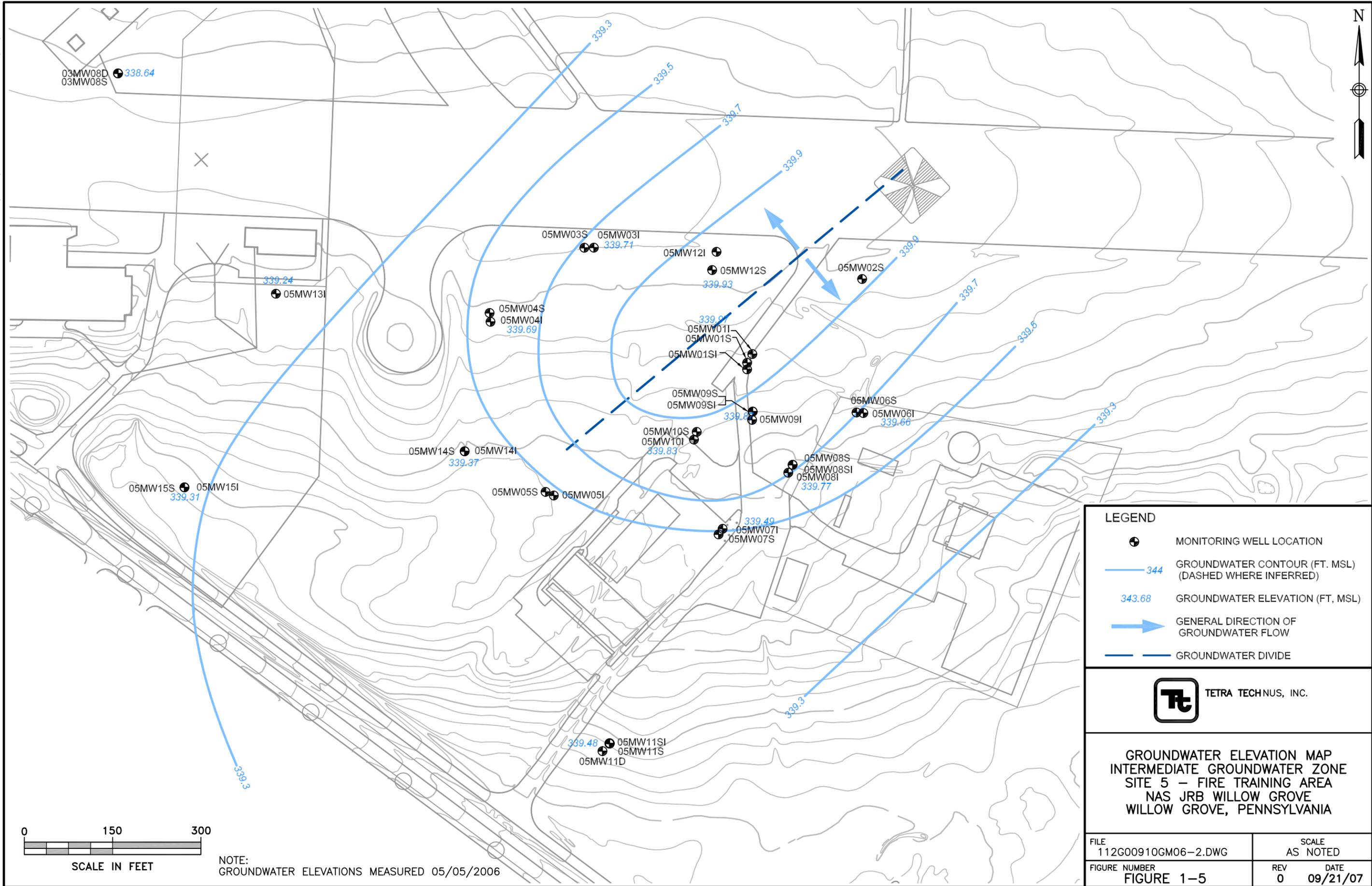


**GROUNDWATER ELEVATION MAP
SHALLOW GROUNDWATER ZONE
SITE 5 – FIRE TRAINING AREA
NAS JRB WILLOW GROVE
WILLOW GROVE, PENNSYLVANIA**



NOTE:
GROUNDWATER ELEVATIONS MEASURED 05/05/2006

FILE 112G00910GM06-1.DWG	SCALE AS NOTED
FIGURE NUMBER FIGURE 1-4	REV DATE 0 09/21/06

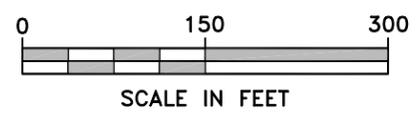


LEGEND	
	MONITORING WELL LOCATION
	344 GROUNDWATER CONTOUR (FT. MSL) (DASHED WHERE INFERRED)
	343.68 GROUNDWATER ELEVATION (FT. MSL)
	GENERAL DIRECTION OF GROUNDWATER FLOW
	GROUNDWATER DIVIDE

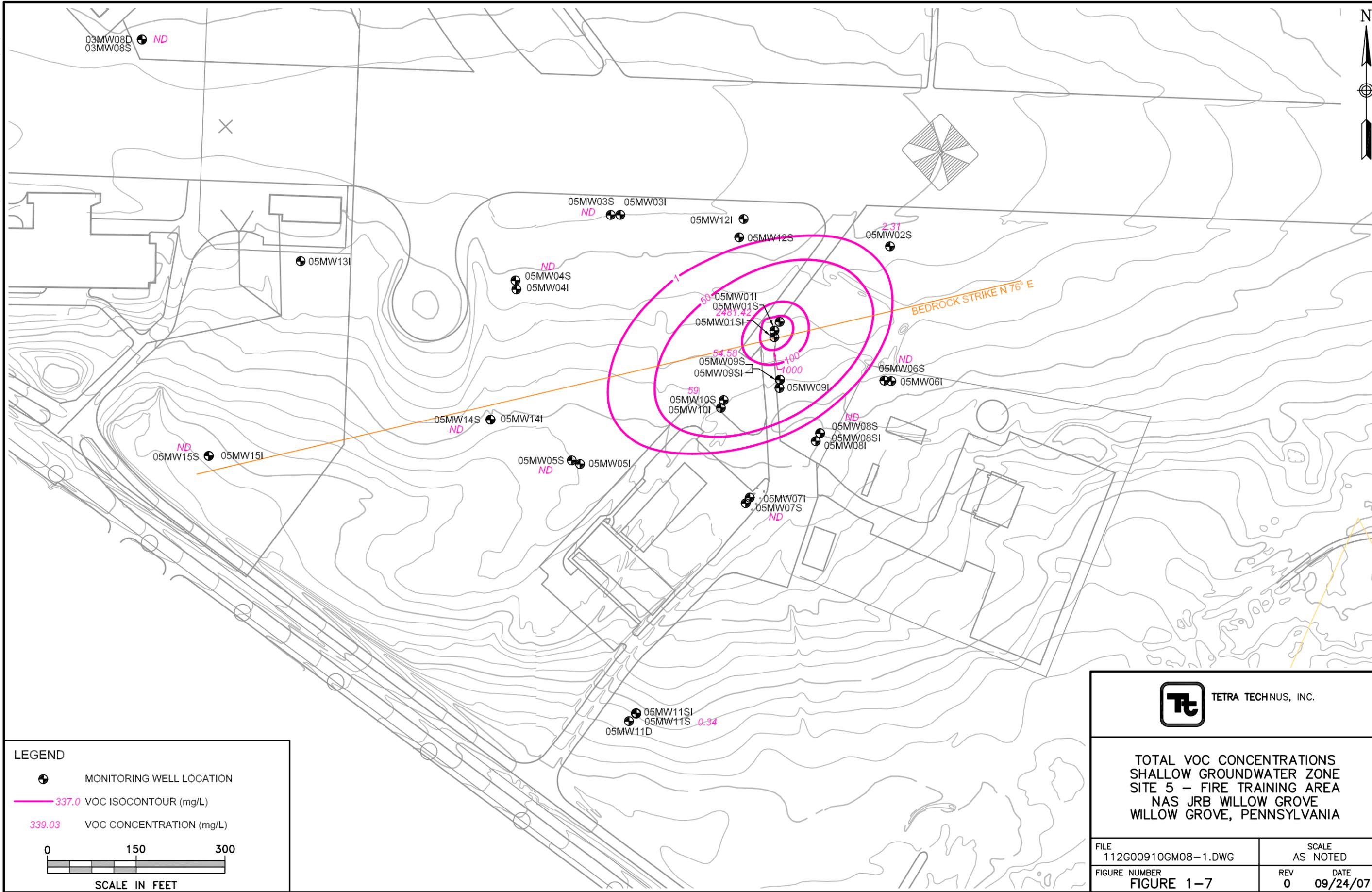


**GROUNDWATER ELEVATION MAP
INTERMEDIATE GROUNDWATER ZONE
SITE 5 – FIRE TRAINING AREA
NAS JRB WILLOW GROVE
WILLOW GROVE, PENNSYLVANIA**

FILE 112G00910GM06-2.DWG	SCALE AS NOTED
FIGURE NUMBER FIGURE 1-5	REV DATE 0 09/21/07



NOTE:
GROUNDWATER ELEVATIONS MEASURED 05/05/2006



LEGEND

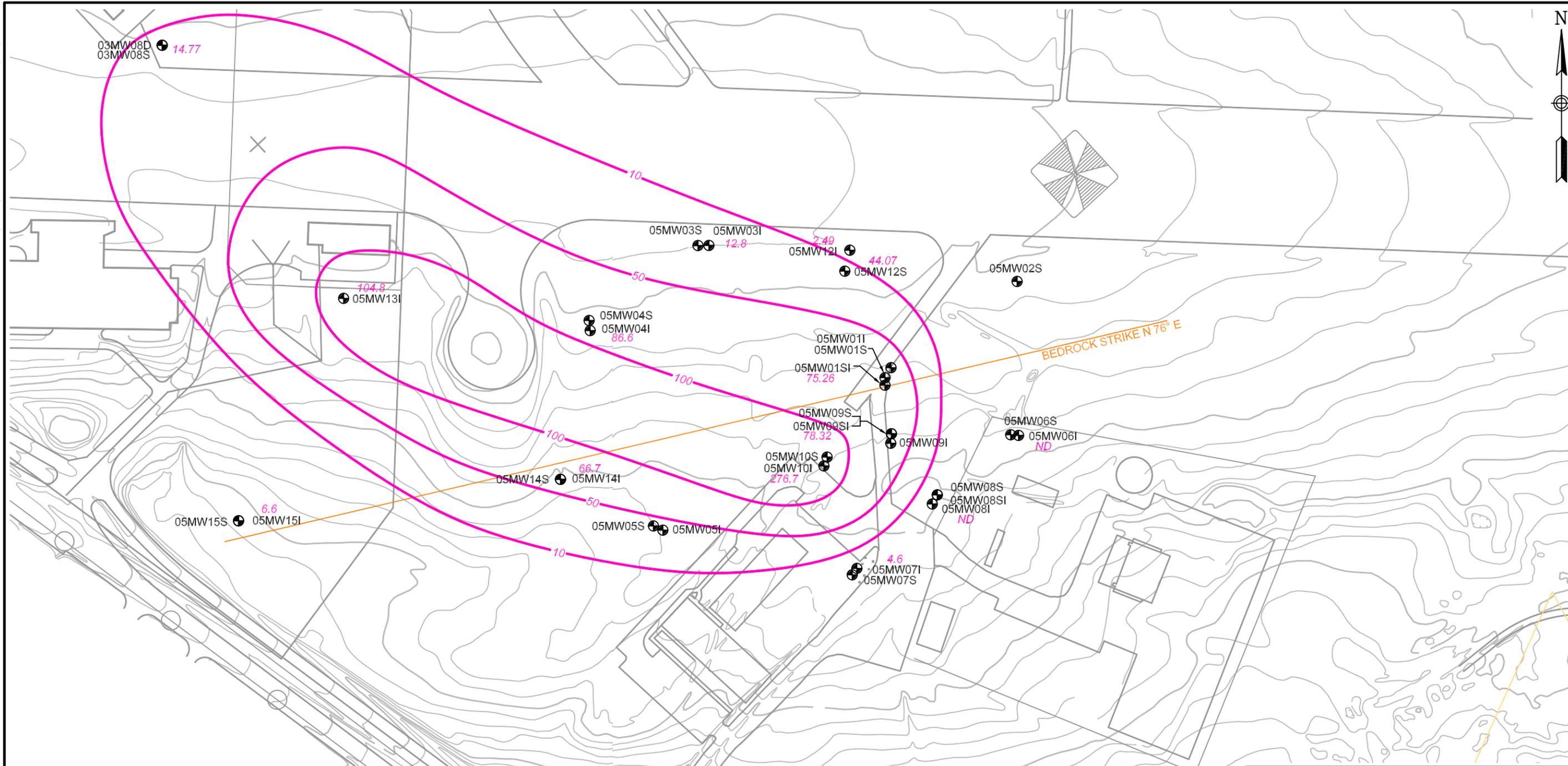
- MONITORING WELL LOCATION
- 337.0 VOC ISOCONTOUR (mg/L)
- 339.03 VOC CONCENTRATION (mg/L)

SCALE IN FEET

TETRA TECHNUS, INC.

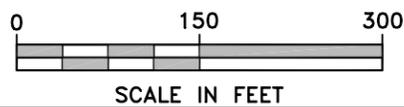
**TOTAL VOC CONCENTRATIONS
SHALLOW GROUNDWATER ZONE
SITE 5 – FIRE TRAINING AREA
NAS JRB WILLOW GROVE
WILLOW GROVE, PENNSYLVANIA**

FILE 112G00910GM08-1.DWG	SCALE AS NOTED
FIGURE NUMBER FIGURE 1-7	REV DATE 0 09/24/07



LEGEND

- MONITORING WELL LOCATION
- 337.0 VOC ISOCONTOUR (mg/L)
- 339.03 VOC CONCENTRATION (mg/L)



TETRA TECHNUS, INC.

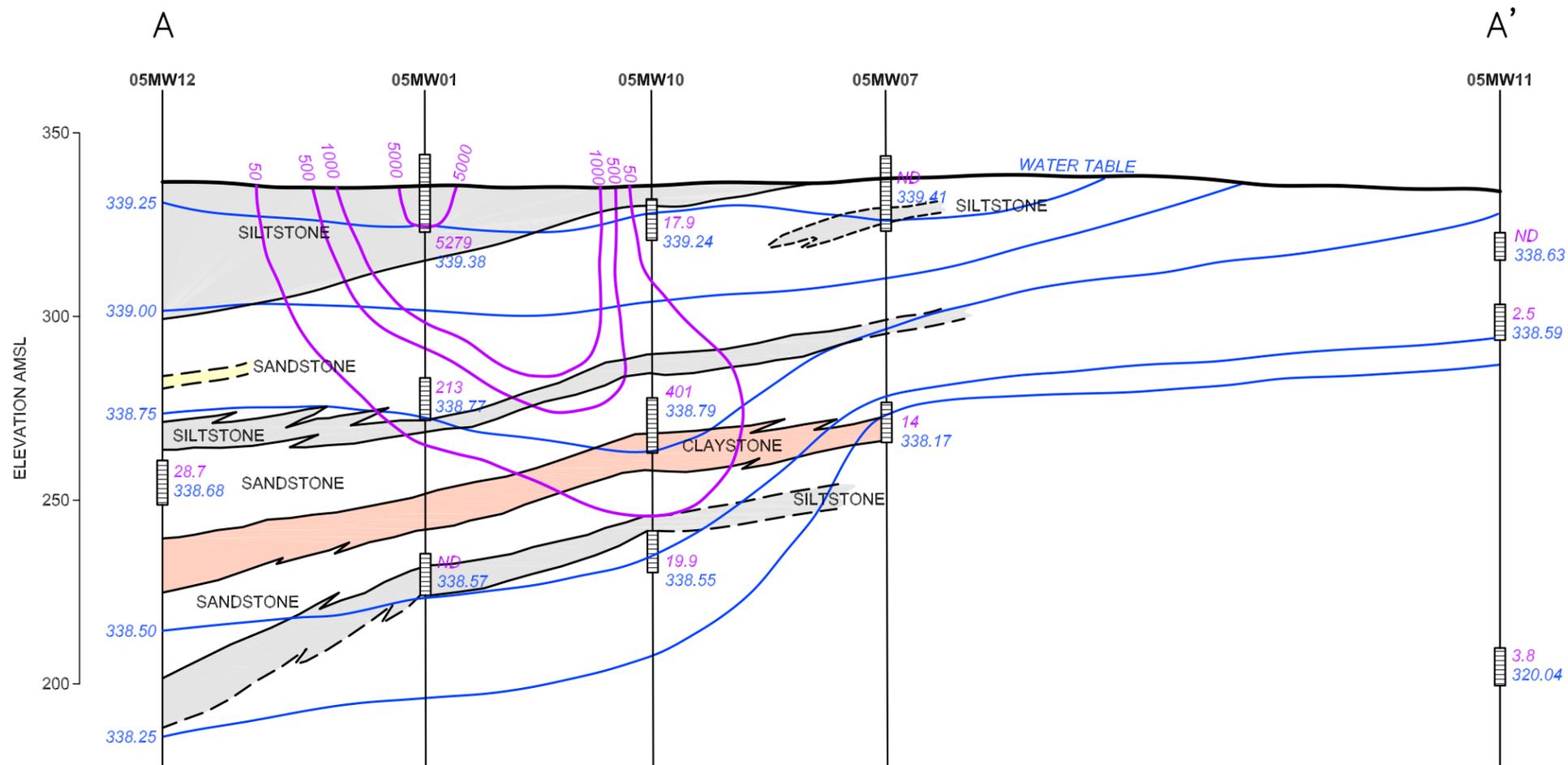
TOTAL VOC CONCENTRATIONS
INTERMEDIATE GROUNDWATER ZONE
SITE 5 - FIRE TRAINING AREA
NAS JRB WILLOW GROVE
WILLOW GROVE, PENNSYLVANIA

FILE
112G00910GM08-2.DWG

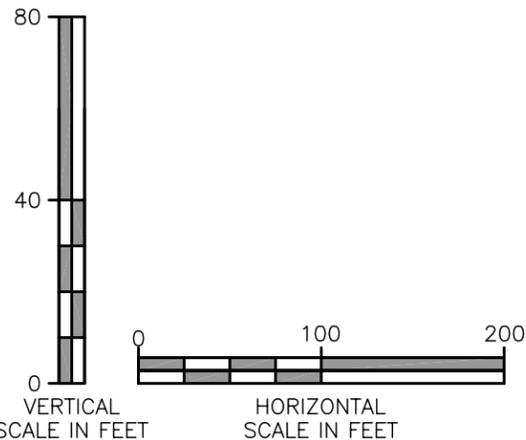
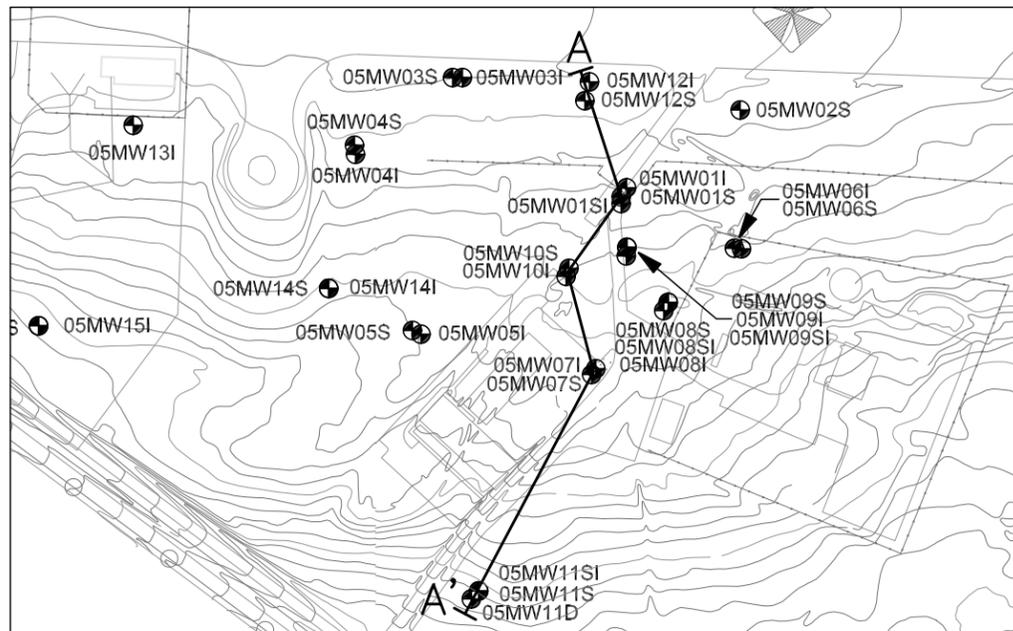
SCALE
AS NOTED

FIGURE NUMBER
FIGURE 1-8

REV DATE
0 09/24/07



CROSS-SECTION LOCATION MAP



LEGEND

- 340 HYDRAULIC HEAD
- 86.6 TOTAL VOCs, ug/L

NOTE:
ANALYTICAL DATA ARE FROM THE SEPTEMBER
2000 SAMPLING EVENT.



TETRA TECHNUS, INC.

HYDROGEOLOGIC CROSS-SECTION A - A'
SITE 5 - FIRE TRAINING AREA
NAS JRB WILLOW GROVE
WILLOW GROVE, PENNSYLVANIA

FILE
2192GS01-1

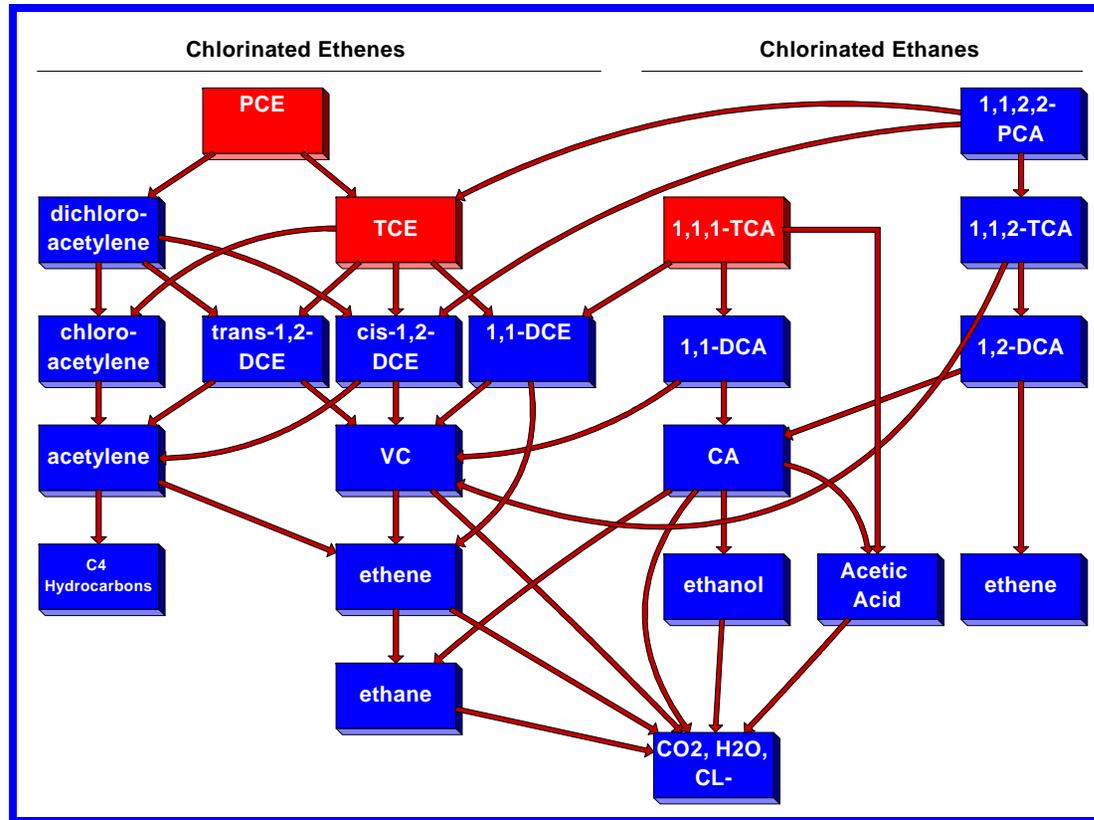
SCALE
AS NOTED

FIGURE NUMBER
FIGURE 1-10

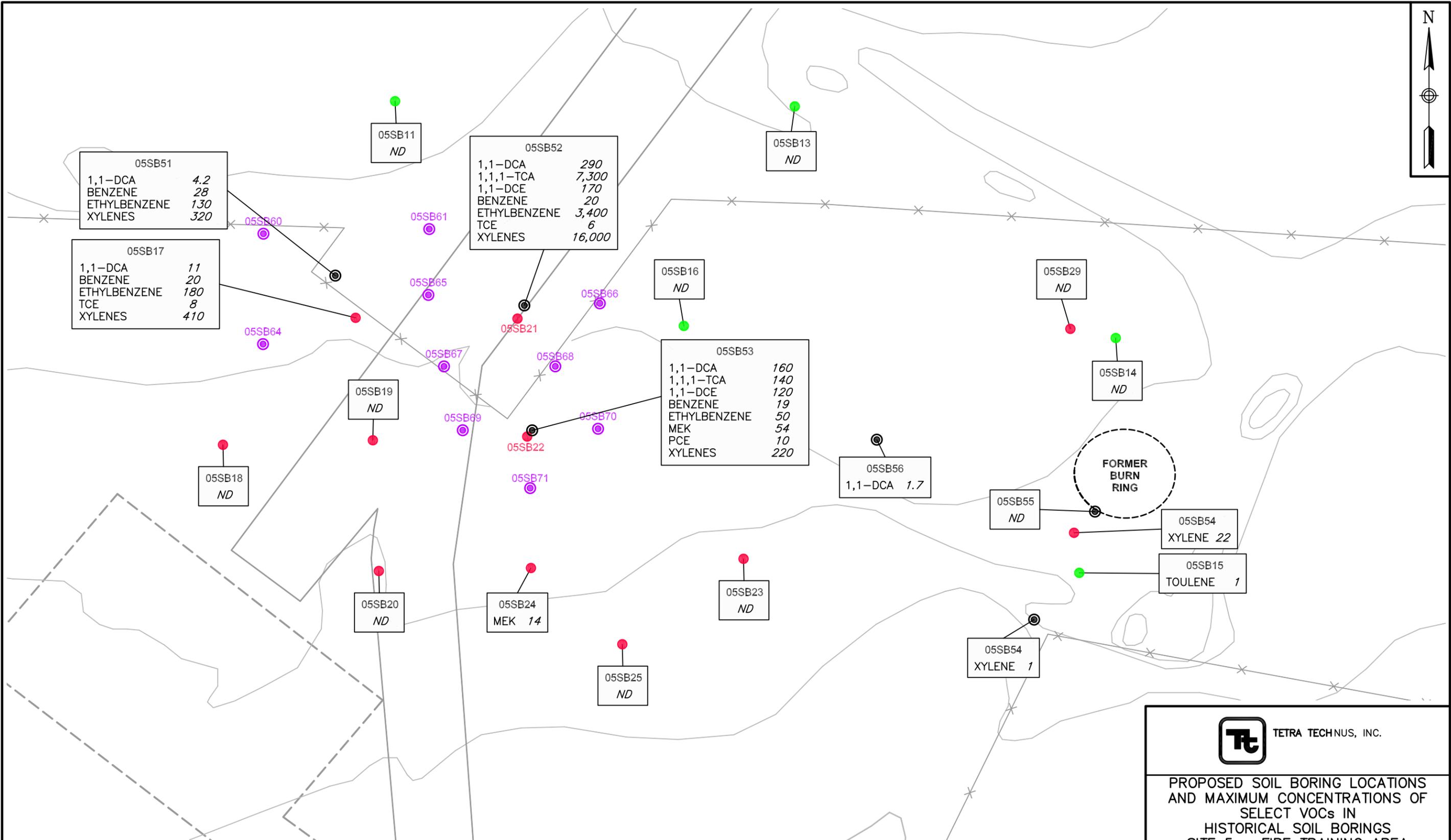
REV DATE
0 02/21/08

FIGURE 1-12

GENERALIZED CHLORINATED ETHENE AND ETHANE DEGRADATION PATHWAYS
SITE 5 FIRE FIGHTING TRAINING AREA
NAS JRB WILLOW GROVE
WILLOW GROVE, PENNSYLVANIA



Abiotic and biotic pathways are shown (both in the presence and absence of zero valent iron). See Section 3.0 for more information. Parent and primary contaminants detected are shown in red and daughter products are shown in blue. Modified after Vogel et al., 1987; Chen, et al., 1996; McCarty and Semprini, 1994; Arnold and Roberts, 2000; Sajeed Jamal, 1997 and EPA Region 4, 1997.



05SB51

1,1-DCA	4.2
BENZENE	28
ETHYLBENZENE	130
XYLENES	320

05SB17

1,1-DCA	11
BENZENE	20
ETHYLBENZENE	180
TCE	8
XYLENES	410

05SB52

1,1-DCA	290
1,1,1-TCA	7,300
1,1-DCE	170
BENZENE	20
ETHYLBENZENE	3,400
TCE	6
XYLENES	16,000

05SB53

1,1-DCA	160
1,1,1-TCA	140
1,1-DCE	120
BENZENE	19
ETHYLBENZENE	50
MEK	54
PCE	10
XYLENES	220

05SB56

1,1-DCA	1.7
---------	-----

05SB54

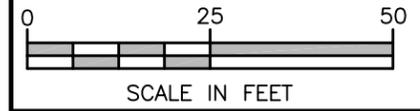
XYLENE	22
--------	----

05SB15

TOULENE	1
---------	---

LEGEND

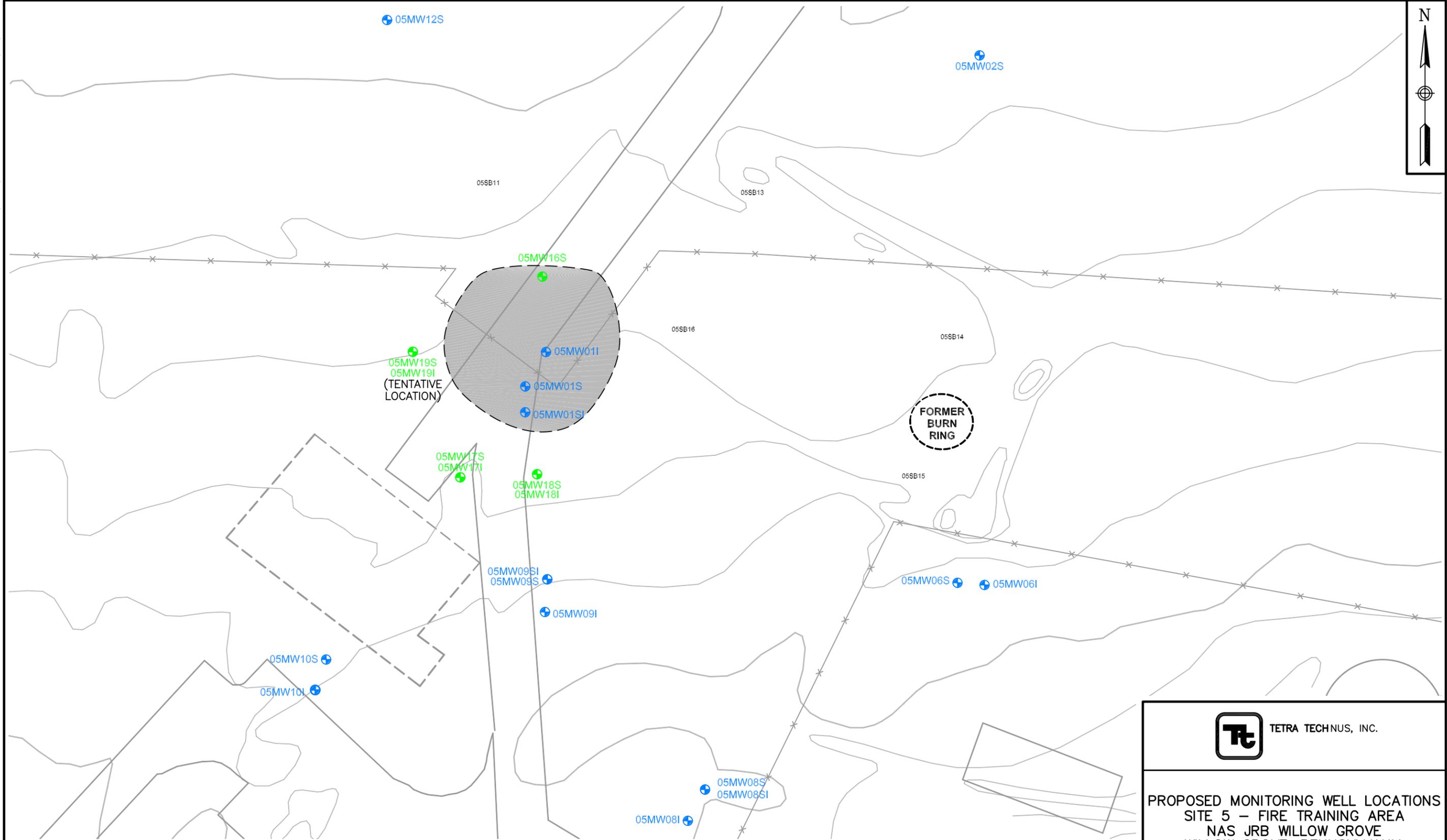
- SOIL BORING/SURFACE SOIL LOCATION, 1991
 - SOIL BORING/SURFACE SOIL LOCATION, 1997
 - SOIL BORING LOCATION, 2005
 - PROPOSED SOIL BORING LOCATION
- 54 VOC CONCENTRATIONS EXPRESSED IN UNITS OF ug/kg



TETRA TECHNUS, INC.

**PROPOSED SOIL BORING LOCATIONS
AND MAXIMUM CONCENTRATIONS OF
SELECT VOCs IN
HISTORICAL SOIL BORINGS
SITE 5 – FIRE TRAINING AREA
NAS JRB WILLOW GROVE
WILLOW GROVE, PENNSYLVANIA**

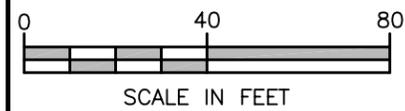
FILE 112G00910GM05-3.DWG	SCALE AS NOTED
FIGURE NUMBER FIGURE 2-1	REV DATE 0 07/29/08



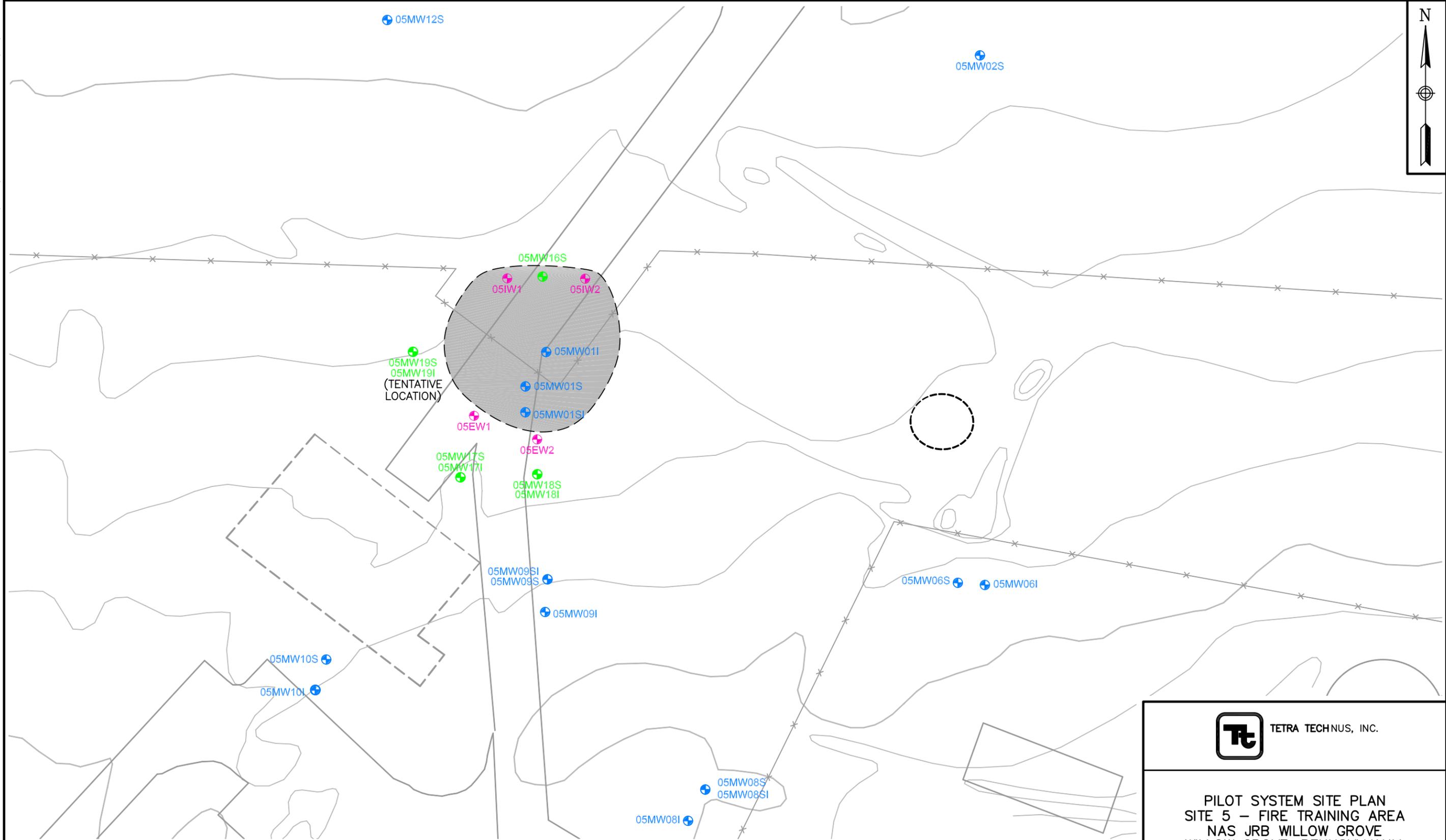
LEGEND

-  EXISTING MONITORING WELL LOCATION
-  PROPOSED NEW MONITORING WELL LOCATION

-  APPROXIMATE EXTENT OF SOURCE AREA BASED ON EXISTING SOIL ANALYTICAL DATA (TO BE REFINED DURING THIS PILOT TEST)

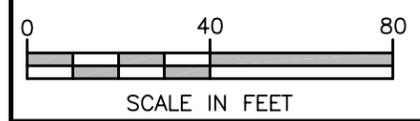


 TETRA TECHNUS, INC.	
PROPOSED MONITORING WELL LOCATIONS SITE 5 – FIRE TRAINING AREA NAS JRB WILLOW GROVE WILLOW GROVE, PENNSYLVANIA	
FILE 112G00910GM01-1.DWG	SCALE AS NOTED
FIGURE NUMBER FIGURE 2-2	REV DATE 0 09/17/07

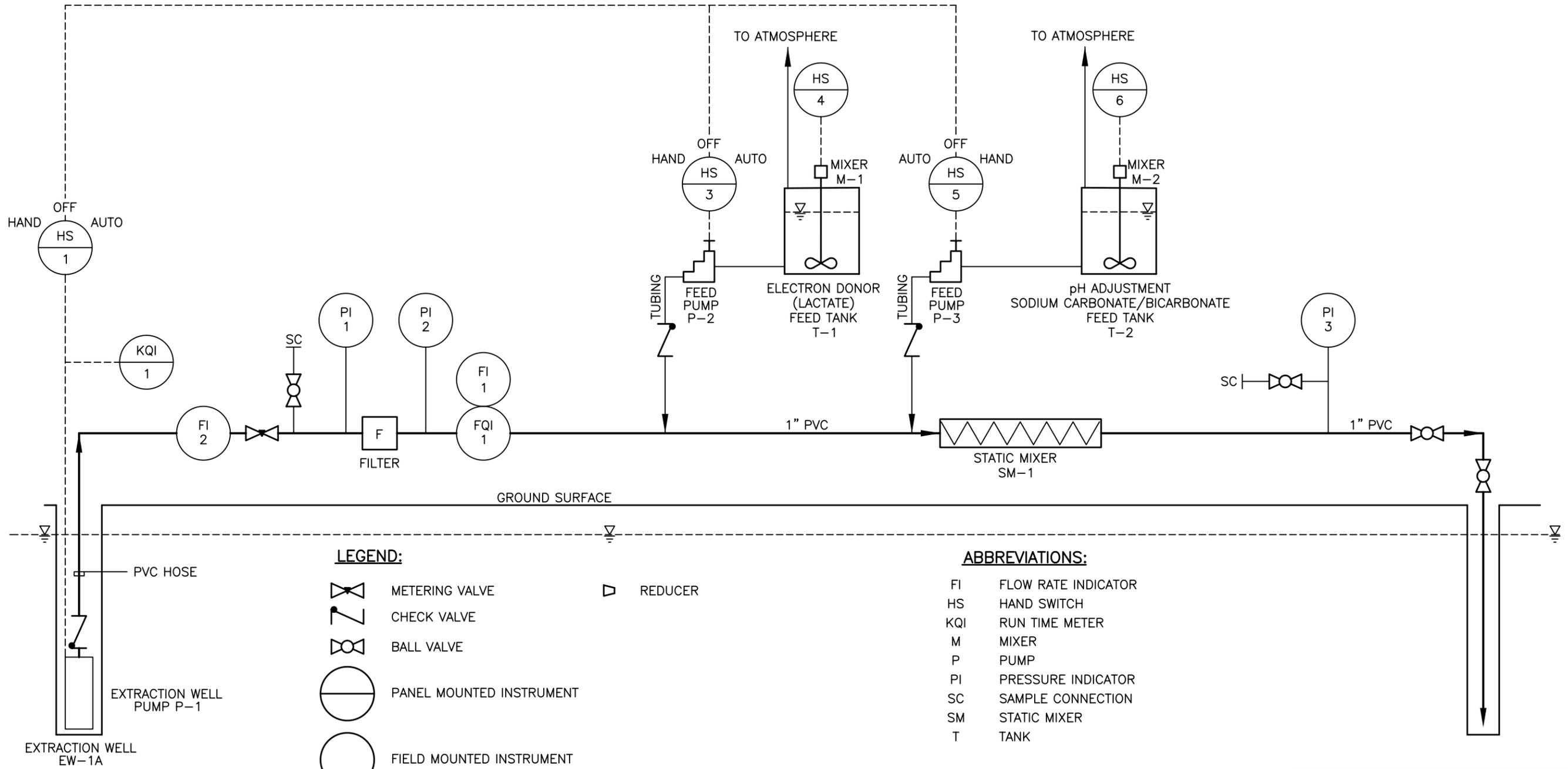


LEGEND

-  EXISTING MONITORING WELL LOCATION
-  GENERAL INJECTION AND EXTRACTION WELL LOCATION (TO BE DETERMINED AFTER THE COMPLETION OF THE PHASE I INVESTIGATION)
-  PROPOSED NEW MONITORING WELL LOCATION
-  APPROXIMATE EXTENT OF SOURCE AREA BASED ON EXISTING SOIL ANALYTICAL DATA (TO BE REFINED DURING THIS PILOT TEST)



 TETRA TECHNUS, INC.	
PILOT SYSTEM SITE PLAN SITE 5 – FIRE TRAINING AREA NAS JRB WILLOW GROVE WILLOW GROVE, PENNSYLVANIA	
FILE 112G00910GM01-2.DWG	SCALE AS NOTED
FIGURE NUMBER FIGURE 4-1	REV DATE 0 09/17/07



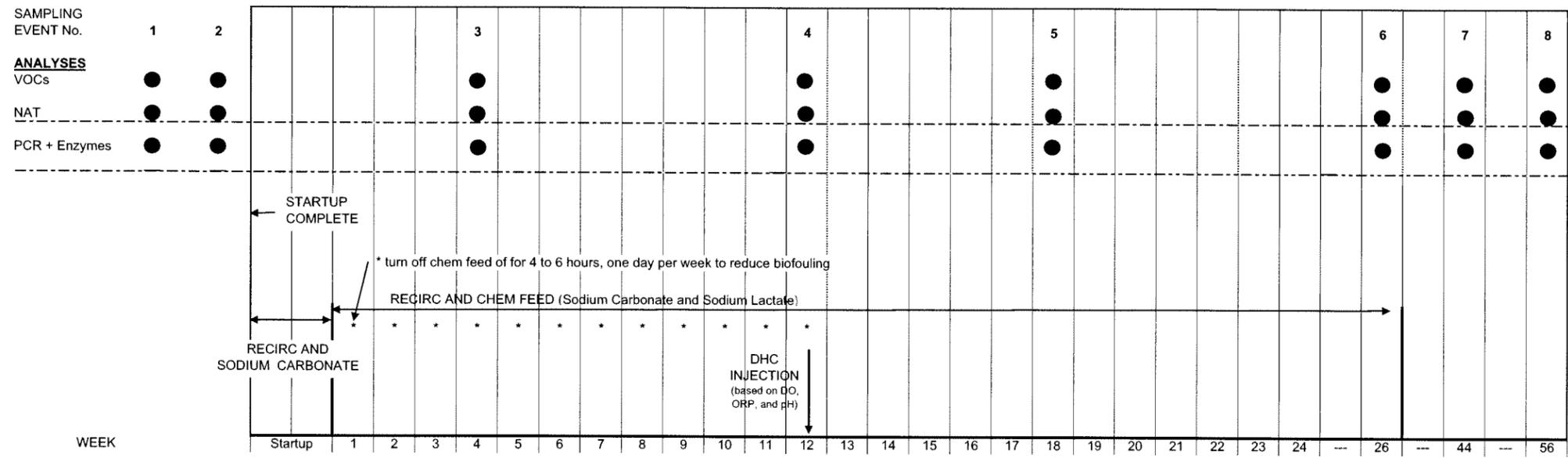
- LEGEND:**
- METERING VALVE
 - CHECK VALVE
 - BALL VALVE
 - PANEL MOUNTED INSTRUMENT
 - FIELD MOUNTED INSTRUMENT
 - WATER LEVEL
 - METERING PUMP
 - ELECTRICAL CONNECTION
 - REDUCER

- ABBREVIATIONS:**
- FI FLOW RATE INDICATOR
 - HS HAND SWITCH
 - KQI RUN TIME METER
 - M MIXER
 - P PUMP
 - PI PRESSURE INDICATOR
 - SC SAMPLE CONNECTION
 - SM STATIC MIXER
 - T TANK



PIPING AND INSTRUMENTATION DIAGRAM
 PILOT STUDY WORK PLAN
 SITE 5 – FIRE TRAINING AREA
 NAS JRB WILLOW GROVE
 WILLOW GROVE, PENNSYLVANIA

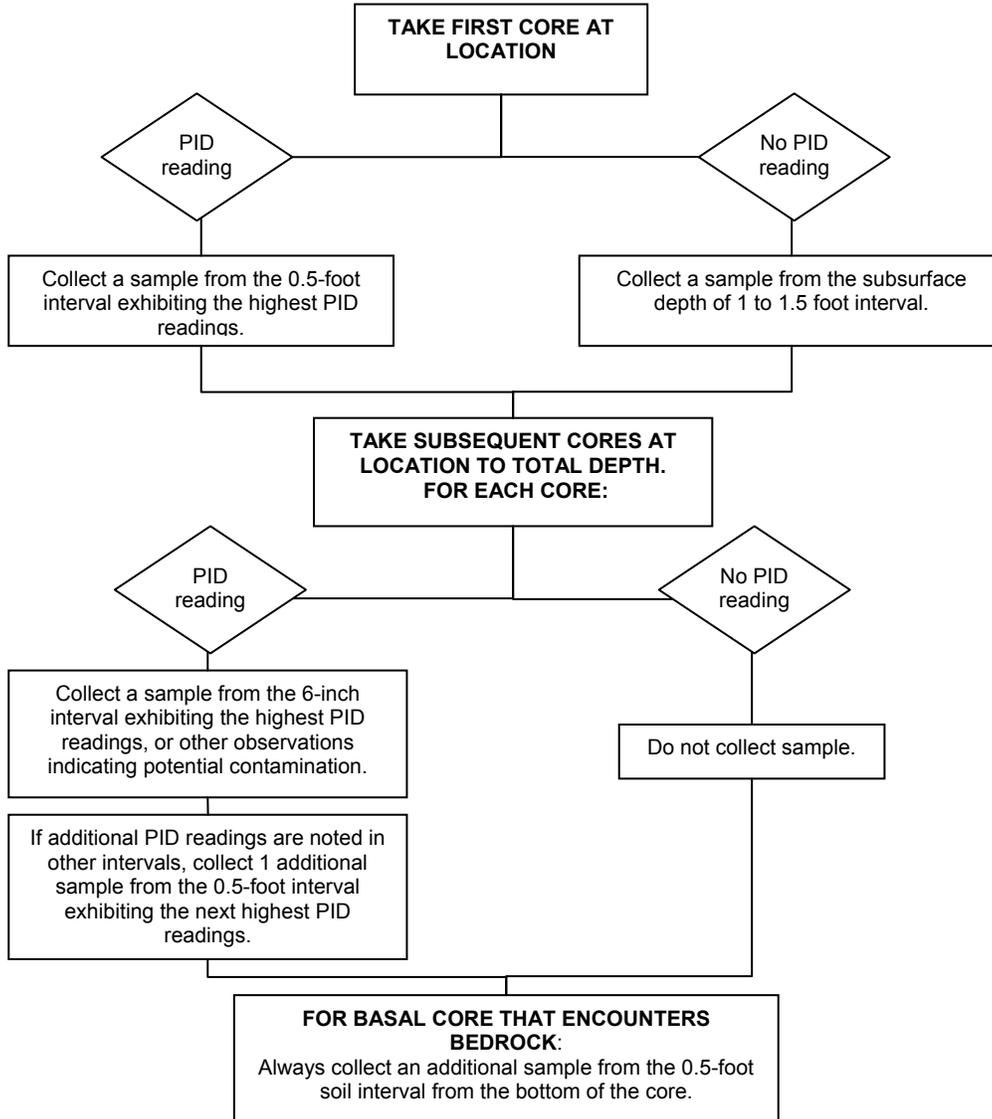
FILE FILE.DWG	SCALE AS NOTED
FIGURE NUMBER FIGURE 4-2	REV DATE 0 09/24/07



Notes
 ● Parameters to be analyzed for the sampling event shown.
 Event 1 is the pre-treatability study event and is to be performed early Fall 2007. Event 2 is the baseline event and will be a comprehensive round prior to system startup (anytime prior to startup).
 This figure only shows general events. Refer to the Work Plan for details.
 NAT = Natural attenuation parameters.

FIGURE 4-3
GENERAL SCHEDULE OF ACTIVITIES
PILOT STUDY WORK PLAN
SITE 5

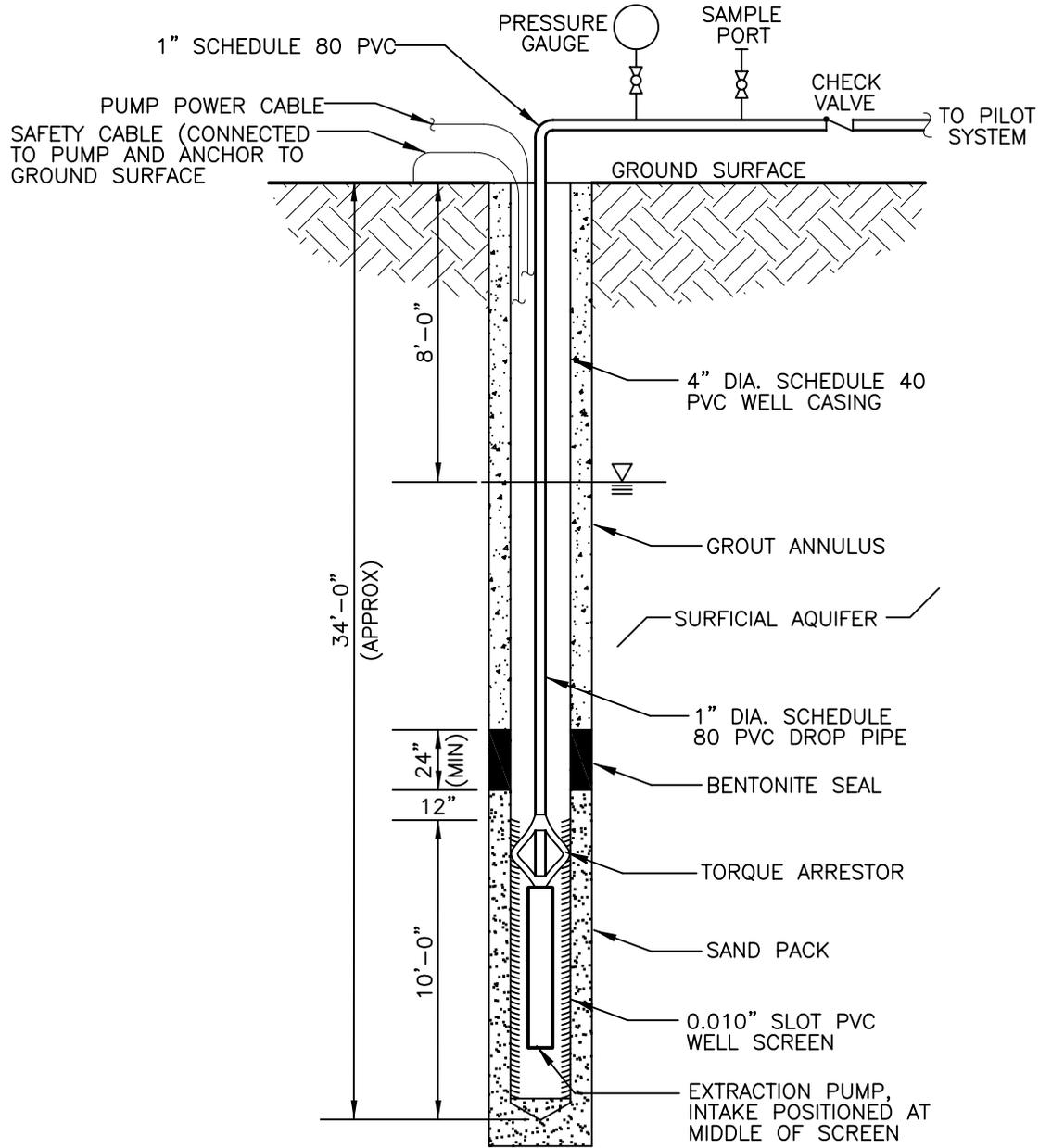
**FIGURE 4-4
DECISION MATRIX FOR SOIL SAMPLE SELECTION
SITE 5 - FIRE TRAINING AREA
NAS JRB WILLOW GROVE, PENNSYLVANIA**



SAMPLE SUBMISSION

2 SAMPLES PER BORING WILL BE SUBMITTED FOR LABORATORY ANALYSIS.

1. For borings where no PID readings were detected and no other observations indicating potential contamination were noted, submit the samples taken from the first core at the 1 to 1.5 foot depth interval and the last core at the basal 0.5-foot interval.
2. For borings where elevated PID readings were detected in only one core, submit the 2 samples representing the highest PID readings within that core. If only one elevated PID reading was detected, submit the sample taken from that interval and the sample taken from the deepest core at the basal 0.5-foot interval above the top of bedrock.
3. For borings where elevated PID readings were detected in multiple cores, submit the sample representing the highest PID readings and the sample representing the deepest (closest to bedrock) elevated PID reading.



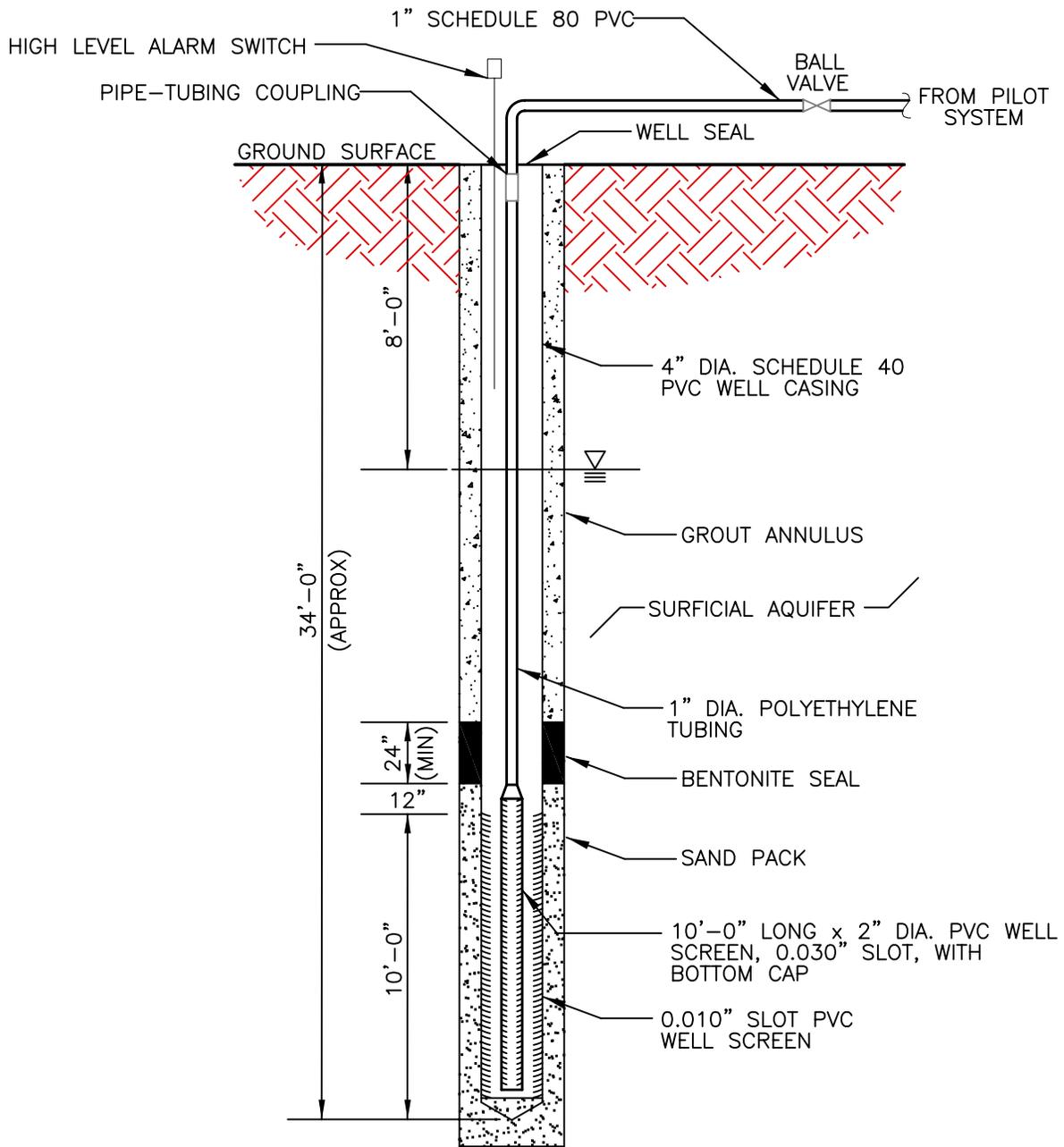
NOTE:
 WELLS ARE EXPECTED TO BE COMPLETED AS OPEN-BEDROCK BOREHOLES.
 RETROFITTING WELL AS ILLUSTRATED IS A CONTINGENCY FOR UNSTABLE BOREHOLES.



TETRA TECH NUS, INC.

EXTRACTION WELL DETAIL
 PILOT STUDY WORK PLAN
 SITE 5 – FIRE TRAINING AREA
 NAS JRB WILLOW GROVE
 WILLOW GROVE, PENNSYLVANIA

SCALE AS NOTED	
FILE 112G00910CD01	
REV 0	DATE 09/21/07
FIGURE NUMBER FIGURE 5-1	



NOTE:
 WELLS ARE EXPECTED TO BE COMPLETED AS OPEN-BEDROCK BOREHOLES.
 RETROFITTING WELL AS ILLUSTRATED IS A CONTINGENCY FOR UNSTABLE BOREHOLES.



TETRA TECH NUS, INC.

INJECTION WELL DETAIL
 PILOT STUDY WORK PLAN
 SITE 5 – FIRE TRAINING AREA
 NAS JRB WILLOW GROVE
 WILLOW GROVE, PENNSYLVANIA

SCALE AS NOTED	
FILE 112G00910CD02	
REV 0	DATE 09/24/07
FIGURE NUMBER FIGURE 5-2	

APPENDIX A

TTNUS STANDARD OPERATING PROCEDURES

- **CT-04 Sample Nomenclature**
- **DV-02 Data Validation – Non-CLP Organics for Solid Matrices**
- **DV-04 Data Validation - Non-CLP Inorganics for Solid and Aqueous Matrices**
- **DV-08 Data Validation - Miscellaneous Inorganics**
- **GH-1.2 Evaluation of Existing Monitoring Wells and Water Level Measurements**
- **GH-1.5 Borehole and Sample Logging**
- **GH-2.8 Groundwater Monitoring Well Installation**
- **HS-1.0 Utility Locating and Excavation Clearance**
- **SA-1.1 Groundwater Sample Acquisition and Onsite Water Quality Testing**
- **SA-1.3 Soil Sampling**
- **SA-1.6 Natural Attenuation Parameter Collection**
- **SA-2.5 Direct Push Technology (Geoprobe®/Hydropunch™)**
- **SA-6.1 Non-Radiological Sample Handling**
- **SA-6.3 Field Documentation**
- **SA-7.1 Decontamination of Field Equipment**



TETRA TECH NUS, INC.

STANDARD OPERATING PROCEDURES

Number	CT-04	Page	1 of 6
Effective Date	09/03	Revision	1
Applicability	Tetra Tech NUS, Inc.		
Prepared	Risk Assessment Department		
Approved	D. Senovich <i>ds</i>		

Subject
SAMPLE NOMENCLATURE

TABLE OF CONTENTS

<u>SECTION</u>	<u>PAGE</u>
1.0 PURPOSE.....	2
2.0 SCOPE.....	2
3.0 GLOSSARY	2
4.0 RESPONSIBILITIES.....	2
5.0 PROCEDURES.....	2
5.1 INTRODUCTION.....	2
5.2 SAMPLE IDENTIFICATION FIELD REQUIREMENTS.....	3
5.3 EXAMPLE SAMPLE FIELD DESIGNATIONS	4
5.4 EXAMPLES OF SAMPLE NOMENCLATURE	5
5.5 FIELD QUALITY ASSURANCE/QUALITY CONTROL (QA/QC) SAMPLE NOMENCLATURE).....	6
5.6 EXAMPLES OF FIELD QA/QC SAMPLE NOMENCLATURE	6
6.0 DEVIATIONS	6

Subject SAMPLE NOMENCLATURE	Number CT-04	Page 2 of 6
	Revision 1	Effective Date 09/03

1.0 PURPOSE

The purpose of this document is to specify a consistent sample nomenclature system that will facilitate subsequent data management in a cost-effective manner. The sample nomenclature system has been devised such that the following objectives can be attained:

- Sorting of data by matrix.
- Sorting of data by depth.
- Maintenance of consistency (field, laboratory, and data base sample numbers).
- Accommodation of all project-specific requirements.
- Accommodation of laboratory sample number length constraints (maximum of 20 characters).

2.0 SCOPE

The methods described in this procedure shall be used consistently for all projects requiring electronic data.

3.0 GLOSSARY

None.

4.0 RESPONSIBILITIES

Program Manager - It shall be the responsibility of the Program Manager (or designee) to inform contract-specific Project Managers of the existence and requirements of this Standard Operating Procedure.

Project Manager - It shall be the responsibility of the Project Manager to determine the applicability of this Standard Operating Procedure based on: (1) program-specific requirements, and (2) project size and objectives. It shall be the responsibility of the Project Manager (or designee) to ensure that the sample nomenclature is thoroughly specified in the relevant project planning document (e.g., sampling and analysis plan) and is consistent with this Standard Operating Procedure if relevant. It shall be the responsibility of the project manager to ensure that the Field Operations Leader is familiar with the sample nomenclature system.

Field Operations Leader - It shall be the responsibility of the Field Operations Leader to ensure that all field technicians or sampling personnel are thoroughly familiar with this Standard Operating Procedure and the project-specific sample nomenclature system. It shall be the responsibility of the Field Operations Leader to ensure that the sample nomenclature system is used during all project-specific sampling efforts.

5.0 PROCEDURES

5.1 Introduction

The sample identification (ID) system can consist of as few as 8 but not more than 20 distinct alphanumeric characters. The sample ID will be provided to the laboratory on the sample labels and chain-of-custody forms. The basic sample ID provided to the lab has three segments and shall be as follows where "A" indicates "alpha," and "N" indicates "numeric":

A or N 3- or 4-Characters	AAA 2- or 3-Characters	A or N 3- to 6-Characters
Site Identifier	Sample Type	Sample Location

Subject SAMPLE NOMENCLATURE	Number CT-04	Page 3 of 6
	Revision 1	Effective Date 09/03

Additional segments may be added as needed. For example:

(1) Soil and Sediment Sample ID

A or N 3- or 4-Characters	AAA 2- or 3-Characters	A or N 3- to 6-Characters	NNNN 4-Characters
Site Identifier	Sample Type	Sample Location	Sample Depth

(2) Aqueous (groundwater or surface water) Sample ID

A or N 3- or 4-Characters	AAA 2- or 3-Characters	A or N 3- to 6-Characters	NN 2-Characters	-A
Site Identifier	Sample type	Sample Location	Round Number	Filtered Sample only

(3) Biota Sample ID

A or N 3- or 4-Characters	AAA 2- or 3-Characters	A or N 3- to 6-Characters	AA 2-Characters	NNN 3-Characters
Site Identifier	Sample Type	Sample Location	Species Identifier	Sample Group Number

5.2 Sample Identification Field Requirements

The various fields in the sample ID will include but are not limited to the following:

- Site Identifier
- Sample Type
- Sample Location
- Sample Depth
- Sampling Round Number
- Filtered
- Species Identifier
- Sample Group Number

The site identifier must be a three- or four-character field (numeric characters, alpha characters, or a mixture of alpha and numeric characters may be used). A site number is necessary since many facilities/sites have multiple individual sites, SWMUs, operable units, etc. Several examples are presented in Section 5.3 of this SOP.

The sample type must be a two- or three-character alpha field. Suggested codes are provided in Section 5.3 of this SOP.

The sample location must be at least a three-character field but may have up to six-characters (alpha, numeric, or a mixture). The six-characters may be useful in identifying a monitoring well to be sampled or describing a grid location.

The sample depth field is used to note the depth below ground surface (bgs) at which a soil or sediment sample is collected. The first two numbers of the four-number code specify the top interval, and the third and fourth specify the bottom interval in feet bgs of the sample. If the sample depth is equal to or greater than 100, then only the top interval would be represented and the sampling depth would be truncated to

Subject SAMPLE NOMENCLATURE	Number CT-04	Page 4 of 6
	Revision 1	Effective Date 09/03

three-characters. The depths will be noted in whole numbers only; further detail, if needed, will be recorded on the sample log sheet, boring log, logbook, etc.

A two-digit round number will be used to track the number of aqueous samples taken from a particular aqueous sample location. The first sample collected from a location will be assigned the round identifier 01, the second 02, etc. This applies to both existing and proposed monitoring wells and surface water locations.

Aqueous samples that are field filtered (dissolved analysis) will be identified with an "-F" in the last field segment. No entry in this segment signifies an unfiltered (total) sample.

The species identifier must be a two-character alpha field. Several suggested codes are provided in Section 5.3 of this SOP.

The three digit sample group number will be used to track the number of biota sample groups (a particular group size may be determined by sample technique, media type, the number of individual caught, weight issues, time, etc.) by species and location. The first sample group of a particular species collected from a given location will be assigned the sample group number 001 and the second sample group of the same species collected from the same location will be assigned the sample group number 002.

5.3 Example Sample Field Designations

Examples of each of the fields are as follows:

Site Identifier - Examples of site numbers/designations are as follows:

- A01 - Area of Concern Number 1
- 125 - Solid Waste Management Unit Number 125
- 000 - Base or Facility Wide Sample (e.g., upgradient well)
- BBG - Base Background

The examples cited are only suggestions. Each Project Manager (or designee) must designate appropriate (and consistent) site designations for their individual project.

Sample Type - Examples of sample types are as follows:

- AH - Ash Sample
- AS - Air Sample
- BM - Building Material Sample
- BSB - Biota Sample Full Body
- BSF - Biota Sample Fillet
- CP - Composite Sample
- CS - Chip Sample
- DS - Drum Sample
- DU - Dust Sample
- FP - Free Product
- IDW - Investigation Derived Waste Sample
- LT - Leachate Sample
- MW - Monitoring Well Groundwater Sample
- OF - Outfall Sample
- RW - Residential Well Sample
- SB - Soil Boring Sample
- SD - Sediment Sample
- SC - Scrape Sample

Subject SAMPLE NOMENCLATURE	Number CT-04	Page 5 of 6
	Revision 1	Effective Date 09/03

- SG - Soil Gas Sample
- SL - Sludge Sample
- SP - Seep Sample
- SS - Surface Soil Sample
- ST - Storm Sewer Water Sample
- SW - Surface Water Sample
- TP - Test Pit Sample
- TW - Temporary Well Sample
- WC - Well Construction Material Sample
- WP - Wipe Sample
- WS - Waste/Solid Sample
- WW - Wastewater Sample

Sample Location - Examples of the location field are as follows:

- 001 - Monitoring Well 1
- N32E92 - Grid location 32 North and 92 East
- D096 - Investigation derived waste drum number 96

Species Identifier - Examples of species identifier are as follows:

- BC - Blue Crab
- GB - Blue Gill
- CO - Corn
- SB - Soybean

5.4 Examples of Sample Nomenclature

The first round monitoring well groundwater sample collected from existing monitoring well 001 at SWMU 16 for a filtered sample would be designated as 016MW00101-F.

The second round monitoring well groundwater sample collected from existing monitoring well C20P2 at Site 23 for an unfiltered sample would be designated as 023MWC20P202.

The second surface water sample collected from point 01 at SWMU 130 for an unfiltered sample would be designated as 130SW00102.

A surface soil sample collected from grid location 32 North and 92 East at Site 32 at the 0- to 2-foot interval would be designated as 032SSN32E920002.

A subsurface soil sample from soil boring 03 at SWMU 32 at an interval of 4 to 5 feet bgs would be designated as 032SB0030405.

A sediment sample collected at SWMU 19 from 0 to 6 inches at location 14 would be designated as 019SD0140001. The sample data sheet would reflect the precise depth at which this sample was collected.

During biota sampling for full body analysis the first time a minnow trap was checked at grid location A25 of SWMU 1415 three small blue gills were captured, collected and designated with the sample ID of 1415BSBA25BG001. The second time blue gill were collected at the same location (grid location A25 at SWMU 1415) the sample ID designation given was 1415BSBA25BG002.

Note: No dash (-) or spacing is used between the segments with the exception of the filtered segment. The "F" used for a filtered aqueous sample is preceded by a dash "-F".

Subject SAMPLE NOMENCLATURE	Number CT-04	Page 6 of 6
	Revision 1	Effective Date 09/03

5.5 Field Quality Assurance/Quality Control (QA/QC) Sample Nomenclature

Field QA/QC will be designated using a different coding system. The QC code will consist of a three- to four-segment alpha-numeric code that identifies the sample QC type, the date the sample was collected, and the number of this type of QC sample collected on that date.

AA	NNNNNN	NN	-F
QC Type	Date	Sequence Number (per day)	Filtered (aqueous only, if needed)

The QC types are identified as:

TB = Trip Blank
 RB = Rinsate Blank (Equipment Blank)
 FD = Field Duplicate
 AB = Ambient Conditions Blank
 WB = Source Water Blank

The sampling time recorded on the Chain-of-Custody Form, labels, and tags for duplicate samples will be 0000 so that the samples are "blind" to the laboratory. Notes detailing the sample number, time, date, and type will be recorded on the routine sample log sheets and will document the location of the duplicate sample (sample log sheets are not provided to the laboratory). Documentation for all other QC types (TB, RB, AB, and WB) will be recorded on the QC Sample Log sheet (see SOP on Field Documentation).

5.6 Examples of Field QA/QC Sample Nomenclature

The first duplicate of the day for a filtered ground water sample collected on June 3, 2000 would be designated as FD06030001-F.

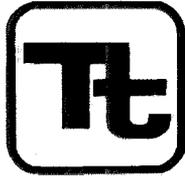
The third duplicate of the day taken of a subsurface soil sample collected on November 17, 2003 would be designated as FD11170303.

The first trip blank associated with samples collected on October 12, 2000 would be designated as TB10120001.

The only rinsate blank collected on November 17, 2001 would be designated as RB11170101.

6.0 **DEVIATIONS**

Any deviation from this SOP must be addressed in detail in the site specific planning documents.



TETRA TECH NUS, INC.

STANDARD OPERATING PROCEDURES

Number	DV-02	Page	1 of 32
Effective Date	08/13/01	Revision	0
Applicability	Tetra Tech NUS, Inc.		
Prepared	Risk Assessment Department		
Approved	D. Senovich <i>DS</i>		

Subject
DATA VALIDATION - NON-CLP ORGANICS FOR SOLID MATRICES

TABLE OF CONTENTS

<u>SECTION</u>	<u>PAGE</u>
1.0 SW-846 ORGANICS BY GC/MS.....	3
1.1 VOLATILES (METHOD 8260B).....	3
1.1.1 Applicability.....	3
1.1.2 Interferences.....	3
1.1.3 General Laboratory Practices.....	3
1.1.4 Sample Preparation.....	4
1.1.5 Data Overview Prior to Validation.....	4
1.1.6 Technical Evaluation Summary.....	4
1.1.7 Deliverables Guidance.....	7
1.2 SEMIVOLATILES (METHOD SW8250A, 8270C).....	7
1.2.1 Applicability.....	7
1.2.2 Interferences.....	8
1.2.3 General Laboratory Practices.....	8
1.2.4 Sample Preparation.....	8
1.2.5 Data Overview to Validation.....	9
1.2.6 Technical Evaluation Summary.....	9
1.2.7 Deliverables Guidance.....	12
2.0 SW846 NON-CLP ORGANICS BY GAS CHROMATOGRAPHY.....	12
2.1 VOLATILES (SW 5030/SW 8011/8015B/8021A/8031).....	12
2.1.1 Applicability.....	12
2.1.2 Interferences.....	14
2.1.3 General Laboratory Practices.....	14
2.1.4 Sample Preparation.....	14
2.1.5 Data Overview Prior to Validation.....	15
2.1.6 Technical Evaluation Summary.....	15
2.1.7 Deliverables Guidance.....	17
2.2 SEMIVOLATILES (SW8041/8061A/8091/8310).....	18
2.2.1 Applicability.....	18
2.2.2 Interferences.....	19
2.2.3 General Laboratory Practices.....	19
2.2.4 Sample Preparation.....	19
2.2.5 Data Overview Prior to Validation.....	20
2.2.6 Technical Evaluation Summary.....	20
2.2.7 Deliverables Guidance.....	22
2.3 ORGANOCHLORINE PESTICIDES AND POLYCHLORINATED BIPHENYLS (PCBS), ORGANOPHOSPHOROUS PESTICIDES, CHLORINATED HERBICIDES (SW 8081A/8082/8141A/ 8151A).....	23
2.3.1 Applicability.....	23
2.3.2 Interferences.....	24

Subject DATA VALIDATION - NON-CLP ORGANICS FOR SOLID MATRICES	Number DV-02	Page 2 of 32
	Revision 0	Effective Date 08/13/01

TABLE OF CONTENTS (Continued)

<u>SECTION</u>	<u>PAGE</u>
2.3.3 General Laboratory Practices	25
2.3.4 Sample Preparation	25
2.3.5 Data Overview Prior to Validation	25
2.3.6 Technical Evaluation Summary	25
2.3.7 Deliverables Guidance.....	28
2.4 EXPLOSIVES/NITROAROMATICS/NITROAMINES(SW 8330)	28
2.4.1 Applicability	28
2.4.2 Interferences.....	29
2.4.3 General Laboratory Practices	29
2.4.4 Sample Preparation	29
2.4.5 Data Overview Prior to Validation	29
2.4.6 Technical Evaluation Summary	30

Subject DATA VALIDATION - NON-CLP ORGANICS FOR SOLID MATRICES	Number DV-02	Page 3 of 32
	Revision 0	Effective Date 08/13/01

1.0 SW-846 ORGANICS BY GC/MS

1.1 Volatiles (Method 8260B)

1.1.1 Applicability

Method 8260B is used to determine volatile organic compounds in most waste matrices including groundwater, sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments.

Method 8260B analyte list includes of the volatile CLP 3/90 Target Compound List (TCL) (Section 1.1.1) plus the following compounds*:

Acetonitrile	trans-1,2-Dichloroethene
Acrolein	Ethyl methacrylate
Acrylonitrile	Iodomethane
Allyl chloride	Methacrylonitrile
Chloropropene	Methyl methacrylate
1,2-Dibromo-3-chloropropane	2-Picoline
1,2-Dibromoethane	Pyridine
Dibromomethane	Trichlorofluoromethane
trans-1,4-Dichloro-2-butene	1,2,3-Trichloropropane
Dichlorodifluoromethane	Vinyl acetate

* Appendix IX target compounds

Method 8260B is based upon a purge-and-trap, gas chromatographic/mass spectrometric (GC/MS) procedure. Prior to analysis, samples must be prepared by Method 5030.

1.1.2 Interferences

Samples can be contaminated by diffusion of volatile organics (particularly chlorofluorocarbons and methylene chloride) through the sample container septum during shipment and storage. Associated field quality control blanks are analyzed in order to monitor this.

Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe or purging device is rinsed out between samples with reagent water. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of reagent water to check for cross contamination.

If sample or matrix interferences are encountered, a secondary or alternate analytical column may be used to resolve the compounds of interest.

1.1.3 General Laboratory Practices

A method blank consisting of organic free water spiked with surrogates and internal standards should be analyzed immediately following each daily calibration and also after the analysis of every high concentration sample.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) analyses should be conducted to determine the effects of sample matrix upon the compounds of interest.

Subject DATA VALIDATION - NON-CLP ORGANICS FOR SOLID MATRICES	Number DV-02	Page 4 of 32
	Revision 0	Effective Date 08/13/01

1.1.4 Sample Preparation

Method 5030 is a purge-and-trap procedure performed to prepare and extract volatile compounds from samples and introduce those compounds into the GC/MS.

For highly volatile matrices, direct injection preceded by dilution should be used to prevent gross contamination of the instrumentation. For pastes, dilution of the sample until it becomes free-flowing is used to ensure adequate interfacial area. The success of this method depends on the level of interferences in the sample; results may vary due to the large variability and complicated matrices of solid waste samples.

1.1.5 Data Overview Prior to Validation

Before commencing validation, the reviewer must preview the associated Chain-of-Custody (COC) reports to determine:

- If the appropriate number of samples are present in the data package and if each sample was correctly analyzed for the parameters and methods specified.
- The identity of all associated field quality control blanks and field duplicate pairs.

Because many samples may have required dilutions, re-extractions and/or re-analyses, the validator should preview the data package contents to determine which analyses represent the better quality data.

Unless specifically directed by client protocol, never annotate the laboratory data package. Before beginning evaluation, prepare working copies (i.e., photocopies) of all Form I reports (including those for samples, laboratory method blanks and MS/MSD analyses) and all laboratory quality control summary forms (including all initial and continuing calibration summary statistics).

1.1.6 Technical Evaluation Summary

All data evaluations must be conducted in accordance with applicable USEPA Regional protocols and/or specific client contract requirements. The applicable documents must be referenced during the data evaluation process as this S.O.P. is only intended as a general procedure for the data validation tasks.

General parameters such as Data Completeness, Overall System Performance, Chromatographic Quality, Detection Limits and Compound Identification are evaluated concurrently with the parameters discussed in the following subsections.

1.1.6.1 Holding Times

Holding times are evaluated by reviewing the COC reports, the individual sample Form I reports, and the associated laboratory raw data. Holding times are calculated from date of collection to date of analysis.

The technical maximum holding time allowance for aqueous samples preserved with hydrochloric acid (HCL) is 14 days.

No technical holding times for solid matrices have been promulgated; a 14-day maximum holding time allowance is currently being used.

For unpreserved aqueous samples, generally a 7-day maximum holding time allowance for aromatic compounds, along with a 14-day maximum holding time allowance for chlorinated hydrocarbons is used.

Subject DATA VALIDATION - NON-CLP ORGANICS FOR SOLID MATRICES	Number DV-02	Page 5 of 32
	Revision 0	Effective Date 08/13/01

Positive results in affected samples are generally qualified as estimated (J); nondetects (UJ). These results are biased low. Some USEPA Regions apply the bias qualifiers, L and UL, instead. If the holding times are exceeded by a factor of 2 or more, the holding time exceedance is considered to be gross and positive results are generally qualified as estimated (J); nondetects are generally considered to be unreliable and are qualified (R). Results for which the holding time was grossly exceeded are biased low.

1.1.6.2 Calibration

Check that an initial calibration was performed for each instrument used for analysis and that all calibrations were performed at all appropriate concentration levels within 12 hours of the associated instrument tuning.

Review the data package Form Vs (tuning) using the applicable USEPA Regional Functional Guidelines, and qualify the data as appropriate.

Review initial calibration Form VIs and the associated laboratory raw data. Determine which compounds have average Relative Response Factors (RRFs) <0.050 and which compounds have Percent Relative Standard Deviations (%RSDs) >50% and between 30% and 50%. Circle these noncompliances on your working copies of these Forms. Spot-check (i.e., recalculate) a few of the RRFs and %RSDs to verify the laboratory's computation.

Determine which samples are affected by reviewing the continuing calibration Form VIIs. Check the initial calibration date(s) noted in the headings of the Form VIIs to determine which continuing calibrations are associated with which initial calibrations. Next, review the sample listings given on the data package Form Vs. Match the indicated continuing calibration run with the appropriate Form VII by matching the laboratory file ID numbers. Write the affected samples (those listed on the matched Form V) on your working copies of the appropriate Form VI and VII. Spot-check (i.e., recalculate) a few of the RRFs and %Ds to verify the laboratory's computation.

Review the continuing calibration Form VIIs and the associated laboratory raw data. Determine which compounds have RRFs <0.050 and which compounds have Percent Differences (%Ds) >25%; circle the noncompliances on your working copies of these Forms.

Generally, affected positive results for compounds whose RRFs are <0.050 are qualified as estimated (J); nondetects are rejected (R). In accordance with some USEPA Regional protocol, the (L) qualifier may be used instead of (J), when qualifying positive results. Bias for these results is low.

Generally, positive results for compounds for which %RSD exceeds 50% or %D exceeds 25% are qualified as estimated (J); nondetects (UJ). Check the specific applicable data validation protocol for further guidance as there are some protocol which reject nondetects if the %RSD or %D is excessive. Bias for these results cannot be determined.

Generally, positive results for compounds for which %RSD is between 30%-50% are qualified as estimated (J). Qualification of nondetects is protocol-specific. Follow the rules given in the appropriate validation protocol.

1.1.6.3 Blank Contamination

When using the information given below and in the appropriate USEPA Regional Functional Guidelines, keep in mind that the validation action levels derived are sample-specific and must be adjusted for dilution, sample aliquot used for analysis, and sample moisture content (when applicable).

Subject DATA VALIDATION - NON-CLP ORGANICS FOR SOLID MATRICES	Number DV-02	Page 6 of 32
	Revision 0	Effective Date 08/13/01

The rules for qualifying data based on the occurrence of blank contamination vary based on regional protocols. The guidelines provided in the appropriate protocol should be followed.

Generally the blank contamination review process is completed by first considering the maximum amount of a particular contaminant occurring in the laboratory method blanks. (Do not consider lab blanks run after high concentration samples for purposes of determining carryover as laboratory method blanks!) Then repeat the process for contaminants occurring in the associated field quality control blanks. Action levels for qualification (10X or 5X depending upon whether or not the contaminant is a common contaminant) are then set. The list of common contaminants may vary among protocols. Additionally, some hierarchy among the field quality control blanks apply, and the manner in which the qualifiers are applied vary [i.e. use of (U) or (B); replacement by CRQL, etc.]. Refer to appropriate protocol for specific guidance.

1.1.6.4 Surrogates

Surrogates are evaluated by reviewing the laboratory data package Form II reports and the laboratory raw data. The quality control ranges are given on the laboratory data package Form IIs; circle any noncompliances on your working copies of these Forms.

Results for all compounds in an affected sample are qualified if any one of the surrogate spike compounds fail to meet the quality control criteria provided. Generally, for samples having a surrogate recovery <10%, positive results are qualified as estimated (J), nondetects are rejected (R). These results are biased low. For samples having a surrogate recovery which is low but >10%, positive results are generally qualified as estimated (J); nondetects (UJ). The bias qualifiers (L, UL) may be used instead, depending upon the specific USEPA Regional guidance. For samples having a surrogate recovery which is high, positive results are generally qualified as estimated (J, K) based on regional guidance, nondetects are not qualified based on high surrogate recovery.

1.1.6.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

Generally, no data are qualified based upon MS/MSD results alone. If qualification does occur, generally only the result for that particular noncompliant compound is qualified in the original unspiked sample. Refer to the applicable data validation protocol for specific procedures for appropriately evaluating MS/MSD analyses.

1.1.6.6 Internal Standards

Internal standards are evaluated by reviewing the data package Form VIIIs and the laboratory raw data. The quality control ranges are given on the Form VIIIs. Circle any noncompliances on your working copies of these forms; evaluate and qualify as stipulated in the appropriate data validation protocol.

1.1.6.7 Tentatively Identified Compounds (TICs)

TICs are evaluated using the laboratory data package Form I VOA-TIC reports and the laboratory raw data. The guidance given in the March 1990 National Functional Guidelines for USEPA Region III is very concise; use the information in this document to evaluate and qualify accordingly.

1.1.6.8 Other Considerations

Laboratory precision can be evaluated by comparing the unspiked sample results with MS/MSD analyses results for unspiked compounds. Consider nondetects and results reported at concentrations less than

Subject DATA VALIDATION - NON-CLP ORGANICS FOR SOLID MATRICES	Number DV-02	Page 7 of 32
	Revision 0	Effective Date 08/13/01

the Contract Required Quantitation Limit (CRQL) to be in agreement. Use professional judgment in determining whether to qualify sample results based on the comparison.

Likewise, compare the positive compound results for field duplicate samples. Generally, the Relative Percent Difference (RPD) between field duplicate results for the aqueous matrix should be <35%; for soil matrix results, <50%. Qualification of the sample data is limited to the specific field duplicate pair. Positive results for compounds showing imprecision are qualified as estimated (J); nondetects (UJ). Bias for these results cannot be determined.

In some USEPA Regions, a "Percent Solids" rule applies. For example, if a sediment sample contains <50% solids in USEPA Region II, all associated data are considered to be estimated and are qualified accordingly. Follow the appropriate protocol guidance when applicable.

1.1.6.9 Quantitation

Verify and record the quantitation of at least one compound per analytical fraction. If no positive results are reported, use the MS/MSD data to confirm proper computation by the laboratory. Validator and laboratory quantitations must agree within 10 percent.

1.1.7 **Deliverables Guidance**

In addition to any specific USEPA Regional requirements (e.g. data validation memorandum, data summary spreadsheets, Regional worksheets), all laboratory data package quality control summary forms, sample Form I reports, method blank Form Is, and the Chain-of-Custody report must be given to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct (in accordance with the appropriate USEPA Regional or client requirements) and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

1.2 **Semivolatiles (Method SW8250A, 8270C)**

1.2.1 **Applicability**

Methods are applicable to most types of samples, regardless of water content, including groundwater, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments.

These methods can be used to quantify most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of elution without derivatization as sharp peaks from a gas chromatographic column. Such compounds include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols, including nitrophenols.

Subject DATA VALIDATION - NON-CLP ORGANICS FOR SOLID MATRICES	Number DV-02	Page 8 of 32
	Revision 0	Effective Date 08/13/01

The above methods specifically analyze for the semivolatile Target Compound List (TCL) (Section 1.1.2) plus the following compounds*:

Acetophenone	Hexachlorophene	N-nitrosodimethylethylamine
Aniline	Hexachloropropene	N-nitroso-di-n-butylamine
Benzyl alcohol	Isodrin	N-nitrosomorpholine
Bis(2-chloroisopropyl)ether	Isosafrole	N-nitrosopiperidine
Chlorobenzilate	Kepone	Pentachlorobenzene
Diallate	Methapyrilene	Pentachloronitrobenzene
2,6-Dichlorophenol	3-Methylcholanthrene	Phenacetin
Dimethoate	Methyl methanesulfonate	p-Phenylenediamine
p-Dimethylaminoazobenzene	3-Methylphenol	Phorate
7,12-Dimethylbenz(a)anthracene	1,4-Naphthoquinone	2-Picoline
3,3'-Dimethylbenzidine	4-Nitroquinoline-1-oxide	Pronamide
a,a-Dimethylphenylamine	1-Naphthylamine	Safrole
1,3-Dinitrobenzene	2-Naphthylamine	1,2,4,5-Tetrachlorobenzene
Diphenylamine	5-Nitro-o-toluidine	Thionazin
Ethyl methanesulfonate	N-nitrosodiethylamine	o,o,o-Triethylphosphorothioate
Famphur	N-nitrosodimethylamine	1,3,5-Trinitrobenzene

* Appendix IX target compounds

The preceding methods are based upon solvent extractions followed by gas chromatographic/mass spectrometric (GC/MS) procedures, Method 8270C uses GC/MS capillary column technique.

1.2.2 Interferences

Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines causing misinterpretation of chromatograms. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by running method blanks. The use of high purity reagents and solvents helps to minimize interference problems; purification of solvents by distillation in all-glass systems may be required.

Interferences co-extracted from the samples will vary considerably from source to source, depending upon the diversity of the industrial complex or waste being sampled.

1.2.3 General Laboratory Practices

An extraction blank should be prepared with each batch of samples extracted.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) analyses should be conducted to determine the effects of sample matrix upon the compounds of interest.

1.2.4 Sample Preparation

Prior to analysis, the samples must be extracted using the appropriate techniques. Aqueous samples are extracted at the appropriate pH with methylene chloride as a solvent using a separatory funnel (Method 3510) or a continuous liquid-liquid extractor (Method 3520). Both neat and diluted organic liquids may be analyzed by direct injection. Solid samples are extracted at the appropriate pH with methylene chloride using either Soxhlet Extraction (Method 3540) or sonication (Method 3550) procedures.

Subject DATA VALIDATION - NON-CLP ORGANICS FOR SOLID MATRICES	Number DV-02	Page 9 of 32
	Revision 0	Effective Date 08/13/01

1.2.5 Data Overview to Validation

Before commencing validation, the reviewer must preview the associated Chain-of-Custody (COC) reports to determine:

- If the appropriate number of samples are present in the data package and if each sample was correctly analyzed for the parameters and methods specified.
- The identity of all associated field quality control blanks and field duplicate pairs.

Because many samples may have required dilutions, re-extraction and/or reanalyses, the data validator should preview the data package contents to determine which analyses represent the better quality data.

The data package should never be annotated unless specifically directed by client protocol. All Form I reports (including those for samples, laboratory method blanks, and MS/MSD analyses) and all laboratory quality control summary forms (including all initial and continuing calibration summary statistics) should be photocopied for use as working copies.

1.2.6 Technical Evaluation Summary

All data evaluations must be conducted in accordance with the appropriate USEPA Regional protocols and/or specified client contract requirements. The applicable documents must be referenced during the data validation process as this S.O.P. is only intended as a general procedure for all data validation tasks.

General parameters such as Data Completeness, Overall System Performance, Chromatographic Quality, Detection Limits and Compound Identification are evaluated concurrently with the parameters discussed in the following subsections.

1.2.6.1 Holding Times

Holding times are evaluated by reviewing the COC reports, the individual sample Form I reports, and the associated laboratory raw data. Holding times for extraction are calculated from date of collection to date of extraction.

The technical holding times for aqueous and solid matrices are as follows:

- Extraction:
 - Water samples: 7 days
 - Solid samples: 14 days
- Analysis: 40 days from date of extraction

Affected positive results are generally qualified as estimated (J), nondetects (UJ). Alternately, the L or UL bias qualifiers may be used dependent upon the applicable USEPA Regional Guidance. If the sample was extracted beyond 14 days from collection (28 days for solid samples), the holding time exceedance is considered to be gross and positive results are qualified as estimated (J) or (L); nondetects are rejected (R). Generally, if the holding time until extraction is exceeded, the affected sample results are considered to be biased low. If the holding time until analysis has been exceeded (and potentially, some of the extract may have evaporated), the affected sample results may be considered to be biased high. Follow the qualification guidance given in the appropriate data validation protocol.

Subject DATA VALIDATION - NON-CLP ORGANICS FOR SOLID MATRICES	Number DV-02	Page 10 of 32
	Revision 0	Effective Date 08/13/01

1.2.6.2 Calibration

Check that an initial calibration was performed for each instrument used for analysis and that all calibrations were performed at all appropriate concentration levels within 12 hours of the associated instrument tuning.

Review the data package Form Vs (tuning) using the applicable USEPA Regional Functional Guidelines, and qualify the data as appropriate.

Review initial calibration Form VIs and the associated laboratory raw data. Determine which compounds have average Relative Response Factors (RRFs) <0.050 and which compounds have Percent Relative Standard Deviations (%RSDs) >50% and between 30% and 50%. Circle these noncompliances on your working copies of these Forms. Spot-check (i.e., recalculate) a few of the RRFs and %RSDs to verify the laboratory's computation.

Determine which samples are affected by reviewing the continuing calibration Form VIIs. Check the initial calibration date(s) noted in the headings of the Form VIIs to determine which continuing calibrations are associated with which initial calibrations. Next, review the sample listings given on the data package Form Vs. Match the indicated continuing calibration run with the appropriate Form VII by matching the laboratory file ID numbers. Write the affected samples (those listed on the matched Form V) on your working copies of the appropriate Form VI and VII. Spot-check (i.e., recalculate) a few of the RRFs and %Ds to verify the laboratory's computation.

Review the continuing calibration Form VIIs, and the associated laboratory raw data. Determine which compounds have RRFs <0.050 and which compounds have Percent Differences (%Ds) >30%; circle the noncompliances on your working copies of these Forms.

Generally, affected positive results for compounds for which RRFs are <0.050 are qualified as estimated (J); nondetects are rejected (R). In accordance with some USEPA Regional protocol, the (L) qualifier may be used instead of (J) when qualifying positive results. Bias for these results is low.

Generally, positive results for compounds for which %RSD exceeds 50% or %D exceeds 30%, are qualified as estimated (J); nondetects (UJ). Check the specific applicable data validation protocol for further guidance as there are some protocol which reject nondetects if the %RSD or %D is excessive. Bias for these results cannot be determined.

Generally, positive results for compounds for which %RSD is between 30%-50% are qualified as estimated (J). Qualification of nondetects is protocol-specific. Follow the rules given in the appropriate validation protocol.

1.2.6.3 Blank Contamination

Note that unlike VOA fraction analyses, a laboratory method blank does not have to be analyzed after every continuing calibration standard. Be very sure, however, that one semivolatile method blank was extracted for each day that associated samples were extracted (with a maximum of 20 samples per batch).

The action levels for qualification are 10X the maximum amount of phthalates found in the blanks (phthalates are common contaminants) and 5X the maximum amount of other contaminants found in the blanks. The actual action level applied is sample-specific and must be adjusted for dilution, sample aliquot used for analysis, and moisture content. The type and manner in which the qualifiers are applied vary with protocol [i.e., use of (U) or (B); replacement by CRQL, etc.]. Refer to appropriate data validation protocol for specific guidance.

Subject DATA VALIDATION - NON-CLP ORGANICS FOR SOLID MATRICES	Number DV-02	Page 11 of 32
	Revision 0	Effective Date 08/13/01

1.2.6.4 Surrogates

Surrogates are evaluated by reviewing the laboratory data package Form II reports and the associated laboratory raw data. The quality control ranges are given on the laboratory data package Form IIs; circle any noncompliances on your working copies of these Forms.

Semivolatile compounds are divided into two classes, base-neutral compounds and acid-extractable compounds. Each class of compounds has its own associated surrogates. If the recovery is <10% for any one surrogate, positive results for all compounds in that class in the affected sample are qualified as estimated, (J) or (L), and nondetects are rejected, (R). These results are biased low.

No qualification actions are taken for samples having any one surrogate recovery which is noncompliant but >10%.

If the recoveries for any two surrogates of the same class are noncompliant but above 10%, all sample results for that class of compounds in the affected sample are qualified. If the recoveries are low, positive results are generally qualified as estimated (J); nondetects (UJ). In some Regions, the bias qualifiers, L and UL, may be used instead. If the recoveries for any two surrogates of the same class are high, positive results for all compounds in that class in the affected sample are qualified, J or K, depending upon the appropriate USEPA Regional guidance; nondetects are not qualified based on high surrogate recoveries.

1.2.6.5 Matrix Spike/Matrix Spike Duplicates

Generally, no data are qualified based upon MS/MSD results alone. If qualification does occur, generally only the result for that particular noncompliant compound is qualified in the original unspiked sample analysis. Refer to the appropriate validation guidelines for specific procedures for evaluating MS/MSD analyses.

1.2.6.6 Internal Standards

Internal standards are evaluated by reviewing the data package Form VIIIs and the laboratory raw data. The quality control ranges are given on the Form VIIIs. Circle any noncompliances on your working copies of these forms; evaluate and qualify as stipulated in the appropriate protocol.

1.2.6.7 Tentatively Identified Compounds (TICs)

TICs are evaluated using the laboratory data package Form I BNA-TIC reports and the laboratory raw data. The guidance given in the 3/90 National Functional Guidelines for USEPA Region III is very concise; evaluate and qualify accordingly.

1.2.6.8 Other Considerations

Laboratory precision can be evaluated by comparing MS/MSD sample results for unspiked compounds with the unspiked sample results. Consider nondetects and results reported at concentration levels less than the Contract Required Quantitation Limit (CRQL) to be in agreement. Use professional judgment in determining whether to qualify sample results based on the comparison.

Likewise, compare the positive compound results for field duplicate samples. Generally the Relative Percent Difference (RPD) between field duplicate results for the aqueous matrix should be less than 35%; for soil matrix results, less than 50%. Qualification of sample data is limited to that specific field duplicate pair. Positive results for compounds showing imprecision are qualified as estimated (J); and nondetects (UJ). Bias for these results cannot be determined.

Subject DATA VALIDATION - NON-CLP ORGANICS FOR SOLID MATRICES	Number DV-02	Page 12 of 32
	Revision 0	Effective Date 08/13/01

In some USEPA regions a "Percent Solids" rule applies. For example, if a sediment contains less than 50% solids in USEPA Region II, all associated data are considered to be estimated and are qualified accordingly. Follow the appropriate protocol guidance when applicable.

1.2.6.9 Quantitation

Verify and record quantitation of at least one compound per analytical fraction. If no positive results are reported, use the MS/MSD data to confirm proper computation by the laboratory. Validator and laboratory quantitations must agree within 10 percent.

1.2.7 Deliverables Guidance

In addition to any specific USEPA Regional requirements (e.g., data validation memorandum, data summary spreadsheets, USEPA Regional worksheets), all laboratory data package quality control summary forms, sample Form I reports, method blank Form Is, and the Chain-of-Custody report, must be given to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct (in accordance with the appropriate USEPA Regional or client requirements) and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

2.0 SW846 NON-CLP ORGANICS BY GAS CHROMATOGRAPHY

2.1 Volatiles (SW 5030/SW 8011/8015B/8021A/8031)

2.1.1 Applicability

Method 8011 is used to determine the concentration of the following halogenated volatile organic compounds in groundwater, liquid, and solid matrices:

- 1,2-Dibromoethane (EDB)
- 1,2-Dibromo-3-chloropropane (DBCP)

Method 8021A is used to determine the concentration of the following halogenated volatile organic compounds in groundwater, liquid, and solid matrices:

- Allyl chloride
- Benzyl chloride
- Bis (2-chloroethoxy)methane
- Bis (2-chloroisopropyl)ether
- Bromoacetone
- Bromobenzene
- Bromodichloromethane
- Bromoform
- Bromomethane
- Carbon tetrachloride
- Chlorobenzene
- Chloroethane
- 2-Chloroethanol
- Chloroform
- 1-Chlorohexane

Subject DATA VALIDATION - NON-CLP ORGANICS FOR SOLID MATRICES	Number DV-02	Page 13 of 32
	Revision 0	Effective Date 08/13/01

2-Chloroethyl vinyl ether
 Chloromethane
 Chloromethyl methyl ether
 Chloroprene
 4-Chlorotoluene
 Dibromochloromethane
 1,2-Dibromo-3-chloropropane
 Dibromomethane
 1,2-Dichlorobenzene
 1,3-Dichlorobenzene
 1,4-Dichlorobenzene
 Dichlorodifluoromethane
 1,1-Dichloroethane
 1,2-Dichloroethane
 1,1-Dichloroethylene (Vinylidene chloride)
 trans-1,2-Dichloroethylene
 Dichloromethane
 1,2-Dichloropropane
 1,3-Dichloro-2-propanol
 cis-1,3-Dichloropropene
 trans-1,3-Dichloropropene
 Epichlorhydrin
 Ethylene dibromide
 Methyl iodide
 1,1,2,2-Tetrachloroethane
 1,1,1,2-Tetrachloroethane
 Tetrachloroethylene
 1,1,1-Trichloroethane
 1,1,2-Trichloroethane
 Trichloroethylene
 Trichlorofluoromethane
 1,2,3-Trichloropropane
 Vinyl chloride
 Benzene
 Chlorobenzene
 1,2-Dichlorobenzene
 1,3-Dichlorobenzene
 1,4-Dichlorobenzene
 Toluene
 Ethyl benzene
 Xylenes (Dimethyl benzenes)

Method 8015B is used to determine the concentration of the following nonhalogenated volatile organic compounds in groundwater, liquid, and solid matrices:

Diethyl ether	Acrolein	n-butyl Alcohol
Ethanol	Acetonitrile	t-butyl Alcohol
Methyl ethyl ketone (MEK)	Acetone	Methanol
Methyl isobutyl ketone (MIBK)	Allyl Alcohol	1,4-Dioxane

Method 8031 is used to determine the concentration of the following volatile organic compound in groundwater, liquid, and solid matrices:

Acrylonitrile

Subject DATA VALIDATION - NON-CLP ORGANICS FOR SOLID MATRICES	Number DV-02	Page 14 of 32
	Revision 0	Effective Date 08/13/01

All of the above Methods are gas chromatographic (GC) only (i.e., no mass spectrometer detector is employed). Method 8021A analyzes for halogenated and aromatic volatile organics via GC/HECP and GC/PID (Electro Conductivity Detector and Photoionization detector), Method 8015B analyzes for nonhalogenated volatile organics via GC/FID (Flame Ionization Detector), and Method 8031 analyzes for the compounds acrylonitrile using GC/FID. Samples can be analyzed by these methods using direct injection, the headspace method (Method 5021) or the purge-and-trap method (Method 5030B and 5035). Groundwater samples should be determined using Method 5030B.

2.1.2 Interferences

Samples can be contaminated by diffusion of volatile organics (particularly chlorofluorocarbons and methylene chloride) through the sample container septum during shipment and storage. Associated field quality control blanks are analyzed in order to monitor this.

Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe or purging device is rinsed with reagent water between samples. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of reagent water to check for cross contamination.

If sample or matrix interferences are encountered, a secondary or alternate analytical column may be used to resolve the compounds of interest.

2.1.3 General Laboratory Practices

A method blank consisting of organic free water spiked with surrogates and internal standards should be analyzed immediately following each daily calibration, and also after the analysis of every high concentration sample.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) analyses should be conducted to determine the effects of sample matrix upon the compounds of interest.

2.1.4 Sample Preparation

Method 5020 is a static headspace technique for extracting volatile organic compounds in pastes, solids, and liquids. Because of the large variability and complicated matrices of waste samples detection limits for this method may vary widely among samples.

Method 5030 is a purge-and-trap method applicable to nearly all types of samples, regardless of water content, including aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, groundwater, mounds, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments.

Method 5035 is a purge-and-trap method applicable to nearly all types of soil samples, regardless of water content, including oily wastes, soils, and sediments.

For highly volatile matrices, direct injection preceded by dilution should be used to prevent gross contamination of the instrumentation. For pastes, dilution of the sample until it becomes free-flowing is used to ensure adequate interfacial area. The success of this method depends on the level of interferences in the sample; results may vary due to the large variability and complicated matrices of solid waste samples.

Subject DATA VALIDATION - NON-CLP ORGANICS FOR SOLID MATRICES	Number DV-02	Page 15 of 32
	Revision 0	Effective Date 08/13/01

2.1.5 Data Overview Prior to Validation

Before commencing validation, the reviewer must preview the associated Chain-of-Custody (COC) reports to determine:

- If the appropriate number of samples are present in the data package and if each sample was correctly analyzed for the parameters and methods specified.
- The identity of all associated field quality control blanks and field duplicate pairs.

Because many samples may have required dilutions, re-extractions and/or re-analyses, the validator should preview the data package contents to determine which analyses represent the better quality data.

Unless specifically directed by client protocol, never annotate the laboratory data package. Before beginning evaluation, prepare working copies (i.e. photocopies) of all Form I reports (including those for samples, laboratory method blanks and MS/MSD analyses) and all laboratory quality control summary forms (including all initial and continuing calibration summary statistics).

2.1.6 Technical Evaluation Summary

All data evaluations must be conducted in accordance with applicable USEPA Regional protocols and/or specific client contract requirements. The applicable documents must be referenced during the data evaluation process as this S.O.P. is only intended as a general procedure for the data validation tasks.

General parameters such as Data Completeness, Overall System Performance, Chromatographic Quality, Detection Limits and Compound Identification are evaluated concurrently with the parameters discussed in the following subsections.

2.1.6.1 Holding Times

Holding times are evaluated by reviewing the COC reports, the individual sample Form I reports, and the associated laboratory raw data. Holding times are calculated from date of collection to date of analysis.

The technical maximum holding time allowance for aqueous samples preserved with hydrochloric acid (HCL) is 14 days.

No technical holding times for solid matrices have been promulgated; a 14-day maximum holding time allowance is currently being used.

For unpreserved aqueous samples, generally a 7-day maximum holding time allowance for aromatic compounds, along with a 14-day maximum holding time allowance for chlorinated hydrocarbons is used.

Positive results in affected samples are generally qualified as estimated (J); nondetects (UJ). These results are biased low. Some USEPA Regions apply the bias qualifiers, L and UL, instead. If the holding times are exceeded by a factor of 2 or more, the holding time exceedance is considered to be gross and positive results are generally qualified as estimated (J); nondetects are generally considered to be unreliable and are qualified (R). Results for which the holding time was grossly exceeded are biased low.

2.1.6.2 Calibration

Check that an initial calibration was performed for each instrument used for analysis and that all calibrations were performed at all appropriate concentration levels.

Subject DATA VALIDATION - NON-CLP ORGANICS FOR SOLID MATRICES	Number DV-02	Page 16 of 32
	Revision 0	Effective Date 08/13/01

In general, either the correlation coefficient (R) or the Percent Relative Standard Deviation (%RSD) is evaluated in the data validation. If the correlation coefficient is chosen by the laboratory, the calibration curve should be checked for linearity. Generally, associated sample data are qualified as estimated (J, UJ) if the calibration curve correlation coefficient is <0.995. Professional judgment should be used to qualify sample data in cases when sample results fall outside the linear portion of the calibration curve. If the %RSD is used, determine which compounds have Percent Relative Standard Deviations (%RSDs) >40% and between 20%-40%. Circle these noncompliances on your working copies of these Forms. Spot-check (i.e., recalculate) a few of the %RSDs to verify the laboratory's computation.

Determine which samples are affected by reviewing the continuing calibration forms. Determine which continuing calibrations are associated with which initial calibrations. Write the affected samples on your working copies of the appropriate continuing calibration forms. Spot-check (i.e., recalculate) a few of the %Ds to verify the laboratory's computation.

Review the continuing calibration form and the associated laboratory raw data. Determine which compounds have Percent Differences (%Ds) >30% and between 15%-30%; circle the noncompliances on your working copies of these forms.

Generally, positive results for compounds for which %RSD or %D exceeds 40% or 30%, respectively, are qualified as estimated (J); nondetects (UJ). Check the specific applicable data validation protocol for further guidance as there are some protocol which reject nondetects if the %RSD or %D is excessive. Bias for these results cannot be determined.

Generally, positive results for compounds for which %RSD is between 20%-40% or %D is between 15%-30% are qualified as estimated (J). Qualification of nondetects is protocol-specific. Follow the rules provided in the appropriate validation protocol.

2.1.6.3 Blank Contamination

When using the information given below and in the appropriate USEPA Regional Functional Guidelines, keep in mind that the validation action levels derived are sample-specific and must be adjusted for dilution, sample aliquot used for analysis, and sample moisture content (when applicable).

The rules for qualifying data based on the occurrence of blank contamination vary based on regional protocols; the guidelines provided in the appropriate protocol should be followed.

Generally the blank contamination review process is completed by first considering the maximum amount of a particular contaminant occurring in the laboratory method blanks. (Do not consider lab blanks run after high concentration samples for purposes of determining carryover as laboratory method blanks!). Then repeat the process for contaminants occurring in the associated field quality control blanks. Action levels for qualification (10X or 5X depending upon whether or not the contaminant is a common contaminant) are then set. The list of common contaminants may vary among protocols. Additionally, some hierarchy among the field quality control blanks apply and the manner in which the qualifiers are applied vary [i.e. use of (U) or (B); replacement by CRQL, etc.]. Refer to appropriate protocol for specific guidance.

2.1.6.4 Surrogates

Surrogates are evaluated by reviewing the laboratory data package Form II reports and the laboratory raw data. The quality control ranges are given on the laboratory data package Form IIs; circle any noncompliances on your working copies of these Forms.

Subject DATA VALIDATION - NON-CLP ORGANICS FOR SOLID MATRICES	Number DV-02	Page 17 of 32
	Revision 0	Effective Date 08/13/01

All results for all compounds in an affected sample are qualified if any one of the surrogate spike compounds fails to meet the quality control criteria provided. Generally, for samples having a surrogate recovery <10%, positive results are qualified as estimated (J), nondetects are rejected (R). These results are biased low. For samples having a surrogate recovery which is low but >10%, positive results are generally qualified as estimated (J); nondetects (UJ). The bias qualifiers (L, UL) may be used instead, depending upon the specific USEPA Regional guidance. For samples having a surrogate recovery which is high, positive results are generally qualified as estimated (J, K) based on regional guidance; these results are biased high. Nondetects are not qualified based on high surrogate recoveries.

2.1.6.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

Generally, no data are qualified based upon MS/MSD results alone. If qualification does occur, generally only the result for that particular noncompliant compound is qualified in the original unspiked sample analysis. Refer to the applicable data validation protocol for specific procedures for evaluating MS/MSD analyses.

2.1.6.6 Other Considerations

Laboratory precision can be evaluated by comparing the unspiked sample results with MS/MSD analyses results for unspiked compounds. Consider nondetects and results reported at concentrations less than the Contract Required Quantitation Limit (CRQL) to be in agreement. Use professional judgment in determining whether to qualify sample results based on the comparison.

Likewise, compare the positive compound results for field duplicate samples. Generally, the Relative Percent Difference (RPD) between field duplicate results for the aqueous matrix should be <35%; for soil matrix results, <50%. Qualification of the sample data is limited to the specific field duplicate pair. Positive results for compounds showing imprecision are qualified as estimated (J); nondetects (UJ). Bias for these results cannot be determined.

In some USEPA Regions, a "Percent Solids" rule applies. For example, if a sediment sample contains <50% solids in USEPA Region II, all associated data are considered to be estimated and are qualified accordingly. Follow the appropriate protocol guidance when applicable.

2.1.6.7 Quantitation

Verify and record the quantitation of at least one compound per analytical fraction. If no positive results are reported, use the MS/MSD data to confirm proper computation by the laboratory. Validator and laboratory quantitations must agree within 10 percent.

2.1.7 **Deliverables Guidance**

In addition to any specific USEPA Regional requirements (e.g. data validation memorandum, data summary spreadsheets, Regional worksheets), all laboratory data package quality control summary forms, sample Form I reports, method blank Form Is, and the Chain-of-Custody report must be given to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct (in accordance with the appropriate USEPA Regional or client requirements) and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

Subject DATA VALIDATION - NON-CLP ORGANICS FOR SOLID MATRICES	Number DV-02	Page 18 of 32
	Revision 0	Effective Date 08/13/01

2.2 Semivolatiles (SW8041/8061A/8091/8310)

2.2.1 Applicability

Method 8041 is used to determine the concentration of the following phenolic compounds in groundwater, liquid, and solid matrices:

Phenol
2-Chlorophenol
2,4-Dichlorophenol
2,6-Dichlorophenol
Trichlorophenols
Tetrachlorophenols
Pentachlorophenol
Cresols (methyl phenols)
4-Chloro-3-methylphenol
2,4-Dimethylphenol
2-Nitrophenol
4-Nitrophenol
2,4-Dinitrophenol
2-sec-Butyl-4,6-dinitrophenol (DNBP)
2-Cyclohexyl-4,6-dinitrophenol
2-Methyl-4,6-dinitrophenol

Method 8061A is used to determine the concentration of the following phthalate esters in groundwater, liquid, and solid sample matrices:

Benzyl butyl phthalate
Bis(2-ethylhexyl)phthalate
Di-n-butyl phthalate
Di-n-octyl phthalate
Diethyl phthalate
Dimethyl phthalate

Method 8091 is used to determine the concentration of the following nitroaromatic and cyclic ketone compounds in groundwater, liquid, and solid sample matrices:

Nitrobenzene
Dinitrobenzene
2,4-Dinitrotoluene
2,6-Dinitrotoluene
Isophorone
Naphthoquinone

Method 8310 is used to determine the concentration of the following polynuclear aromatic hydrocarbons (PAHs) in liquid and solid sample matrices:

Acenaphthene
Acenaphthylene
Anthracene
Benzo(a)anthracene
Benzo(a)pyrene
Benzo(b)fluoranthene

Subject DATA VALIDATION - NON-CLP ORGANICS FOR SOLID MATRICES	Number DV-02	Page 19 of 32
	Revision 0	Effective Date 08/13/01

Benzo(ghi)perylene
Benzo(k)fluoranthene
Chrysene
Dibenzo(a,h)anthracene
Fluoranthene
Fluorene
Indeno(1,2,3-cd)pyrene
Naphthalene
Phenanthrene
Pyrene

All of the above methods are gas chromatographic (GC), with the exception of Method 8310 which is a High Performance Liquid Chromatography (HPLC) technique, only (i.e., no mass spectrometer detector is employed). These methods use either an electron capture detector (ECD), a flame ionization detector (FID), a ultraviolet detector (UV), or a fluorescence detector.

2.2.2 Interferences

Solvents, reagents, glassware, and other sample-processing hardware may yield discrete artifacts and/or elevated baselines causing misinterpretation of gas chromatograms. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by running method blanks. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be required.

Interferences co-extracted from samples will vary considerably from source to source depending upon the waste being sampled. While general cleanup techniques such as Method 3530 are provided as part of these methods, unique samples may require additional cleanup.

If sample or matrix interferences occur, a secondary column may be employed in addition to the primary column so as to resolve any questionable compound results.

2.2.3 General Laboratory Practices

An extraction blank should be prepared with each batch of samples extracted.

Matrix Spike/Matrix Spike Duplicate (MS/MSD) analyses should be conducted to determine the effects of sample matrix upon the compounds of interest.

2.2.4 Sample Preparation

Prior to analysis, the samples must be extracted using the appropriate techniques. Aqueous samples are extracted at the appropriate pH with methylene chloride as a solvent using Method 3510 (separatory funnel extraction) or Method 3520 (continuous liquid-liquid extraction). Both neat and diluted organic liquids may be analyzed by direct injection. Solid samples are extracted at the appropriate pH with methylene chloride using either Soxhlet Extraction (Method 3540) or Sonication (Method 3550) procedures.

2.2.5 Data Overview Prior to Validation

Before commencing validation the reviewer must preview the associated Chain-of-Custody (COC) reports to determine:

Subject DATA VALIDATION - NON-CLP ORGANICS FOR SOLID MATRICES	Number DV-02	Page 20 of 32
	Revision 0	Effective Date 08/13/01

- If the appropriate number of samples are present in the data package and if each sample was correctly analyzed for the parameters and methods specified.
- The identity of all associated field quality control blanks and field duplicate pairs.

Because many samples may have required dilutions, re-extractions and/or reanalyses, the data validator should preview the data package contents to determine which analyses represent the better quality data.

The data package should never be annotated unless specifically directed by client protocol. All Form I reports (including those for samples, laboratory method blanks, and MS/MSD analyses) and all laboratory quality control summary forms (including all initial and continuing calibration summary statistics) should be photocopied for use as working copies.

2.2.6 Technical Evaluation Summary

All data evaluations must be conducted in accordance with the appropriate USEPA Regional protocols and/or specified client contract requirements. The applicable documents must be referenced during the data validation process as this S.O.P. is only intended as a general procedure for the data validation tasks.

General parameters such as Data Completeness, Overall System Performance, Chromatographic Quality, Detection Limits and Compound Identification are evaluated concurrently with the parameters discussed in the following subsections.

2.2.6.1 Holding Times

Holding times are evaluated by reviewing the COC reports, the individual sample Form I reports, and the associated laboratory raw data. Holding times for extraction are calculated from date of collection to date of extraction.

The technical holding times for aqueous and solid matrices are as follows:

- Extraction:
 - Water samples: 7 days
 - Solid samples: 14 days
- Analysis: 40 days from date of extraction

Generally, positive results affected by noncompliances are qualified as estimated (J); nondetects (UJ). These results are considered to be biased low. Alternately, the bias qualifiers L and UL may be used. Nondetects may be rejected (R) when the sample was extracted after 14 days (28 days for solid samples). If the holding time until analysis has been exceeded (and potentially, some of the extract may have evaporated), the affected sample results may be considered to be biased high. Refer to the appropriate data validation protocol for specific guidance.

2.2.6.2 Calibration

Check that an initial calibration was performed for each instrument used for analysis and that all calibrations were performed at all appropriate concentration levels.

In general, either the correlation coefficient (R) or the Percent Relative Standard Deviation (%RSD) is evaluated in the data validation. If the correlation coefficient is chosen by the laboratory, the calibration curve should be checked for linearity. Generally, associated sample data are qualified as estimated (J, UJ) if the calibration curve correlation coefficient is <0.995. Professional judgment should be used to

Subject DATA VALIDATION - NON-CLP ORGANICS FOR SOLID MATRICES	Number DV-02	Page 21 of 32
	Revision 0	Effective Date 08/13/01

qualify sample data in cases when sample results fall outside the linear portion of the calibration curve. If the %RSD is used, determine which compounds have Percent Relative Standard Deviations (%RSDs) >40% and between 20%-40%. Circle these noncompliances on your working copies of these Forms. Spot-check (i.e., recalculate) a few of the %RSDs to verify the laboratory's computation.

Determine which samples are affected by reviewing the continuing calibration forms. Determine which continuing calibrations are associated with which initial calibrations. Write the affected samples on your working copies of the appropriate continuing calibration forms. Spot-check (i.e., recalculate) a few of the %Ds to verify the laboratory's computation.

Review the continuing calibration form and the associated laboratory raw data. Determine which compounds have Percent Differences (%Ds) >30%, and between 15%-30%; circle the noncompliances on your working copies of these forms.

Generally, positive results for compounds for which %RSD or %D exceeds 40% or 30%, respectively, are qualified as estimated (J); nondetects (UJ). Check the specific applicable data validation protocol for further guidance as there are some protocol which reject nondetects if the %RSD or %D is excessive. Bias for these results cannot be determined.

Generally, positive results for compounds for which %RSD is between 20%-40% or whose %D is between 15%-30% are qualified as estimated (J). Qualification of nondetects is protocol-specific. Follow the rules provided in the appropriate validation protocol.

2.2.6.3 Blank Contamination

When using the information given below and in the appropriate USEPA Regional Functional Guidelines, keep in mind that the validation action levels derived are sample-specific and must be adjusted for dilution, sample aliquot used for analysis, and sample moisture content (when applicable).

The rules for qualifying data based on the occurrence of blank contamination vary based on regional protocols; the guidelines provided in the appropriate protocol should be followed.

Generally the blank contamination review process is completed by first considering the maximum amount of a particular contaminant occurring in the laboratory method blanks. (Do not consider lab blanks run after high concentration samples for purposes of determining carryover as laboratory method blanks!) Then repeat the process for contaminants occurring in the associated field quality control blanks. Action levels for qualification (10X or 5X depending upon whether or not the contaminant is a common contaminant) are then set. The list of common contaminants may vary among protocols. Additionally, some hierarchy among the field quality control blanks apply and the manner in which the qualifiers are applied vary [i.e. use of (U) or (B); replacement by CRQL, etc.]. Refer to appropriate protocol for specific guidance.

2.2.6.4 Surrogates

Surrogates are evaluated by reviewing the laboratory data package Form II reports and the laboratory raw data. The quality control ranges are given on the laboratory data package Form IIs; circle any noncompliances on your working copies of these Forms.

All results for all compounds in an affected sample are qualified if any one of the surrogate spike compounds fails to meet the quality control criteria provided. Generally, for samples having a surrogate recovery <10%, positive results are qualified as estimated (J), nondetects are rejected (R). These results are biased low. For samples having a surrogate recovery which is low but >10%, positive results are

Subject DATA VALIDATION - NON-CLP ORGANICS FOR SOLID MATRICES	Number DV-02	Page 22 of 32
	Revision 0	Effective Date 08/13/01

generally qualified as estimated (J); nondetects (UJ). The bias qualifiers (L, UL) may be used instead, depending upon the specific USEPA Regional guidance. For samples having a surrogate recovery which is high, positive results are generally qualified as estimated (J, K) based on regional guidance; these results are biased high. Nondetects are not qualified based on high surrogate recovery.

2.2.6.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

Generally, no data are qualified based upon MS/MSD results alone. If qualification does occur, generally only the result for that particular noncompliant compound is qualified in the original unspiked sample. Refer to the applicable data validation protocol for specific procedures for evaluating MS/MSD analyses.

2.2.6.6 Other Considerations

Laboratory precision can be evaluated by comparing the unspiked sample results with MS/MSD analyses results for unspiked compounds. Consider nondetects and results reported at concentrations less than the Contract Required Quantitation Limit (CRQL) to be in agreement. Use professional judgment in determining whether to qualify sample results based on the comparison.

Likewise, compare the positive compound results for field duplicate samples. Generally, the Relative Percent Difference (RPD) between field duplicate results for the aqueous matrix should be <35%; for soil matrix results, <50%. Qualification of the sample data is limited to the specific field duplicate pair. Positive results for compounds showing imprecision are qualified as estimated (J); nondetects (UJ). Bias for these results cannot be determined.

In some USEPA Regions, a "Percent Solids" rule applies. For example, if a sediment sample contains <50% solids in USEPA Region II, all associated data are considered to be estimated, and are qualified accordingly. Follow the appropriate protocol guidance when applicable.

2.2.6.7 Quantitation

Verify and record the quantitation of at least one compound per analytical fraction. If no positive results are reported, use the MS/MSD data to confirm proper computation by the laboratory. Validator and laboratory quantitations must agree within 10 percent.

2.2.7 **Deliverables Guidance**

In addition to any specific USEPA Regional requirements (e.g. data validation memorandum, data summary spreadsheets, Regional worksheets), all laboratory data package quality control summary forms, sample Form I reports, method blank Form Is, and the Chain-of-Custody report, must be given to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct (in accordance with the appropriate USEPA Regional or client requirements), and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

Subject DATA VALIDATION - NON-CLP ORGANICS FOR SOLID MATRICES	Number DV-02	Page 23 of 32
	Revision 0	Effective Date 08/13/01

2.3 Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs), Organophosphorous Pesticides, Chlorinated Herbicides (SW 8081A/8082/8141A/8151A)

2.3.1 Applicability

Methods 8081A/8082 are used to determine the concentration of the following organochlorine pesticides and polychlorinated biphenyls (PCBs) in groundwater, liquid, and solid sample matrices:

Aldrin
alpha-BHC
beta-BHC
delta-BHC
gamma-BHC (Lindane)
Chlordane
4,4'-DDD
4,4'-DDE
4,4'-DDT
Dieldrin
Endosulfan I
Endosulfan II
Endosulfan sulfate
Endrin
Endrin aldehyde
Heptachlor
Heptachlor epoxide
Methoxychlor
Toxaphene
Aroclor-1016
Aroclor-1221
Aroclor-1232
Aroclor-1242
Aroclor-1248
Aroclor-1254
Aroclor-1260

Similarly, Method 8141A is used to determine the following pesticides in groundwater and waste samples:

Azinphos methyl
Bolstar (Sulprofos)
Chlorpyrifos
Coumaphos
Demeton-O
Demeton-S
Diazinon
Dichlorvos
Disulfoton
Ethoprop
Fensulfothion
Fenthion
Merphos
Mevinphos
Naled

Subject DATA VALIDATION - NON-CLP ORGANICS FOR SOLID MATRICES	Number DV-02	Page 24 of 32
	Revision 0	Effective Date 08/13/01

Parathion methyl
Phorate
Ronnel
Stirophos (Tetrachlorvinphos)
Tokuthion (Prothiofos)
Trichloronate

Note that when Method 8141A is used to analyze unfamiliar samples, compound identifications should be supported by at least one additional qualitative technique if mass spectroscopy is not employed.

Method 8151A is used to determine the following chlorinated acid herbicides in groundwater and waste samples:

2,4-D
2,4-DB
2,4,5-T
2,4,5-TP (Silvex)
Dalapon
Dicamba
Dichloroprop
Dinoseb
MCPA
MCP
4-Nitrophenol
Pentachlorophenol

Since these compounds are produced and used in various forms (i.e., acid, salt, ester, etc.), Method 8151A includes a hydrolysis step to convert the herbicide to the acid form prior to analysis. When Method 8151A is used to analyze unfamiliar samples, compound identifications should be supported by at least one additional qualitative technique. This method describes analytical conditions for a second gas chromatographic column that can be used to confirm measurements made with the primary column; alternately, the compounds of interest can be confirmed by detection via a mass spectrometer.

All of the above Methods are Gas Chromatographic (GC) in which sample extracts are analyzed by direct injection. Methods 8081A and 8082 analyze for organochlorine pesticide compounds and PCBs via GC/ECD (Electron Capture Detector; an equivalent Halogen-Specific Detector may also be used). Method 8141A analyzes for organophosphorous pesticide compounds via GC/FID (Flame Ionization Detector), and Method 8151A analyzes for chlorinated herbicide compounds via GC/ECD (alternately, a Microcoulometric Detector or Hall Electrolytic Conductivity Detector may be used).

2.3.2 Interferences

The sensitivity of these methods usually depends on the level of interferences rather than on instrumental limitations. Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines causing misinterpretation of gas chromatograms. The use of high purity reagents and solvents helps to minimize these interference problems. Extraction blanks are analyzed as method blanks in order to monitor the occurrences of interferences.

Interferences co-extracted from the sample will vary considerably, and will dictate the nature and extent of clean-up procedures used. Phthalate esters are a common interference to organochlorine pesticide analyses; phenols and organic acids may act as interferents when analyzing for chlorinated herbicides.

Subject DATA VALIDATION - NON-CLP ORGANICS FOR SOLID MATRICES	Number DV-02	Page 25 of 32
	Revision 0	Effective Date 08/13/01

2.3.3 General Laboratory Practices

Matrix Spike/Matrix Spike Duplicate (MS/MSD) analyses should be conducted to determine the effects of sample matrix upon the compounds of interest.

Standard quality assurance practices such as the analyses of field replicate and laboratory duplicates should also be employed.

Note that herbicides, being strong organic acids, react readily with alkaline substances and may be lost during analysis. Therefore, when performing Method 8151A, glassware and glass wool must be acid-rinsed and sodium sulfate must be acidified with sulfuric acid prior to use to avoid this possibility.

2.3.4 Sample Preparation

Prior to the use of Methods 8081, 8082, and 8141A, aqueous samples are extracted at a neutral pH with methylene chloride as a solvent using a separatory funnel (Method 3510) or a continuous liquid- liquid extractor (Method 3520). Solid samples are extracted with hexane:acetone (1:1) using either the Soxhlet extraction (Method 3540) or sonication (Method 3550) procedures.

Method 8151A provides its own specific preparation procedures for aqueous and solid samples which include extraction with acetone and diethyl ether followed by esterification using diazomethane as a derivatizing agent.

2.3.5 Data Overview Prior to Validation

Before commencing validation, the reviewer must preview the associated Chain-of-Custody (COC) reports to determine:

- If the appropriate number of samples are present in the data package and if each sample was correctly analyzed for the parameters and methods specified.
- The identity of all associated field quality control blanks and field duplicate pairs.

Because many samples may have required dilutions, re-extractions and/or re-analyses, the validator should preview the data package contents to determine which analyses represent the better quality data.

Unless specifically directed by client protocol, never annotate the laboratory data package. Before beginning evaluation, prepare working copies (i.e., photocopies) of all Form I reports (including those for samples, laboratory method blanks and MS/MSD analyses) and all laboratory quality control summary forms.

2.3.6 Technical Evaluation Summary

All data evaluations must be conducted in accordance with applicable USEPA Regional protocols and/or specific client contract requirements. The applicable documents must be referenced during the data evaluation process as this S.O.P. is only intended as a general procedure for the data validation tasks.

General parameters such as Data Completeness, Overall System Performance, Chromatographic Quality, Detection Limits, and Compound Identification are evaluated concurrently with the parameters discussed in the following subsections.

Subject DATA VALIDATION - NON-CLP ORGANICS FOR SOLID MATRICES	Number DV-02	Page 26 of 32
	Revision 0	Effective Date 08/13/01

2.3.6.1 Holding Times

Holding times are evaluated by reviewing the COC reports, the individual sample Form I reports, and the associated laboratory raw data. Holding times for extraction are calculated from date of collection to date of extraction.

The technical holding times for aqueous and solid matrices are as follows:

- Extraction:
 - Water samples: 7 days
 - Solid samples: 14 days
- Analysis: 40 days from date of extraction

When the holding time criteria are not met, positive results in affected samples are generally qualified as estimated (J); nondetects (UJ). These results are biased low. Some USEPA Regions apply the bias qualifiers, L and UL, instead. If the holding times are exceeded by a factor of 2 or more, the holding time exceedance is considered to be gross and positive results are generally qualified as estimated (J); nondetects are generally considered to be unreliable and are rejected (R). These results are biased very low.

2.3.6.2 Calibration

Data pertaining to the initial calibration (i.e., evaluation check for linearity) is found on the data package Form VIs or equivalent. Check that an initial calibration was performed for each instrument used and at all appropriate concentration levels.

In general, either the correlation coefficient (R) or the Percent Relative Standard Deviation (%RSD) is evaluated in the data validation. If the correlation coefficient is chosen by the laboratory, the calibration curve should be checked for linearity. Generally, associated sample data are qualified as estimated (J, UJ) if the calibration curve correlation coefficient is <0.995. Professional judgment should be used to qualify sample data in cases when sample results fall outside the linear portion of the calibration curve. If the %RSD is used, determine which compounds have Percent Relative Standard Deviations (%RSDs) >40% and between 20%-40%. Circle these noncompliances on your working copies of these Forms. Spot-check (i.e., recalculate) a few of the %RSDs to verify the laboratory's computation.

Determine which samples are affected by reviewing the continuing calibration forms. Determine which continuing calibrations are associated with which initial calibrations. Write the affected samples on your working copies of the appropriate continuing calibration forms. Spot-check (i.e., recalculate) a few of the %Ds to verify the laboratory's computation.

Review the continuing calibration form and the associated laboratory raw data. Determine which compounds have Percent Differences (%Ds) >30% and between 15%-30%; circle the noncompliances on your working copies of these forms.

Generally, positive results for compounds for which %RSD or %D exceeds 40% or 30%, respectively, are qualified as estimated (J); nondetects (UJ). Check the specific applicable data validation protocol for further guidance as there are some protocol which reject nondetects if the %RSD or %D is excessive. Bias for these results cannot be determined.

Generally, positive results for compounds for which %RSD is between 20%-40% or %D is between 15%-30% are qualified as estimated (J). Qualification of nondetects is protocol-specific. Follow the rules provided in the appropriate validation protocol.

Subject DATA VALIDATION - NON-CLP ORGANICS FOR SOLID MATRICES	Number DV-02	Page 27 of 32
	Revision 0	Effective Date 08/13/01

Method 8081A requires analysis of a DDT/Endrin breakdown check standard. The DDT/Endrin Breakdown should not exceed 20%. Generally, if % breakdown for DDT exceeds 20%, estimate (J) all positive results for DDT, DDE and DDD following the in-last control standard until the next in-control standard (see analytical sequence). If there are no positive results for DDT but there are positive results for DDD or DDE then reject (R) nondetects for DDT in associated samples. Generally, if Endrin % Breakdown exceeds 20%, estimate (J) positive results for Endrin, Endrin Aldehyde, and Endrin Ketone in all samples following the last in-control standard until the next acceptable standard. If there are positive results for Endrin Aldehyde or Endrin Ketone but none for Endrin, reject (R) nondetect Endrin results.

2.3.6.3 Blank Contamination

When using the information provided below and in the appropriate USEPA Regional Functional Guidelines, keep in mind that the validation action levels derived are sample-specific, and must be adjusted for dilution, sample aliquot used for analysis, and sample moisture content (when applicable).

The rules for qualifying data based on the occurrence of blank contamination vary based on regional protocols; guidelines provided in the appropriate data validation protocol should be followed.

An action level of 5X the maximum amount of contaminant found is used to evaluate the sample data. The manner in which the qualifiers are applied vary [i.e. use of (U) or (B); replacement by CRQL, etc.]. Refer to appropriate validation protocol for specific guidance.

2.3.6.4 Surrogates

Surrogates are evaluated by reviewing the laboratory data package Form II reports and the associated laboratory raw data. The advisory limits are given on the laboratory data package Form IIs; circle any recoveries outside these limits on your working copies of these Forms.

No qualifications are made for surrogates which show zero recoveries because they were "diluted out." Generally, positive results affected by low surrogate recovery are qualified as estimated (J) or the (L) bias qualifier is used when applicable; nondetects are qualified (UJ) or (UL), accordingly. If a positive sample result is affected by high surrogate recovery, the result is qualified as estimated (J) or the (K) bias qualifier is used when applicable; nondetects are not qualified based on high surrogate recovery. Because the surrogate recovery limits for these fractions are advisory, generally no results are rejected.

The pesticide/PCB surrogates decachlorobiphenyl (DCB) and tetrachloro-m-xylene (TCX) retention times found on data package Form VIII or equivalent must be 0.10 for DCB and 0.05 for TCX. If DCB and TCX retention time criteria are not met, the raw data must be checked for misidentified GC peak. The validator's professional judgment for qualifications should be used.

2.3.6.5 Matrix Spike/Matrix Spike Duplicates

Generally, no data are qualified based upon MS/MSD results alone. If qualification does occur, generally only the result for that particular noncompliant compound is qualified in the original unspiked sample analysis. Refer to the appropriate data validation guidelines for specific procedures for evaluating MS/MSD analyses.

2.3.6.6 Other Considerations

Laboratory precision can be evaluated by comparing the unspiked sample results with MS/MSD analyses results for unspiked compounds. Consider nondetects and results reported at concentrations less than the Contract Required Quantitation Limit (CRQL) to be in agreement. Use professional judgment in determining whether to qualify sample results based on the comparison.

Subject DATA VALIDATION - NON-CLP ORGANICS FOR SOLID MATRICES	Number DV-02	Page 28 of 32
	Revision 0	Effective Date 08/13/01

Likewise, compare the positive compound results for field duplicate samples. Generally, the Relative Percent Difference (RPD) between field duplicate results for the aqueous matrix should be <35%; for soil matrix results, <50%. Qualification of the sample data is limited to the specific field duplicate pair. Positive results for compounds showing imprecision are qualified as estimated (J); nondetects (UJ). Bias for these results cannot be determined.

In some USEPA Regions, a "Percent Solids" rule applies. For example, if a sediment sample contains <50% solids in USEPA Region II, all associated data are considered to be estimated and are qualified accordingly. Follow the appropriate protocol guidance when applicable.

2.3.6.7 Quantitation

Verify and record the quantitation of at least one compound per analytical fraction. If no positive results are reported, use the MS/MSD data to confirm proper computation by the laboratory. Validator and laboratory quantitations must agree within 10%.

2.3.7 **Deliverables Guidance**

In addition to any specific USEPA Regional requirements (e.g. data validation memorandum, data summary spreadsheets, USEPA Regional worksheets), all laboratory data package quality control summary forms, sample Form I reports, method blank Form Is, and the Chain-of-Custody report, must be given to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct (in accordance with the appropriate USEPA Regional or client requirements), and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

2.4 **Explosives/Nitroaromatics/Nitroamines(SW 8330)**

2.4.1 **Applicability**

Method 8330 is used to determine the concentration of the following explosives, nitroaromatics, and nitroamines in water, soil, or sediment matrices:

- Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
- Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)
- 1,3,5-Trinitrobenzene (1,3,5-TNB)
- 1,3-Dinitrobenzene (1,2-DNB)
- Methyl-2,4,6-trinitrophenylnitramine (Tetryl)
- Nitrobenzene (NB)
- 2,4,6-Trinitrotoluene (2,4,6-TNT)
- 4-Amino-2,6-dinitrotoluene (4-Am-DNT)
- 2-Amino-4,6-dinitrotoluene (2-Am-DNT)
- 2,4-Dinitrotoluene (2,4-DNT)
- 2,6-Dinitrotoluene (2,6-DNT)
- 2-Nitrotoluene (2-NT)
- 3-Nitrotoluene (3-NT)
- 4-Nitrotoluene (4-NT)
- Nitroguanidine
- Nitroglycerin
- Pentaerythritol Tetranitrate (PETN)

Subject DATA VALIDATION - NON-CLP ORGANICS FOR SOLID MATRICES	Number DV-02	Page 29 of 32
	Revision 0	Effective Date 08/13/01

The analysis of the compounds listed above is conducted by High Performance Liquid Chromatography equipped with a 254 nm Ultra Violet (UV) detector. This method is capable of determining part per billion (ppb) detection levels in water and soil matrices.

The method requires the use of both a primary (C-18 reverse phase) and a confirmation (CN reverse phase) column.

2.4.2 Interferences

The sensitivity of this method usually depends on the level of interferences rather than on instrumental limitations. Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines causing misinterpretation of gas chromatograms. The use of high purity reagents and solvents helps to minimize these interference problems. Extraction blanks are analyzed as method blanks in order to monitor the occurrences of interferences.

2,4-Dinitrotoluene and 2,6-dinitrotoluene may co-elute. High concentrations of one of the two isomers may cause interference of the other isomer. In instances where this is applicable, both isomers should be reported as one. Baseline resolution should be present for all compounds.

Decomposition of Tetryl occurs rapidly and when exposed to heat. Samples expected to contain Tetryl should not be exposed to temperatures above room temperature.

2.4.3 General Laboratory Practices

Method blanks and instrumentation blanks should be conducted to access laboratory contamination.

Matrix spike/Matrix Spike Duplicate (MS/MSD) analyses should be conducted to determine the effects of sample matrix upon the compounds of interest.

Standard quality assurance practices such as the analyses of field and laboratory duplicates may also be employed.

2.4.4 Sample Preparation

Method 8330 provides its own specific preparation procedures for aqueous and solid samples which include extraction with acetonitrile and a salting-out procedure for aqueous samples. Soil samples are air dried prior to preparation, thus percent moisture is not a consideration when calculating compound concentrations.

2.4.5 Data Overview Prior to Validation

Before commencing validation, the reviewer must preview the associated Chain-of-Custody (COC) reports to determine:

- If the appropriate number of samples are present in the data package.
- If each sample was correctly analyzed and identified for the specified parameters.
- The identity of all associated field quality control blanks and field duplicate pairs.

Because many samples may have required dilutions, re-extractions and /or re-analyses, the validator should preview the data package contents to determine which analyses represent the best data quality.

Subject DATA VALIDATION - NON-CLP ORGANICS FOR SOLID MATRICES	Number DV-02	Page 30 of 32
	Revision 0	Effective Date 08/13/01

Unless specifically directed by the client, never annotate the laboratory data package. Before beginning evaluation, prepare working copies(i.e. photocopies) of all Form I reports (including those for samples, laboratory method blanks and MS/MSD analyses) and all laboratory quality control summary forms.

2.4.6 Technical Evaluation Summary

All data evaluations must be conducted in accordance with applicable USEPA Regional protocols, method requirements, and/or specific client contract requirements. The applicable documents must be referenced during the data evaluation process as this SOP is only intended as a general procedure for the data validation task.

Deficiencies, omissions, and/or other anomalies noted during the review require the data validator to contact the laboratory.

General parameters such as Data Completeness, Overall System Performance, Chromatographic Quality, Detection Limits, and Compound Identification are evaluated concurrently with the parameters discussed in the following subsections.

2.4.6.1 Holding Times

Holding times are evaluated by reviewing the COC reports, the individual sample Form I reports, and the associated laboratory raw data. Holding times for extraction are calculated from the date of collection to the date of extraction.

The technical holding times for aqueous and solid matrices are as follows:

- Extraction:
 - Water Samples: 7 days
 - Solid Samples: 14 days
- Analysis: 40 days from date of extraction

When the holding times criteria are not met, positive results in affected samples are generally qualified as estimated, (J); nondetected results, (UJ). These results are considered biased low. Some USEPA Regions apply the bias qualifiers, L and UL, instead. If holding times are exceeded by a factor of two or more, the holding time exceedance is considered to be gross and positive results are generally qualified as estimated (J); nondetects are generally considered to be unreliable and are rejected, (R). These results are considered to be biased very low.

2.4.6.2 Calibrations

Data pertaining to the initial calibration (i.e. evaluation check for linearity) is found on the data package Form VIs or equivalent. Check that an initial calibration was performed for each instrument used and at all appropriate concentration levels. The initial calibration should consist of a minimum of five concentration levels for each compound of interest.

In general, either the correlation coefficient (r) or the Percent Relative Standard Deviation (%RSD) is evaluated in the data validation. If the correlation coefficient is chosen the laboratory, the calibration curve should be checked for linearity. Generally, associated sample data are qualified as estimated (J, UJ) if the calibration curve correlation coefficient is < 0.995. Professional judgment should be used to qualify sample data in cases when sample results fall outside the linear portion of the calibration curve. If the %RSD is used, determine which compounds have %RSDs greater than 20%. Generally, associated data are qualified as estimated (J/UJ) if the calibration %RSD is >20%. Circle these noncompliances on working copies of calibration forms.

Subject DATA VALIDATION - NON-CLP ORGANICS FOR SOLID MATRICES	Number DV-02	Page 31 of 32
	Revision 0	Effective Date 08/13/01

Determine which samples are affected by reviewing the continuing calibration forms. Determine which continuing calibrations are associated with each initial calibration by instrument. Write the affected samples on working copies of the appropriate continuing calibration forms. Spot-check (i.e. recalculate) a few of the %Ds to verify the laboratory's computation.

A continuing calibration or daily calibration must be performed at the beginning, midpoint and end of the analytical sequence. The continuing calibration response factor for each analyte must be compared to the response factor of the initial calibration. The continuing calibration response factor must agree within 15% of the initial response factor. Generally, positive and nondetected results are qualified as estimated (J/UJ) if the Percent Difference (%D) is >15%.

2.4.6.3 Blank Contamination

A review of all method and instrument blanks (if provided) is conducted to evaluate laboratory contaminants. An additional review of all relevant field quality control blanks is also conducted. Contaminants, if present, are summarized and the maximum concentration of each contaminant is selected and used to establish blank action levels.

An action level of 5X the maximum amount of each contaminant is used to evaluate sample data. Blank action levels must consider the aliquot used for analysis and sample dilution. Positive results less than the action level are qualified as false positives. The manner in which the qualifiers are applied varies [i.e., use of (U) or (B); replacement by the Reporting Limit]. General regional guidance procedures dictate the most appropriate validation action qualification.

2.4.6.4 Surrogates

Surrogates are evaluated by reviewing the laboratory data package Form II or equivalent and the associated laboratory raw data. The advisory limits are given on the laboratory data package Form IIs. Circle any recoveries outside these limits on working copies.

Generally, positive results affected by low surrogate recoveries are estimated, (J) or (L), indicating low bias; nondetected results are qualified, (UJ) or (UL), accordingly. If a positive sample result is affected by high surrogate recovery, the result is qualified as estimated, (J), or the bias qualifier (K), is used when applicable. Nondetected results are not qualified based upon high surrogate recoveries. It should be noted that consideration of interferences may affect surrogate recoveries. If a trend of noncompliance is noted, an evaluation of sample chromatograms should be conducted when surrogate recoveries are noncompliant and a matrix effect is suspected.

No qualifications are made for surrogates which have been diluted out.

Generally, positive results associated with surrogate recoveries <10% are qualified as estimated, (J) or biased low (L). Nondetected results associated with surrogate recoveries <10 are considered unreliable and are qualified rejected (R).

2.4.6.5 Matrix Spike/Matrix Spike Duplicates

Generally, no data are qualified based upon MS/MSD results alone. If qualification does occur, generally only the result for that particular noncompliant compound is qualified in the unspiked sample. Typically, Percent Recoveries (%Rs) and the Relative Percent Difference (RPD) are evaluated based upon the laboratory provided control limits.

Subject DATA VALIDATION - NON-CLP ORGANICS FOR SOLID MATRICES	Number DV-02	Page 32 of 32
	Revision 0	Effective Date 08/13/01

2.4.6.6 Other Considerations

Laboratory precision can be evaluated by comparing the unspiked samples results with MS/MSD analyses result for unspiked compounds. Consider nondetected results and results reported at concentrations less than the reporting limit to be in agreement. Use professional judgment in determining whether to qualify sample results based upon the comparison. The comparison may be presented in terms of a %RSD or an RPD.

Likewise, compare positive compound results for field duplicate samples. Generally, an RPD between field duplicate results for the aqueous matrix should be <35%; for soil matrix results <50%. Qualification of the sample data is limited to specific field duplicate pair. Positive results for compounds showing imprecision are qualified as estimated (J); nondetected results (UJ)

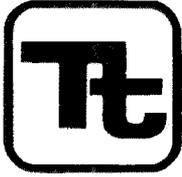
2.4.6.7 Quantitation

Verify and record the quantitation of at least one compound per fraction. If no positive results are reported, use the MS/MSD data to confirm proper computation by the laboratory. The validator and laboratory quantitations must agree within 10%. If quantitation differences are significant, the laboratory must be contacted to investigate and resolve the discrepancy.

2.2.4.7 Deliverable Guidance

In addition to any specific USEPA Regional requirements (i.e., data validation memorandum, data summary spreadsheets, USEPA Regional Worksheets), all laboratory data package quality summary forms, sample Form I reports method blank results and the Chain of Custody records must be included in the validation report.

The validator should ensure that the format of the data validation deliverable is complete and correct (in accordance with the appropriate USEPA Regional or client requirements) and that the narrative is free of transcription and typographical errors before submitting all requested items for quality assurance review.



TETRA TECH NUS, INC.

STANDARD OPERATING PROCEDURES

Number DV-04	Page 1 of 8
Effective Date 08/13/01	Revision 0
Applicability Tetra Tech NUS, Inc.	
Prepared Risk Assessment Department	
Approved D. Senovich <i>[Signature]</i>	

Subject
DATA VALIDATION - NON-CLP INORGANICS FOR
SOLID AND AQUEOUS MATRICES

TABLE OF CONTENTS

<u>SECTION</u>	<u>PAGE</u>
1.0 INORGANICS (SW-846 6010B/7470A/7471A/9010A&B/7470/9010)	2
1.1 APPLICABILITY	2
1.2 DATA OVERVIEW PRIOR TO VALIDATION PROCESS	2
1.2.1 Data Completeness	2
1.3 TECHNICAL EVALUATION SUMMARY	3
1.3.1 Holding Times	3
1.3.2 Initial Calibration Requirements	4
1.3.3 Initial and Continuing Calibration Verification (ICV/CCV)	4
1.3.4 Laboratory Method and Field Quality Control Blanks	4
1.3.5 ICP Interference Check Sample Results	5
1.3.6 Matrix Spike Sample Analysis (Pre-digestion)	6
1.3.7 Laboratory Duplicate Precision	6
1.3.8 Field Duplicate Precision	7
1.3.9 Laboratory Control Sample Results	7
1.3.10 Method of Standard Additions (MSA)	7
1.3.11 ICP Serial Dilution Analysis	8
1.3.12 Analysis Run Logs Form 14	8
1.3.13 Further GFAA Evaluations	8
1.4 DELIVERABLES GUIDANCE	8

Subject DATA VALIDATION - NON-CLP INORGANICS OR SOLID AND AQUEOUS MATRICES	Number DV-04	Page 2 of 8
	Revision 0	Effective Date 08/13/01

1.0 INORGANICS (SW-846 6010B/7470A/7471A/9010A&B/7470/9010)

Inductively Coupled Plasma Emission Spectroscopy (ICP) - Analytes commonly analyzed using ICP include: aluminum, barium, beryllium, cadmium, calcium, chromium, cobalt, copper, iron, magnesium, manganese, nickel, potassium, silver, sodium, vanadium, and zinc.

Graphite Furnace Atomic Absorption Spectroscopy (GFAA) - Analytes commonly analyzed using GFAA include: antimony, arsenic, lead, selenium, and thallium.

Cold Vapor Methodology - Mercury is commonly analyzed using cold vapor methodology.

Automated Colorimetric Technique - Cyanide is commonly analyzed using automated colorimetric methodology.

1.1 Applicability

These methods are applicable to a large number of matrices including EP extracts, TCLP extracts, industrial wastes, soils, groundwater, aqueous samples, sludges, sediments, and other solid wastes. All matrices require digestion prior to analysis.

Detection limits for analytes are established on a quarterly basis and are both laboratory and instrument specific.

1.2 Data Overview Prior to Validation Process

1.2.1 Data Completeness

The data reviewer must initially verify that all forms are present and complete (i.e., Forms 1 through 14 must be provided). Areas of special attention when accounting for required forms will include:

Verify at least one Initial and Continuing Calibration Verification (ICV/CCV) Percent Recovery (%R) calculation as noted on the Calibration Summary (Form 2A or equivalent).

Verify that a matrix-specific laboratory generated preparation blank has been analyzed for each respective matrix as noted on the blank summary (Form 3 or equivalent) (note, filtered and unfiltered aqueous matrices are to be treated as distinctly different matrices).

Verify that all ICP analytes are present in both ICSA and ICSAB solutions. Also, verify from the raw data that the laboratory reported all analytes present in solution A to the nearest whole number. It is not uncommon for laboratories to incorrectly report "zeros" or simply leave blank the appropriate solution A columns.

Check that one matrix spike was analyzed for each particular matrix per analytical batch. Laboratories typically will not include an aqueous matrix for waters if the only aqueous samples contained in the SDG are field quality control blanks (i.e., equipment rinsate blanks and/or field blanks). This is generally accepted without data validation letter text comment. Additionally, the data reviewer may want to verify spiking levels.

Verify that laboratory duplicate analyses were performed for each matrix. **NOTE:** Field quality control blanks are never to be designated for quality control analyses.

Subject DATA VALIDATION - NON-CLP INORGANICS OR SOLID AND AQUEOUS MATRICES	Number DV-04	Page 3 of 8
	Revision 0	Effective Date 08/13/01

Check that one Laboratory Control Sample (LCS) was analyzed for each batch of samples per matrix within an SDG. **NOTE:** An aqueous LCS is not required for mercury and cyanide analysis.

The Method of Standard Additions (MSA) (Form 8 or equivalent) may or may not be present as dictated by Post Digestion Spike (PDS) %Rs. See Section 4.1.3.11 for further details.

Verify that at least one ICP serial dilution analysis was performed for each matrix within an SDG. **NOTE:** Typically one serial dilution will serve to monitor a given set of samples within an SDG. However, special contractual requirements may necessitate one serial dilution analysis per sample. Ascertain atypical serial dilution frequency requirements through the project manager.

Simply check that the Form 11 ICP Interelement Correction Factors (Annually) is present.

Verify that all ICP analytical results fall within the ICP Quarterly Linear Ranges provided on the Form 12 (or equivalent). Verify that no GFAA analytical results exceed the highest standard in the associated GFAA calibration.

Verify that the Preparation Log accounts for aqueous/soil ICP, AA, mercury, and cyanide digestions/distillation as applicable.

Examine the Form 14s (or equivalent) to verify that one and only one "X" flag has been used to signify each reported field sample result or quality control sample result. Laboratories are often careless when entering the "X" flag. The validator must verify reported results in instances of discrepancies, amend appropriate forms, and mention in letter text.

Actions - Notify the appropriate laboratory contact of required resubmittals when discrepancies are noted on the forms discussed above.

1.3 Technical Evaluation Summary

All data evaluations must be conducted in accordance with current and applicable USEPA Regional protocols and/or specific client contractual requirements and obligations. The applicable documents must be referenced to during the data evaluation process as this Standard Operating Procedure (S.O.P) is intended as proprietary in-house guidance for general inorganic validation practices only.

General parameters such as Data Completeness, Overall System Performance, and Detection Limits must be evaluated concurrently with the parameters discussed below.

1.3.1 Holding Times

Holding times are calculated from date of sample collection to date of sample analysis. The date of sample collection must be obtained from the Chain-of-Custody (COC) form. The date of sample analysis is best retrieved from the raw data but may also be obtained from the Form 14.

Sample preservation and holding time requirements are as follows:

Metals - 6 months; pH <2
Mercury - 28 days; pH <2
Cyanide - 14 days; pH >12

Preservation requirements as noted above are applicable to aqueous samples only. Solid samples do not receive preservative but require maintenance at 4 C (2 C) during shipment and storage.

Subject DATA VALIDATION - NON-CLP INORGANICS OR SOLID AND AQUEOUS MATRICES	Number DV-04	Page 4 of 8
	Revision 0	Effective Date 08/13/01

The above holding times do not apply to leachate analyses. It is suggested that the data reviewer reference SW-846 Method 1311 for any questions regarding TCLP quality control requirements and analytical procedural requirements; these vary significantly from non-TCLP analyses.

Actions - Holding time exceedances result in potentially low-biased results; thus, positive results and nondetects shall be qualified as estimated, (J) and (UJ), respectively. **NOTE:** Gross holding time noncompliances are defined as holding times which are exceeded by a factor or 2X. In these extreme cases, it is practice to reject (R) nondetects while positive results are qualified based upon professional judgment regarding the reliability of the associated data.

1.3.2 Initial Calibration Requirements

Calibration must be initiated daily and prior to sample analysis. The following calibration standard requirements must be verified:

- **ICP analyses** - must employ a blank and at least one standard
- **GFAA analyses** - must employ a blank and at least three standards. Additionally, the calibration correlation coefficient (r) must be checked for linearity for each GFAA analysis performed (i.e. $r = 0.995$ or greater)
- **Mercury analyses** - must employ a blank and at least three standards ($r = 0.995$ or greater).
- **Cyanide analyses** - must employ a blank and at least three standards ($r = 0.995$ or greater). **NOTE:** At least two additional standards (a high or low) must be distilled and compared to similar values on the curve. Values of distilled standards should agree within 10% of undistilled standards.

1.3.3 Initial and Continuing Calibration Verification (ICV/CCV)

The ICV/CCV %R quality control limits are 90-110% for ICP metals, 80-120% for GFAA metals and mercury, and 85-115% for cyanide.

Actions - If ICV/CCV %Rs are low, qualify as estimated, (J) positive results and (UJ) nondetects. If ICV/CCV %Rs are high, qualify as estimated (J) positive results; nondetects remain unaffected. **NOTE:** Qualify results of only those samples associated with the noncompliant ICV or CCV (generally, those samples immediately preceding or following the noncompliant standard until the nearest in-control standard).

1.3.4 Laboratory Method and Field Quality Control Blanks

Verify that a preparation blank was analyzed for each matrix and for each batch of 20 samples or each sample batch digested, whichever is more frequent. Continuing Calibration Blanks (CCBs) must be run at a frequency of 10% or every 2 hours which ever is more frequent.

The data reviewer will select the maximum contaminant level for each analyte in a particular matrix from which shall be calculated an "action level." The action level shall be established as 5X the maximum contaminant level but must be adjusted for dilution factor, moisture content, and sample weight prior to application.

ICB/CCB contamination shall be applied to all samples within an SDG. Preparation blank contamination shall be applied to samples of the same matrix only. Common practice shall be to qualify as nondetected (U) any contaminant present in a sample which is considered a laboratory artifact (i.e., < the established action level). Professional judgment must be employed when discerning the validity of a concentration

Subject DATA VALIDATION - NON-CLP INORGANICS OR SOLID AND AQUEOUS MATRICES	Number DV-04	Page 5 of 8
	Revision 0	Effective Date 08/13/01

present in a field quality control blank. In many instances, contamination present in these blanks can be attributable to "dirty" laboratory practice and not actual field contaminant conditions.

Negative concentrations detected in the laboratory method blanks are indicative of instrumental problems and base-line drifting. Generally, any negative concentration > IDL shall warrant estimation [(J) positives and (UJ) nondetects] of the associated sample data regardless of matrix. Action levels shall not be established for negative concentration levels.

Actions - Qualify as nondetected (U) any positive result within the action level. Qualify as estimated (J) positive results and (UJ) nondetects for analytes for which negative concentrations were noted in the laboratory method blanks (i.e., ICBs, CCBs, and/or preparation blanks).

1.3.5 ICP Interference Check Sample Results

Verify that all recoveries for the ICP ICS solution fall within the 80-120% quality control window established for the ICS AB solution.

Actions - For ICS %Rs <80%, qualify as estimated (J) positive results and (UJ) nondetects in affected samples. For ICS %Rs >120%, qualify as estimated (J) positive results in affected samples; nondetects are unaffected by high ICS solution AB recovery. **NOTE:** Affected samples include all samples analyzed between the initial and final solutions or within the eight hour working shift whichever occurs more frequently) which contain Al, Ca, Fe, or Mg at levels >50% of the respective concentration of Al, Ca, Fe, or Mg in the ICS True Solution A.

Next, review concentrations of the four common interfering analytes (aluminum, calcium, iron, and magnesium) in the environmental samples. Any aforementioned interferant present in the environmental samples at concentrations which exceed those present in the ICS solution for that same analyte will require calculation of estimated elemental interference stemming from high interfering analyte concentration. If the previous condition is met; review the ICP/ICS Form 4 or equivalent and note any analytes present in the ICS solution A at levels which exceed the IDL and which are not present in the ICS True solution A. Positive results in the ICS solution A indicate potentially elevated results for this analyte in the affected sample, while negative results in the ICS solution A indicate potentially suppressed results for this analyte in the affected sample.

Next, an estimated elemental interference must be calculated for each analyte > IDL present in the ICS solution A which is not present in the ICS True solution A. The following equation shall be employed:

$$\text{Estimated elemental intf.} = \frac{[\text{Conc. affected analyte in ICS Soln A}] \times [\text{Interferent}] [\text{Conc. Sample}]}{\text{Interferent Conc. in ICS Soln A}}$$

It is advisable, although not necessary, to routinely choose the lowest concentration for the interferant level in the ICS so as to calculate the highest estimated interference possible. This method lends itself to a more conservative overall data quality review.

Estimated interferences for each affected analyte > IDL in the ICSA solution must now be compared to the reported environmental sample result for that particular analyte.

Actions - For estimated interferences <10% of the reported sample concentration for a particular affected analyte, take no action; interference is considered negligible. For estimated interferences >10% of the reported sample concentration for a particular affected analyte, qualify (J) positive result and/or (UJ) nondetect for affected analyte in affected sample. (**NOTE:** Calculation of an estimated positive (potentially elevated) interference will have no effect on a reported nondetect; thus, no action is necessary).

Subject DATA VALIDATION - NON-CLP INORGANICS OR SOLID AND AQUEOUS MATRICES	Number DV-04	Page 6 of 8
	Revision 0	Effective Date 08/13/01

1.3.6 Matrix Spike Sample Analysis (Pre-digestion)

Verify that at least one matrix spike was performed for each matrix for a given set of samples (maximum of 20 samples) within an SDG. **NOTE:** Filtered and unfiltered samples are to be treated as distinctly different sample matrices and qualified accordingly. Any deviations from the referenced method shall be noted and require laboratory contact for correction.

Aqueous and soil Matrix Spike (MS) recoveries must be within the 75-125% quality control window in instances where the initial sample result is <4X amount spiked. If the initial sample result is >4X the amount spiked and the MS %R is noncompliant, no actions shall be taken.

Actions - For MS %Rs <30%, qualify as estimated (J) positive results and reject (R) nondetects in affected samples. For MS %Rs <75% but >30%, qualify as estimated (J) positive results and (UJ) nondetects in affected samples. For MS %Rs >125%, qualify as estimated (J) positive results in affected samples; nondetects are not compromised by high MS recovery; thus, no actions are warranted.

1.3.7 Laboratory Duplicate Precision

Verify that one duplicate sample analysis was performed for each group of samples (maximum of 20 samples) of a similar matrix within an SDG. Control criteria used to evaluate the aqueous laboratory duplicates are as follows:

- a control limit of 20% for relative percent difference when sample and duplicate results are >5X CRDL
- a control limit of 1X CRDL for the difference between the sample values when sample and/or duplicate results are <5X CRDL

Control criteria used to evaluate solid laboratory duplicates are as follows:

- a control limit of 35% for relative percent difference when sample and duplicate results are >5X CRDL
- a control limit of 2X CRDL for the difference between the sample values when sample and/or duplicate results are <5X CRDL

NOTE: Review Duplicate Summary (Form 6 or equivalent) carefully and verify that the laboratory has in fact reported a %RPD of 200% and not simply recorded the %RPD as noncalculable (in instances where the sample result is positive but the duplicate result is nondetect). Overlooking this minor point may result in incomplete sample data qualification in some instances.

Actions - For any situation involving laboratory duplicate imprecision, qualify as estimated (J) positive results and (UJ) nondetects in affected samples. **NOTE:** It is important to note in the letter text the cause of laboratory duplicate imprecision (i.e., noncompliant %RPD or noncompliant difference between sample and duplicate results).

1.3.8 Field Duplicate Precision

Field duplicates can be determined via Project Manager informational documents (i.e., sampling logs) or obtained from Chain-of-Custody (COC) forms. Field duplicates are generally identified as samples having identical sample collection times and dates. In instances where field duplicate samples are included with the sample data set, the following control criteria are generally used to evaluate aqueous field duplicates:

Subject DATA VALIDATION - NON-CLP INORGANICS OR SOLID AND AQUEOUS MATRICES	Number DV-04	Page 7 of 8
	Revision 0	Effective Date 08/13/01

- a control limit of 30% for relative percent difference when sample and duplicate results are >5X CRDL
- a control limit of 2X CRDL for the difference between the sample values when sample and/or duplicate results are <5X CRDL

Similarly, the following control criteria are generally used to evaluate solid field duplicates:

- a control limit of 50% for relative percent difference when sample and duplicate results are >5X CRDL
- a control limit of 4X CRDL for the difference between the sample values when sample and/or duplicate results are <5X CRDL

NOTE: The %RPD should reflect a difference of 200% and should not simply be recorded as noncalculable in instances where the sample result is positive but the field duplicate result is nondetect. Overlooking this minor point may result in incomplete sample data qualification in some instances.

Actions - For any situation involving field duplicate imprecision, qualify as estimated (J) positive results and (UJ) nondetects in affected samples. **NOTE:** It is important to note in the letter text the cause of field duplicate imprecision (i.e., noncompliant %RPD or noncompliant difference between sample and duplicate results). Furthermore, field duplicate data qualifications, as per Brown & Root Environmental convention, shall be matrix-specific but otherwise "across-the-board" for TAL inorganic analyses.

1.3.9 Laboratory Control Sample Results

Verify that an LCS was analyzed for each matrix and for each batch of twenty samples or batch of samples digested (whichever is more frequent) within an SDG. The quality control criteria established for evaluation of aqueous LCS analyses are 80-120%. **NOTE:** An aqueous LCS is not required for mercury and cyanide analysis. Verify that all solid "found values" fall within the EPA established control limits for soils.

Actions - Aqueous LCS: In instances where aqueous LCS %R <80%, qualify as estimated (J) positive results and (UJ) nondetects, If aqueous LCS %R >120, qualify as estimated (J) positive results. Solid LCS: In instances where solid found value is below lower quality control limit, qualify as estimated (J) positive results and (UJ) nondetects. If solid LCS found value exceeds EPA upper limit for soils, qualify as estimated (J) positive results.

1.3.10 Method of Standard Additions (MSA)

Review MSA Form 8 or equivalent and verify instrument linearity by checking that all calibration correlation coefficients (r) are greater than or equal to 0.995. MSAs for a particular analyte in a particular sample may be run more than once. Check reanalyses in instances where initial MSA analysis yields (r) <0.995. It is good practice to review one or two GFAA post-digestion spike (PDS) %Rs via reviewing unspiked and spiked sample concentrations and associated PDS recovery to verify that the Furnace Atomic Absorption Analysis Scheme has been followed as per directional guidance in the method.

Actions - If calibration correlation coefficient (r) <0.995, qualify as estimated (J) positive result and/ or (UJ) nondetect in affected sample.

Subject DATA VALIDATION - NON-CLP INORGANICS OR SOLID AND AQUEOUS MATRICES	Number DV-04	Page 8 of 8
	Revision 0	Effective Date 08/13/01

1.3.11 ICP Serial Dilution Analysis

Verify that all ICP analytes are included on the Form 9 (or equivalent) with corresponding recovery calculations. Check the calculated Percent Difference (%D) column in instances where the diluted sample result is nondetected. In this situation, the laboratory should report a %D of 100% and not simply list the %D as noncalculable. Overlooking this minor point may result in incomplete sample data qualification in some instances. Amend the Form 9 if necessary. All %Ds for ICP serial dilution analyses should be <10% when concentrations of corresponding analytes in the original (undiluted) sample are minimally a factor of 50X IDL.

Actions - If %D >10% for an analyte, and the corresponding sample concentration is >50 IDL, qualify as estimated (J) positive results for that analyte in all samples of the same matrix. NOTE: The possibility of suppressed results exists when the ICP serial dilution %D >10% and the diluted sample result is significantly > original (undiluted) sample result. Qualify as estimated (J) positive results and (UJ) nondetects in such instances.

1.3.12 Analysis Run Logs Form 14

The Form 14 or equivalent serves several useful functions. It can be used to obtain sample analysis dates as noted in the heading of the page. Secondly, it is used to record any dilutions as applicable to ICP, GFAA, mercury, and cyanide analyses. And finally, it can be used to verify GFAA PDS percent recoveries within the 85-115% quality control limits. Additionally, the data reviewer should be careful to note that one and only one "X" flag has been used to indicate each reported sample result or quality control sample result; this can be an area of frequent laboratory error.

Actions - If the PDS %R is <85%, qualify as estimated (J) the corresponding positive result and/or (UJ) nondetect in affected sample. If the PDS %R is >115%, qualify as estimated (J) the corresponding positive result in the affected sample; nondetects are not qualified based on high PDS % R.

1.3.13 Further GFAA Evaluations

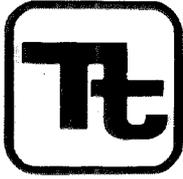
It is necessary to review the raw data for GFAA analyses and verify that all Coefficients of Variation Relative Standard Deviations (%RSDs) are <20% for reported sample results which exceed the CRDL.

Actions - If the CV or %RSD exceeds 20% and the reported sample result is > CRDL, qualify as estimated (J) positive result in affected sample.

1.4 Deliverables Guidance

In addition to any specific USEPA Regional requirements (e.g., data validation memorandum, data summary spreadsheets, USEPA Regional worksheets), all laboratory data package quality control summary forms, sample Form I reports, method blank Form Is, and the Chain-of-Custody report must be given to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct (in accordance with the appropriate USEPA Regional or client requirements) and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.



TETRA TECH NUS, INC.

STANDARD OPERATING PROCEDURES

Number DV-08	Page 1 of 24
Effective Date 08/13/01	Revision 5
Applicability Tetra Tech NUS, Inc.	
Prepared Risk Assessment Department	
Approved D. Senovich	

Subject
DATA VALIDATION - MISCELLANEOUS INORGANICS

TABLE OF CONTENTS

<u>SECTION</u>	<u>PAGE</u>
1.0 CARBONATE/BICARBONATE ALKALINITY (EPA 600 SERIES METHOD 310.2)	4
1.1 APPLICABILITY.....	4
1.2 INTERFERENCES.....	4
1.3 HOLDING TIMES.....	4
1.4 QUALITY CONTROL.....	4
1.4.1 Calibration.....	4
1.4.2 Blanks.....	4
1.4.3 Spikes.....	5
1.4.4 Duplicates.....	5
1.4.5 Sample Quantitation.....	5
1.5 DELIVERABLES GUIDANCE.....	5
2.0 ANIONS (EPA METHOD 300.0)	5
2.1 APPLICABILITY.....	5
2.2 INTERFERENCES.....	6
2.3 GENERAL LABORATORY PRACTICES.....	6
2.4 HOLDING TIMES.....	7
2.5 SAMPLE PREPARATION.....	7
2.6 CALIBRATION AND TESTING.....	7
2.7 BLANK CONTAMINATION.....	8
2.8 SAMPLE QUANTITATION.....	8
2.9 DELIVERABLES GUIDANCE.....	8
3.0 BROMIDE (EPA 600 SERIES METHOD 320.1)	8
3.1 APPLICABILITY.....	8
3.2 INTERFERENCES.....	9
3.3 HOLDING TIMES.....	9
3.4 QUALITY CONTROL.....	9
3.4.1 Verification Standard.....	9
3.4.2 Blanks.....	9
3.4.3 Spikes.....	9
3.4.4 Duplicates.....	10
3.4.5 Sample Quantitation.....	10
3.5 DELIVERABLES GUIDANCE.....	10
4.0 FLUORIDE (EPA 600 SERIES METHOD 340.2)	10
4.1 APPLICABILITY.....	10
4.2 INTERFERENCES.....	10
4.3 HOLDING TIMES.....	10

Subject DATA VALIDATION - MISCELLANEOUS INORGANICS	Number DV-09	Page 2 of 24
	Revision 0	Effective Date 08/13/01

TABLE OF CONTENTS (Continued)

<u>SECTION</u>	<u>PAGE</u>
4.4	QUALITY CONTROL.....11
4.4.1	Calibration.....11
4.4.2	Blanks.....11
4.4.3	Spikes.....11
4.4.4	Duplicates.....11
4.4.5	Sample Quantitation.....12
4.5	DELIVERABLES GUIDANCE.....12
5.0	NITROGEN (VARIOUS).....12
5.1	NITRATE-NITROGEN (EPA 300 SERIES METHOD 352.1).....12
5.1.1	Applicability.....12
5.1.2	Interferences.....12
5.1.3	Holding Times.....12
5.1.4	Quality Control.....13
5.1.5	Deliverables Guidance.....14
5.2	NITRATE-NITRITE OR NITRITE (EPA 300 SERIES METHOD 353.2).....14
5.2.1	Applicability.....14
5.2.2	Interferences.....14
5.2.3	Holding Times.....14
5.2.4	Quality Control.....14
5.2.5	Deliverables Guidance.....15
6.0	PHOSPHORUS (EPA 600 SERIES METHOD 365.4).....16
6.1	APPLICABILITY.....16
6.2	INTERFERENCES.....16
6.3	HOLDING TIMES.....16
6.4	QUALITY CONTROL.....16
6.4.1	Calibration.....16
6.4.2	Blanks.....16
6.4.3	Spikes.....16
6.4.4	Duplicates.....17
6.4.5	Sample Quantitation.....17
6.5	DELIVERABLES GUIDANCE.....17
7.0	SULFATE (EPA 600 SERIES METHOD 375.4).....17
7.1	APPLICABILITY.....17
7.2	INTERFERENCES.....17
7.3	HOLDING TIMES.....17
7.4	QUALITY CONTROL.....18
7.4.1	Calibration.....18
7.4.2	Blanks.....18
7.4.3	Spikes/Duplicates.....18
7.4.4	Sample Quantitation.....19
7.5	DELIVERABLES GUIDANCE.....19
8.0	SULFIDES (EPA SW-846 METHOD 9030).....19
8.1	APPLICABILITY.....19
8.2	INTERFERENCES.....19
8.3	HOLDING TIMES.....20

Subject DATA VALIDATION - MISCELLANEOUS INORGANICS	Number DV-09	Page 3 of 24
	Revision 0	Effective Date 08/13/01

TABLE OF CONTENTS (Continued)

<u>SECTION</u>	<u>PAGE</u>
8.4	QUALITY CONTROL.....20
8.4.1	Calibration.....20
8.4.2	Blanks.....20
8.4.3	Spikes/Duplicates.....20
8.4.4	Sample Quantitation.....21
8.5	DELIVERABLES GUIDANCE.....21
9.0	TOTAL SUSPENDED SOLIDS (EPA 600 SERIES METHOD 160.2)21
9.1	APPLICABILITY.....21
9.2	INTERFERENCES.....21
9.3	HOLDING TIMES.....22
9.4	QUALITY CONTROL.....22
9.4.1	Verification.....22
9.4.2	Blanks.....22
9.4.3	Duplicates.....22
9.4.4	Sample Quantitation.....22
9.5	DELIVERABLES GUIDANCE.....23
10.0	TOTAL DISSOLVED SOLIDS (EPA 600 SERIES METHOD 160.1)23
10.1	APPLICABILITY.....23
10.2	INTERFERENCES.....23
10.3	HOLDING TIMES.....23
10.4	QUALITY CONTROL.....23
10.4.1	VERIFICATION.....23
10.4.2	Blanks.....24
10.4.3	Duplicates.....24
10.4.4	Sample Quantitation.....24
10.5	DELIVERABLES GUIDANCE.....24

Subject DATA VALIDATION - MISCELLANEOUS INORGANICS	Number DV-09	Page 4 of 24
	Revision 0	Effective Date 08/13/01

1.0 CARBONATE/BICARBONATE ALKALINITY (EPA 600 SERIES METHOD 310.2)

1.1 Applicability

Method 310.2 is an automated method used to measure alkalinity (as CaCO₃) at concentrations ranging from 10 to 200 mg/L in domestic and industrial effluents, and drinking, surface and saline waters.

1.2 Interferences

Since the method of analysis is colorimetric, primary interferences for this method include turbidity and color. Samples can be filtered prior to analysis to reduce interferences from turbidity.

1.3 Holding Times

Samples should be collected in plastic or glass containers and cooled to 4 °C. No preservative is needed.

Holding time is defined as the elapsed time period from sample collection to analysis. The holding time for this method is 14 days. Chain of Custodies (COCs) and raw data are reviewed to determine if holding times were met for all samples. Positive results and nondetects will be qualified as estimated, (J) and (UJ), respectively, if holding times were exceeded. Gross holding time violations (>2X holding time) will warrant rejection, (R), of nondetects.

1.4 Quality Control

Quality control analyses and criteria (i.e., calibrations, blanks, spikes, etc.) are not specified in Method 310.2. However, if these analyses were performed by the laboratory, the following criteria will be used to evaluate the associated sample data.

1.4.1 Calibration

According to the method, a calibration curve should be prepared by plotting peak heights of standards to known concentrations. This curve should be checked for linearity. Generally, associated sample data are qualified as estimated, (J) and (UJ), if the calibration curve correlation coefficient is <0.995. Professional judgment should be used to qualify sample data in cases when sample results fall outside the linear portion of the calibration curve.

If analyzed, the percent recovery (%R) of a continuing calibration verification (CCV) standard will be evaluated using an 85-115% quality control range. Associated sample data will be qualified as estimated, (J) and (UJ), if the CCV %R is <85%. Only positive results in the affected samples will be qualified as estimated, (J), if the CCV %R is >115%.

1.4.2 Blanks

Laboratory method and field quality control blanks, if analyzed, should be identified and assessed for introduced contamination. Field quality control blanks (field, rinsate, equipment, etc.) can be identified by consulting the COCs. If contamination is noted in the associated blanks, positive sample results < the maximum amount detected in the blanks will be qualified as undetected, (U). Sample digestion and moisture content factors will be taken into consideration when qualifying the associated sample data.

Subject DATA VALIDATION - MISCELLANEOUS INORGANICS	Number DV-09	Page 5 of 24
	Revision 0	Effective Date 08/13/01

1.4.3 Spikes

If a spiked sample is analyzed, a 75-125% quality control range will be used to evaluate the spike %R. Associated sample data will be qualified as estimated, (J) and (UJ), when the spiked sample %R is <75%. When the %R is >125%, only positive results are impacted and qualified as estimated, (J). If a spike %R is <30%, associated nondetects will be qualified as rejected, (R), and positive results will be qualified as estimated, (J).

1.4.4 Duplicates

Qualification of sample data based on duplicate precision is left to the professional judgment of the validator. Generally, an aqueous quality control limit of 20% and a solid quality control limit of 30% is used to evaluate the relative percent difference (RPD) between the sample and laboratory duplicate results. An aqueous quality control limit of 30% and a solid quality control limit of 50% are generally used to evaluate the RPD between field duplicate results; qualification based on field duplicate imprecision for general chemistry parameters is applied to the field duplicate pair only.

1.4.5 Sample Quantitation

All reported sample concentrations should fall within the range of the calibration curve. If samples having detected concentrations > the highest calibration standard were not diluted and reanalyzed, associated sample data will be qualified as estimated, (J).

The validator should verify that sample results were properly quantitated.

1.5 Deliverables Guidance

The content and format of the data package generated for alkalinity analysis may vary significantly depending upon the work request.

In addition to any work-request requirements (e.g., data validation memorandum), all laboratory data package quality control summary forms, laboratory summaries of sample results and laboratory method blanks, and COCs must be provided to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

2.0 ANIONS (EPA METHOD 300.0

The Determination of Inorganic Anions in Water by Ion Chromatography

2.1 Applicability

Method 300.0 is a Ion Chromatographic (IC) Procedure used to determine the inorganic anions chloride, fluoride, nitrate (as nitrogen), nitrite (as nitrogen), ortho-phosphate (as phosphorus), and sulfate in drinking water, surface water, and mixed domestic and industrial wastewater.

Subject DATA VALIDATION - MISCELLANEOUS INORGANICS	Number DV-09	Page 6 of 24
	Revision 0	Effective Date 08/13/01

2.2 Interferences

Interferences may be caused by particulates or other substances present in the sample that may have retention times similar to the particular anion of interest. Also, a large concentration of one anion may mask the resolution of an adjacent anion. Sample dilution and/or spiking (to generate a sample-specific calibration) may be employed to resolve these problems. Additionally, method interferences may be caused by contaminants in reagent water, reagents, glassware, and other elements of sample processing.

The fluoride peak, in particular, may be affected by a water dip (a negative peak) that elutes near it. This problem can be eliminated by the addition of 1 mL of concentrated sodium carbonate eluent solution to 100 mL of each standard and sample.

2.3 General Laboratory Practices

The laboratory should spike and analyze a minimum of 10% of all samples to monitor continuing laboratory performance. Field and laboratory duplicates should also be analyzed.

Validation: The validator should check the work request to ascertain what contracted quality control analyses are required. Likewise, the validator should check with the project manager to determine which samples (if any) are field duplicates or field quality control blanks.

Before any analyses are performed the laboratory must demonstrate the ability to generate acceptable accuracy and precision using a blank spike sample (laboratory control sample; LCS), which is a reagent water blank spiked with a known concentration of stock standard solutions at the concentrations stipulated in EPA Method 300.0, Sections 8.2.2 through 8.3.1.

Analysis of this blank spike sample will indicate the accuracy of the measurement via the calculation of Percent Recovery (%R). Upper and lower control limits for %Rs should be calculated. These control limits can then be used to construct control charts that may be useful in observing trends in performance. This blank spike sample should also be duplicated and analyzed to indicate precision of the measurements between identical samples through comparison of the recoveries generated via the blank spike and blank spike duplicate analyses. The blank spike/blank spike duplicate analyses should be performed with the same frequency as matrix spike/matrix spike duplicate analyses.

Validation: The data reviewer shall examine The %Rs to determine if they are within the laboratory generated control limits. If %Rs are below the control limits positive results will be qualified (J) and nondetected results will be qualified (UJ). If %Rs are above the control limits only positive results will be qualified (J). If %Rs are extremely low (less than 10%) the laboratory should reanalyze the blank spike and blank spike duplicate samples. If the laboratory does not reanalyze these samples then qualifications are necessary. Positive results will be qualified as estimated (J) and nondetects will be rejected (R), when %Rs are less than 10%.

The reviewer should also examine the Relative Percent Difference (RPD) between the calculated %Rs. If the RPD is above an acceptable level qualify positive results (J) and use professional judgment to determine if nondetects should be qualified (UJ).

Subject DATA VALIDATION - MISCELLANEOUS INORGANICS	Number DV-09	Page 7 of 24
	Revision 0	Effective Date 08/13/01

2.4 Holding Times

The following table indicates sample preservation and holding time requirements:

Analyte	Preservation	Holding time
Chloride	None required	28 days
Fluoride	None require	28 days
Nitrate-N	Cool to 4°C	48 hours
Nitrite-N	Cool to 4°C	48 hours
o-Phosphate-P	Filter & cool to 4°C	48 hours
Sulfate	Cool to 4°C	28 days

Validation: Holding times are calculated from time of collection obtained from Chain-of-Custody (COC) forms to time of analysis. Positive results in samples analyzed past holding times are qualified as estimated (J); nondetects (UJ). If holding times are exceeded by a factor of 2 or more it is considered to be a gross exceedance; positive results are qualified as estimated, (J), and nondetects are rejected, (R). Results are considered to be biased low when holding times are exceeded.

2.5 Sample Preparation

Samples containing particles greater than 0.45 microns and reagent solutions containing particles greater than 0.20 microns require filtration to prevent damage to the instrument columns and flow systems.

2.6 Calibration and Testing

Per each analyte of interest, calibration standards at a minimum of 3 concentration levels should be prepared (generated from a stock solution and diluted appropriately) and analyzed along with a blank. One of the standard concentrations must be near but above the MDL. A sufficient number of standards should be analyzed to accurately define a calibration curve.

A consistent aliquot (injections of 0.1 to 1.0 mL) for samples and standards must be used. An automated constant volume injection system may be employed.

Calibration for each analyte should be verified daily, or whenever the anion eluent is changed, and after every 20 samples. Retention times must agree within 10%. If agreement is not met a new calibration curve should be generated for that analyte.

Validation: The validator will evaluate the 3-point calibration and verify that one of the points was at a concentration near the MDL. Next, the retention times will be examined to ensure that they agree within $\pm 10\%$. If the retention time is outside the 10% window, the result for the affected analyte will be qualified as estimated; (J) positive results and (UJ) nondetects.

If peak response exceeds the linear calibration range of the instrument, the sample should be diluted with the appropriate amount of reagent water and reanalyzed. If the chromatogram does not produce adequate resolution or if identification of the chromatographic peaks are questionable, the sample should be spiked with the appropriate amount of standard and reanalyzed.

Validation: The validator will review chromatograms to verify the absence of a water dip (see Section 8.2.2) and to verify that peak responses are within the linear range and that adequate resolution was achieved. If any noncompliances exist they should be noted. Qualifications will be made per situation, based upon professional judgment.

Subject DATA VALIDATION - MISCELLANEOUS INORGANICS	Number DV-09	Page 8 of 24
	Revision 0	Effective Date 08/13/01

2.7 Blank Contamination

Method blanks (reagent water) should be analyzed at the beginning of each sample batch (maximum of 20 samples) to ensure that there is no carryover or contamination from glassware and/or reagents.

Validation: Blank results should be reported for each sample data set. If contamination is noted in the blanks, the maximum concentration of each contaminant should be used to set action. Action levels are set using professional judgment based upon comparability of the sample result with concentration of the blank contaminant. Results reported for contaminants found in samples that are greater than the detection limit and within the action level are qualified as undetected, (U). The same process is repeated for field quality control blanks.

2.8 Sample Quantitation

A standard curve should be generated by plotting anion peak size in area units against standard anion solution concentration values. Sample concentration can then be calculated by comparing sample peak response with the standard curve. Sample data results should be reported in mg/L.

Validation: The validator shall compare sample results against standard results to confirm that the samples were properly quantitated.

2.9 Deliverables Guidance

The content and format of the data package generated for anion analysis may vary significantly dependent upon the work request.

In addition to any work-request requirements (e.g., data validation memorandum, data spread sheet), all laboratory data package quality control summary forms, laboratory summaries of sample data results and method blank analyses and the chain-of-custody report must be given to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

3.0 **BROMIDE (EPA 600 SERIES METHOD 320.1)**

3.1 Applicability

Method 320.1 is a titrimetric method used to determine the concentration of bromide in domestic and industrial effluents, and drinking, surface and saline waters. Bromide concentrations ranging from 2 to 20 mg/L can be measured by this method.

3.2 Interferences

Interferences can be caused by the presence of organic matter, iron, and manganese. Pretreatment of samples with calcium oxide removes or reduces these interferences to insignificant concentrations.

Color interferes with the observation of indicator and bromine-water color changes. Steps can be taken during analysis to eliminate this interference (e.g., the use of a pH meter instead of a pH indicator).

Subject DATA VALIDATION - MISCELLANEOUS INORGANICS	Number DV-09	Page 9 of 24
	Revision 0	Effective Date 08/13/01

3.3 Holding Times

Samples should be collected in plastic or glass bottles and cooled to 4 C. No preservative is needed.

Holding time is defined as the elapsed time period from sample collection to analysis. A 28-day holding time is specified for analysis. Chain of Custodies (COCs) and raw data are reviewed to determine if holding times were met for all samples. Positive results and nondetects will be qualified as estimated, (J) and (UJ), respectively, if holding times were exceeded. Gross holding time violations (>2X holding time) will warrant rejection, (R), of nondetects.

3.4 Quality Control

Quality control analyses (i.e., blanks, spikes, etc.) are not specified in Method 320.1. However, if these analyses were performed by the laboratory, the following criteria will be used to evaluate the associated sample data.

3.4.1 Verification Standard

The percent recovery (%R) of a verification standard, if analyzed, will be evaluated using an 85-115% quality control criteria. Associated sample data will be qualified as estimated, (J) and (UJ), if the %R is <85%. Only positive results in the affected samples will be qualified as estimated, (J), if the %R is >115%.

3.4.2 Blanks

Laboratory method and field quality control blanks, if analyzed, should be evaluated for contamination. Field quality control blanks (field, rinsate, equipment, etc.) can be identified by consulting the COCs. If contamination is noted in the associated blanks, positive sample results < the maximum amount detected in the blanks will be qualified as undetected, (U). Sample digestion and moisture content factors will be taken into consideration when qualifying the associated sample data.

3.4.3 Spikes

If a spiked sample is analyzed, a 75-125% quality control range will be used to evaluate the spike %R. Associated sample data will be qualified as estimated, (J) and (UJ), when the spiked sample %R is <75%. When the %R is >125%, only positive results are impacted and qualified as estimated, (J). If a spike %R is <30%, associated nondetects will be qualified as rejected, (R), and positive results will be qualified as estimated, (J).

3.4.4 Duplicates

Qualification of sample data based on duplicate precision is left to the professional judgment of the validator. Generally, an aqueous quality control limit of 20% and a solid quality control limit of 30% is used to evaluate the relative percent difference (RPD) between the sample and laboratory duplicate results. An aqueous quality control limit of 30% and a solid quality control limit of 50% are generally used to evaluate the RPD between field duplicate results; qualification based on field duplicate imprecision for general chemistry parameters is applied to the field duplicate pair only.

3.4.5 Sample Quantitation

All reported sample concentrations should fall within the range of 2 to 20 mg/L. If samples having detected concentrations >20 mg/L were not diluted and reanalyzed, the associated sample data will be qualified as estimated, (J).

Subject DATA VALIDATION - MISCELLANEOUS INORGANICS	Number DV-09	Page 10 of 24
	Revision 0	Effective Date 08/13/01

The validator should verify that sample results were properly quantitated.

3.5 Deliverables Guidance

The content and format of the data package generated for bromide analysis may vary significantly depending upon the work request.

In addition to any work-request requirements (e.g., data validation memorandum), all laboratory data package quality control summary forms, laboratory summaries of sample results and laboratory method blanks, and COCs must be provided to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

4.0 FLUORIDE (EPA 600 SERIES METHOD 340.2)

4.1 Applicability

Method 340.2 is a potentiometric method which uses an ion selective electrode to measure concentrations of fluoride in domestic and industrial effluents, and drinking, surface and saline waters. The practical range of determination is 0.1 to 1,000 mg/L.

4.2 Interferences

The pH of samples can cause significant interferences. The ideal pH range of a sample is between 5 and 9.

Complexing cations, such as Si^{+4} , Fe^{+3} , and Al^{+3} , can produce additional interferences during fluoride determinations. Samples can be treated with a pH 5.0 buffer containing a strong chelating agent to eliminate these interferences.

4.3 Holding Times

Samples are to be collected in plastic bottles. No preservative is required.

A 28-day holding time (elapsed time period from sample collection to analysis) is specified for analysis. Chain of Custodies (COCs) and raw data are reviewed to determine if holding times were met for all samples. Sample data will be qualified as estimated, (J) and (UJ), if holding times were exceeded. Gross holding time violations (>2X holding time) will warrant rejection, (R), of nondetects.

4.4 Quality Control

Quality control analyses (i.e., calibrations, blanks, spikes, etc.) are not specified in Method 340.2. However, if these analyses were performed by the laboratory, the following criteria will be used to evaluate the associated sample data.

Subject DATA VALIDATION - MISCELLANEOUS INORGANICS	Number DV-09	Page 11 of 24
	Revision 0	Effective Date 08/13/01

4.4.1 Calibration

According to the method, the calibration curve should consist of standards ranging in concentration from 0 to 2 mg/L. Semi-logarithmic graph paper should be used to plot the known concentration of the standard versus the electrode potential.

If a continuing calibration verification (CCV) standard is analyzed, the percent recovery of the standard will be evaluated using 85-115% quality control limits. Associated sample data will be qualified as estimated, (J) and (UJ), if the CCV %R is <85%. Only positive results in the affected samples will be qualified as estimated, (J), if the CCV %R is >115%.

4.4.2 Blanks

Laboratory method and field quality control blanks, if analyzed, should be assessed for introduced contamination. Field quality control blanks (field, rinsate, equipment, etc.) can be identified by consulting the COCs. If contamination is noted in the associated blanks, positive sample results < the maximum amount detected in the blanks will be qualified as undetected, (U). Sample digestion and moisture content factors will be taken into consideration when qualifying the associated sample data.

4.4.3 Spikes

If a spiked sample is analyzed, a 75-125% quality control range will be used to evaluate the spike %R. Associated sample data will be qualified as estimated, (J) and (UJ), when the spiked sample %R is <75%. If the %R is >125%, only positive results are impacted and qualified as estimated, (J). If a spike %R is <30%, associated nondetects will be qualified as rejected, (R), and positive results will be qualified as estimated, (J).

4.4.4 Duplicates

Qualification of sample data based on duplicate precision is left to the professional judgment of the validator. Generally, an aqueous quality control limit of 20% and a solid quality control limit of 30% is used to evaluate the relative percent difference (RPD) between the sample and laboratory duplicate results. An aqueous quality control limit of 30% and a solid quality control limit of 50% is used to evaluate the RPD between field duplicate results; qualification based on field duplicate imprecision for general chemistry parameters is applied to the field duplicate pair only.

4.4.5 Sample Quantitation

All reported sample concentrations should fall in the range of the calibration curve. If samples having detected concentrations > the highest calibration standard were not diluted and reanalyzed, associated sample data will be qualified as estimated, (J).

The validator should verify that sample results were properly quantitated.

4.5 Deliverables Guidance

The content and format of the data package generated for fluoride analysis may vary significantly depending upon the work request.

In addition to any work-request requirements (e.g., data validation memorandum), all laboratory data package quality control summary forms, laboratory summaries of sample results and laboratory method

Subject DATA VALIDATION - MISCELLANEOUS INORGANICS	Number DV-09	Page 12 of 24
	Revision 0	Effective Date 08/13/01

blanks, and COCs must be provided to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

5.0 NITROGEN (VARIOUS)

5.1 Nitrate-Nitrogen (EPA 300 Series Method 352.1)

5.1.1 Applicability

Method 352.1 is a brucine, colorimetric method used to measure nitrate-nitrogen at concentrations ranging from 0.1 to 2 mg/L in domestic and industrial effluents, and drinking, surface and saline waters.

5.1.2 Interferences

The following is a list of interferences observed for this method:

- Uniform temperature control is extremely critical during the color development stage. Erratic heating can produce inconsistent results.
- Strong oxidizing or reducing agents, residual chloride, ferrous and ferric iron, quadrivalent manganese, and salinity in samples can create interferences.
- Interferences from naturally colored samples and dissolved organic matter can affect color during heating and produce erroneous results.

5.1.3 Holding Times

Samples should be collected in plastic or glass containers and cooled to 4°C.

The holding time, elapsed time period from sample collection to analysis, for this method is 48 hours. Chain of Custodies (COCs) and raw data are reviewed to determine if holding times were met for all samples. Positive results and nondetects will be qualified as estimated, (J) and (UJ), respectively, if holding times were exceeded. Gross holding time violations (>2X holding time) will warrant rejection, (R), of nondetects.

5.1.4 Quality Control

Quality control analyses (i.e., calibrations, blanks, spikes, etc.) are not specified in this method. However, if these analyses were performed by the laboratory, the following criteria will be used to evaluate the associated sample data.

Calibration

A calibration curve should be prepared by plotting absorbances of standards against known concentrations. Because the color reaction does not always obey Beer's Law, qualification of sample data based on nonlinear calibration curves may be inappropriate. Professional judgment should be used to qualify sample data when nonlinearity (calibration curve correlation coefficient <0.995) is encountered.

Subject DATA VALIDATION - MISCELLANEOUS INORGANICS	Number DV-09	Page 13 of 24
	Revision 0	Effective Date 08/13/01

The percent recovery (%R) of a continuing calibration verification (CCV) standard, if analyzed, will be evaluated using an 85-115% quality control criteria. If the %R is <85%, associated sample data will be qualified as estimated, (J) and (UJ). Only positive results in the affected samples will be qualified as estimated, (J), if the CCV %R is >115%.

Blanks

If analyzed, laboratory method and field quality control blanks should be assessed for contamination. Field quality control blanks (field, rinsate, equipment, etc.) can be identified by consulting the COCs. If contamination is noted in the associated blanks, positive sample results < the maximum amount detected in the blanks will be qualified as undetected, "U." Sample digestion and moisture content factors will be taken into consideration when qualifying the associated sample data.

Spikes

A 75-125% quality control range will be used to evaluate %Rs if a spiked sample was analyzed. Associated sample results will be qualified as estimated, (J) and (UJ), when the spiked sample %R is <75%. If the %R is >125%, only positive results are impacted and qualified as estimated, (J). Associated nondetects will be qualified as rejected, (R), and positive results will be qualified as estimated, (J), if the spike %R is <30%.

Duplicates

Qualification of sample data based on duplicate precision is left to the professional judgment of the validator. Generally, an aqueous quality control limit of 20% and a solid quality control limit of 30% is used to evaluate the relative percent difference (RPD) between the sample and laboratory duplicate results. An aqueous quality control limit of 30% and a solid quality control limit of 50% is used to evaluate the RPD between field duplicate results; qualification based on field duplicate imprecision for general parameters is applied to the field duplicate pair only.

Sample Quantitation

All reported sample concentrations should fall within the range of the calibration curve. If samples having detected concentrations > the highest calibration standard were not diluted and reanalyzed, associated sample data will be qualified as estimated, (J).

The validator should verify that sample results were properly quantitated.

5.1.5 Deliverables Guidance

The content and format of the data package generated for this method of analysis may vary significantly depending upon the work request.

In addition to any work-request requirements (e.g., data validation memorandum), all laboratory data package quality control summary forms, laboratory summaries of sample results and laboratory method blanks, and COCs must be provided to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

Subject DATA VALIDATION - MISCELLANEOUS INORGANICS	Number DV-09	Page 14 of 24
	Revision 0	Effective Date 08/13/01

5.2 **Nitrate-Nitrite or Nitrite (EPA 300 Series Method 353.2)**

5.2.1 **Applicability**

Method 353.2 is a cadmium reduction, automated colorimetric method used to determine the concentration of either nitrite or combined nitrate and nitrite in domestic and industrial effluents, and surface and saline waters. The applicable range of this method is 0.05 to 10 mg/L.

5.2.2 **Interferences**

The presence of suspended matter and high concentrations of oil and grease and some metals (i.e., iron, copper) can create interferences with this method. Samples can be filtered before analysis to minimize the problem of restricted sample flow caused by suspended matter. An organic solvent extraction and the addition of EDTA to samples can eliminate interferences from oil and grease and problematic metals, respectively.

5.2.3 **Holding Times**

Samples should be collected in plastic or glass bottles, preserved with sulfuric acid to a pH <2, and cooled to 4°C.

A 28-day holding time (elapsed time period from sample collection to analysis) is specified for analysis. Chain of Custodies (COCs) and raw data are reviewed to determine if holding times were met for all samples. Positive results and nondetects will be qualified as estimated, (J) and (UJ), respectively, if holding times were exceeded. Gross holding time violations (>2X holding time) will warrant rejection, (R), of nondetects.

5.2.4 **Quality Control**

The method does not specify the analysis of quality control measures (i.e., calibrations, blanks, spikes, etc.). However, if these analyses were performed by the laboratory, the following criteria will be used to evaluate the associated sample data.

Calibration

The calibration curve should be checked for linearity (correlation coefficient curve >0.995). In general, associated sample data are qualified as estimated, (J) and (UJ), when calibration curves are not linear. However, professional judgment should be used to qualify sample data when a nonlinear curve is encountered.

If analyzed, the percent recovery (%R) of a continuing calibration verification (CCV) standard will be evaluated using an 85-115% quality control criteria. If the %R is <85%, associated sample data will be qualified as estimated, (J) and (UJ). Only positive results in the affected samples will be qualified as estimated, (J), if the CCV %R is >115%.

Blanks

Laboratory method and field quality control blanks, if analyzed, should be evaluated for contamination. Field quality control blanks (field, rinsate, equipment, etc.) can be identified by consulting the COCs. If contamination is noted in the associated blanks, positive sample results < the maximum amount detected in the blanks will be qualified as undetected, (U). Sample digestion and moisture content factors will be taken into consideration when qualifying the associated sample data.

Subject DATA VALIDATION - MISCELLANEOUS INORGANICS	Number DV-09	Page 15 of 24
	Revision 0	Effective Date 08/13/01

Spikes

If a spiked sample was analyzed, a 75-125% quality control range will be used to evaluate %Rs. Associated sample results will be qualified as estimated, (J) and (UJ), when the spiked sample %R is <75%. If the %R is >125%, only positive results are impacted and qualified as estimated, (J). Associated nondetects will be qualified as rejected, (R), and positive results will be qualified as estimated, (J), if the spike %R is <30%.

Duplicates

Qualification of sample data based on duplicate precision is left to the professional judgment of the validator. Generally, an aqueous quality control limit of 20% and a solid quality control limit of 30% is used to evaluate the relative percent difference (RPD) between the sample and laboratory duplicate results. An aqueous quality control limit of 30% and a solid quality control limit of 50% is used to evaluate the RPD between field duplicate results; qualification based on field duplicate imprecision for general chemistry parameters is applied to the field duplicate pair only.

Sample Quantitation

All reported sample concentrations should fall within the range of the calibration curve. If samples having detected concentrations the calibration range were not diluted and reanalyzed, associated sample data will be qualified as estimated, (J).

The validator should verify that sample results were properly quantitated.

5.2.5 Deliverables Guidance

The content and format of the data package generated for this method of analysis may vary significantly depending upon the work request.

In addition to any work-request requirements (e.g., data validation memorandum), all laboratory data package quality control summary forms, laboratory summaries of sample results and laboratory method blanks, and COCs must be provided to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

6.0 PHOSPHORUS (EPA 600 SERIES METHOD 365.4)

6.1 Applicability

Method 365.4 is a colorimetric method used to measure the concentration of total phosphorus in domestic and industrial effluents, and drinking and surface waters. The practical range of determination is 0.01 to 20 mg/L.

6.2 Interferences

No interferences noted in the method.

Subject DATA VALIDATION - MISCELLANEOUS INORGANICS	Number DV-09	Page 16 of 24
	Revision 0	Effective Date 08/13/01

6.3 Holding Times

Samples are to be collected in plastic or glass containers, preserved to a pH <2 with sulfuric acid, and cooled to 4°C.

A 28-day holding time between sample collection and analysis is specified. Chain of Custodies (COCs) and raw data are reviewed to determine if holding times were met for all samples. Sample data will be qualified as estimated, (J) and (UJ), if holding times were exceeded. Gross holding time violations (>2X holding time) will warrant rejection, (R), of nondetects.

6.4 Quality Control

Quality control analyses (i.e., calibrations, blanks, spikes, etc.) are not specified in Method 365.4. However, if these analyses were performed by the laboratory, the following criteria will be used to evaluate the associated sample data.

6.4.1 Calibration

The calibration curve should be checked for linearity (correlation coefficient >0.995). In general, associated sample data are qualified as estimated, (J) and (UJ), when calibration curves are not linear.

If a continuing calibration verification (CCV) standard is analyzed, the percent recovery (%R) of the CCV will be evaluated using 85-115% quality control limits. Associated sample data will be qualified as estimated, (J) and (UJ), if the CCV %R is <85%. Only positive results in the affected samples will be qualified as estimated, (J), if the CCV %R is >115%.

6.4.2 Blanks

Laboratory method and field quality control blanks, if analyzed, should be evaluated for contamination. Field quality control blanks (field, rinsate, equipment, etc.) can be identified by consulting the COCs. If contamination is noted in the associated blanks, positive sample results < the maximum amount detected in the blanks will be qualified as undetected, (U). Sample digestion and moisture content factors will be taken into consideration when qualifying the associated sample data.

6.4.3 Spikes

If a spiked sample is analyzed, a 75-125% quality control range will be used to evaluate the spike %R. Associated sample data will be qualified as estimated, (J) and (UJ), when the spiked sample %R is <75%. If the %R is >125%, only positive results are impacted and qualified as estimated, (J). If a spike %R is <30%, associated nondetects will be qualified as rejected, (R), and positive results will be qualified as estimated, (J).

6.4.4 Duplicates

Qualification of sample data based on duplicate precision is left to the professional judgment of the validator. Generally, an aqueous quality control limit of 20% and a solid quality control limit of 30% is used to evaluate the relative percent difference (RPD) between the sample and laboratory duplicate results. An aqueous quality control limit of 30% and a solid quality control limit of 50% is used to evaluate the RPD between field duplicate results; qualification based on field duplicate imprecision for general chemistry parameters is applied to the field duplicate pair only.

Subject DATA VALIDATION - MISCELLANEOUS INORGANICS	Number DV-09	Page 17 of 24
	Revision 0	Effective Date 08/13/01

6.4.5 Sample Quantitation

All reported sample concentrations should fall within the range of the calibration curve. If samples having detected concentrations > the highest calibration standard were not diluted and reanalyzed, associated sample data will be qualified as estimated, (J).

The validator should verify that sample results were properly quantitated.

6.5 Deliverables Guidance

The content and format of the data package generated for phosphorus analysis may vary significantly depending upon the work request.

In addition to any work-request requirements (e.g., data validation memorandum), all laboratory data package quality control summary forms, laboratory summaries of sample results and laboratory method blanks, and COCs must be provided to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

7.0 SULFATE (EPA 600 SERIES METHOD 375.4)

7.1 Applicability

Method 375.4 is used to determine the concentration of sulfate in domestic and industrial effluents, and drinking and surface waters. Although all sulfate concentration ranges can be measured by this turbidimetric method, a sample aliquot should not contain more than 40 mg/L of sulfate since the suspensions lose stability at concentrations >50 mg/L. The minimum detection limit for this method is 1 mg/L.

7.2 Interferences

Interferences are noted from silica concentrations >500 mg/L, suspended matter, and color in samples.

7.3 Holding Times

Samples should be collected in plastic or glass bottles and cooled to 4 C. No preservative is necessary. Holding time, which is specified as 28 days for this method, is defined as the elapsed time period from sample collection to analysis. Chain of Custodies (COCs) and raw data are reviewed to determine if holding times were met for all samples. Sample data will be qualified as estimated, (J) and (UJ), if holding times were exceeded. Gross holding time violations (>2X holding time) will warrant rejection, (R), of nondetects.

7.4 Quality Control

7.4.1 Calibration

The raw data will be reviewed to ensure that the following calibration requirements have been met:

Subject DATA VALIDATION - MISCELLANEOUS INORGANICS	Number DV-09	Page 18 of 24
	Revision 0	Effective Date 08/13/01

- The calibration curve used for sample quantitation should consist of standards at increments of 5 mg/L in the 0 to 40 mg/L sulfate range.
- A continuing calibration verification (CCV) standard is analyzed every 3 or 4 samples.

Associated sample data will be qualified as estimated, (J) and (UJ), if the above requirements have not been met.

The calibration curve should be checked for linearity (correlation coefficient >0.995). In general, sample results associated with nonlinear calibration curves are qualified as estimated, (J) and (UJ). Professional judgment should be used to qualify sample data in instances where sample results fall outside a linear portion of the calibration curve.

The percent recovery (%R) of the CCV should be within an 85-115% quality control range. Associated sample data will be qualified as estimated, (J) and (UJ), if the CCV %R is <85%. Only positive results in the affected samples will be qualified as estimated, (J), if the CCV %R is >115%.

7.4.2 Blanks

Laboratory method blanks (other than the blank used for the calibration curve) should be analyzed and evaluated for contamination. The COCs should be consulted to determine if any field quality control blanks (field, rinsate, equipment, etc.) are associated with the samples. If contamination is noted in the associated blanks, positive sample results < the maximum amount detected in the blanks will be qualified as undetected, (U). Sample digestion and moisture content factors will be taken into consideration when qualifying the associated sample data.

7.4.3 Spikes/Duplicates

The method does not require the analysis of spikes or duplicates. However, if these quality control (QC) analyses were performed by the laboratory the following criteria will be used to evaluate the associated sample data.

QC Parameter	Control Limits
Spike %R	75 - 125%
Duplicate RPD	20% for waters or 30% for solids

If the spike %R is <75%, the associated sample data will be qualified as estimated, (J) and (UJ). Only positive results will be qualified as estimated, (J), when the spike %R is >125%. Associated nondetects will be qualified as rejected, (R), and positive results will be qualified as estimated, (J), in the event that the spike %R is <30%.

Generally, associated sample results are qualified as estimated, (J) and (UJ), if the relative percent difference (RPD) between the sample and laboratory duplicate results did not meet the quality control criterion. However, in some cases, qualification of sample data based on duplicate precision is left to the professional judgment of the validator. An aqueous quality control limit of 30% and a solid quality control limit of 50% is used to evaluate the RPD between field duplicate results; qualification based on field duplicate imprecision for general chemistry parameters is applied to the field duplicate pair only.

7.4.4 Sample Quantitation

All reported sample concentrations should fall within the range of the calibration curve. If samples having detected concentrations > the highest calibration standard were not diluted and reanalyzed, the associated sample data will be qualified as estimated, (J).

Subject DATA VALIDATION - MISCELLANEOUS INORGANICS	Number DV-09	Page 19 of 24
	Revision 0	Effective Date 08/13/01

The validator will verify that sample results were correctly quantitated.

7.5 Deliverables Guidance

The content and format of the data package generated for sulfate analysis may vary significantly depending upon the work request.

In addition to any work-request requirements (e.g., data validation memorandum), all laboratory data package quality control summary forms, laboratory summaries of sample results and laboratory method blanks, and COCs must be provided to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

8.0 SULFIDES (EPA SW-846 METHOD 9030)

8.1 Applicability

Method 9030 is iodometric method used to determine the concentration of total and dissolved sulfides in excess of 1 mg/L in drinking, surface and saline waters. Acid-insoluble sulfides, such as copper sulfide, can not be measured by this titrimetric method.

8.2 Interferences

A main source of interference for this method is the reduction of iodine by various chemicals (thiosulfate, sulfite, and organic compounds). Samples are treated at collection with zinc acetate and sodium hydroxide to minimize interferences.

In addition, sulfides are susceptible to volatilization and reaction with oxygen which can form unmeasurable states of sulfides. Aeration should be minimized during sample collection.

8.3 Holding Times

Samples are preserved with zinc acetate, treated with sodium hydroxide to a pH >9, and cooled to 4 C.

Holding time is defined as the elapsed time period from sample collection to analysis. The following holding times apply to sulfide analyses:

- Unpreserved samples: Immediate analysis
- Preserved samples: 7 days

Chain of Custodies (COCs) and raw data are reviewed to determine if holding times were met for all samples. In the event that holding times are exceeded, positive results and nondetects will be qualified as estimated, (J) and (UJ), respectively. Gross holding time violations (>2X holding time) will warrant rejection, (R), of nondetects.

Subject DATA VALIDATION - MISCELLANEOUS INORGANICS	Number DV-09	Page 20 of 24
	Revision 0	Effective Date 08/13/01

8.4 Quality Control

8.4.1 Calibration

The raw data will be reviewed to ensure that the following calibration requirements have been met:

- The calibration curve should consist of a blank and three standards (at a minimum).
- A new calibration curve should be performed for every hour of continuous sample analysis.
- A continuing calibration verification (CCV) standard should be analyzed every 15 samples.

Associated sample data will be qualified as estimated, (J) and (UJ), if the above requirements have not been met.

The calibration curve should be checked for linearity. Generally, associated sample data is qualified as estimated, (J) and (UJ), if the calibration curve correlation coefficient is <0.995. Professional judgment should be used to qualify sample data in cases when sample results fall outside a linear portion of the calibration curve.

An 85-115% quality control range will be used to evaluate the percent recovery (%R) of a CCV. Associated sample data will be qualified as estimated, (J) and (UJ), if the CCV %R is <85%. Only positive results in the affected samples will be qualified as estimated, (J), if the CCV %R is >115%.

8.4.2 Blanks

At a minimum, one laboratory method blank (other than the blank used for the calibration curve) should be analyzed per sample batch (maximum of 20 samples). The COCs should be consulted to determine if any field quality control blanks (field, rinsate, equipment, etc.) are associated with the samples. If contamination is noted in the associated blanks, positive sample results < the maximum amount detected in the blanks will be qualified as undetected, (U). Sample dilution and moisture content factors will be taken into consideration when qualifying the associated sample data.

8.4.3 Spikes/Duplicates

A spiked sample and spiked duplicate sample should be analyzed for every 10 samples. If a spike or duplicate spike %R is <75%, associated sample data will be qualified as estimated, (J) and (UJ). If the %R is >125%, only positive results are impacted and qualified as estimated, (J). If a spike %R is <30%, associated nondetects will be qualified as rejected, (R), and positive results will be qualified as estimated, (J).

Qualification of sample data based on duplicate precision is left to the professional judgment of the validator. Generally, a 20% aqueous quality control limit and a 30% solid quality control limit are used to evaluate the relative percent difference (RPD) between the spiked sample and spiked duplicate sample results. An aqueous quality control limit of 30% and a solid quality control limit of 50% is used to evaluate the RPD between field duplicate results; qualification based on field duplicate imprecision for general chemistry parameters is applied to the field duplicate pair only.

8.4.4 Sample Quantitation

All reported sample concentrations should fall within the range of the calibration curve. If samples having detected concentrations > the highest calibration standard were not diluted and reanalyzed, the associated sample data will be qualified as estimated, (J).

Subject DATA VALIDATION - MISCELLANEOUS INORGANICS	Number DV-09	Page 21 of 24
	Revision 0	Effective Date 08/13/01

The validator will verify that sample results were properly quantitated.

8.5 Deliverables Guidance

The content and format of the data package generated for sulfide analysis may vary significantly depending upon the work request.

In addition to any work-request requirements (e.g., data validation memorandum), all laboratory data package quality control summary forms, laboratory summaries of sample results and laboratory method blanks, and COCs must be provided to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

9.0 TOTAL SUSPENDED SOLIDS (EPA 600 SERIES METHOD 160.2)

9.1 Applicability

Method 160.2 is a gravimetric method used to determine nonfilterable residue (total suspended solids) in domestic and industrial wastes, and drinking, surface and saline waters. The optimum range of total suspended solids (TSS) determined by this method is 4 to 20,000 mg/L.

9.2 Interferences

Requirements for apparatus and analytical techniques are specified in the method to eliminate or reduce procedural interferences. Saline waters, brines, and samples high in dissolved solids must be analyzed carefully to minimize elevated sample results.

9.3 Holding Times

Samples should be collected in plastic or glass containers and cooled to 4 °C to reduce microbiological decomposition of solids. No preservative is needed.

Holding time is defined as the elapsed time period from sample collection to analysis. Chain of Custodies (COCs) and sample data are reviewed to determine if the 7-day holding time required by this method was met for all samples. Positive results and nondetects will be qualified as estimated, (J) and (UJ), respectively, if holding times were exceeded. Gross holding time violations (>2X holding time) will warrant rejection, (R), of nondetects.

9.4 Quality Control

Method 160.2 does not require specific quality control analyses (i.e., blanks, duplicates, etc.). However, if these analyses were performed by the laboratory, the following criteria will be used to evaluate the associated sample data.

9.4.1 Verification

If a verification standard is analyzed, the percent recovery (%R) of the standard should be within a quality control range of 90-110%. Associated sample data will be qualified as estimated, (J) and (UJ), if the

Subject DATA VALIDATION - MISCELLANEOUS INORGANICS	Number DV-09	Page 22 of 24
	Revision 0	Effective Date 08/13/01

verification %R is <90%. Positive sample results will be qualified as estimated, (J), if the verification %R is >110%; nondetects are not impacted.

9.4.2 Blanks

Laboratory method and field quality control blanks, if analyzed, should be evaluated for contamination. Field quality control blanks (field, rinsate, equipment, etc.) can be identified by consulting the COCs. Positive sample results for TSS < the maximum amount detected in the blanks will be qualified as undetected, (U).

9.4.3 Duplicates

Qualification of sample data based on duplicate precision is left to the professional judgment of the validator. Generally, a quality control limit of 20% is used to evaluate the relative percent difference (RPD) between the sample and duplicate results. An aqueous quality control limit of 30% and a solid quality control limit of 50% is used to evaluate the RPD between field duplicate results; qualification based on field duplicate imprecision for general chemistry parameters is applied to the field duplicate pair only.

9.4.4 Sample Quantitation

The validator should verify that sample results were calculated accurately. The following equation is used to calculate TSS:

$$\text{TSS (mg/L)} = \frac{\text{wt}_{\text{crucible+residue}} - \text{wt}_{\text{crucible}}}{\text{VOL}_{\text{sample aliquot used}}} \times \frac{1,000 \text{ mL}}{1 \text{ L}}$$

where: wt = weight (mg)
vol = volume (mL)

9.5 Deliverables Guidance

In addition to any work-request requirements (e.g., data validation memorandum), all laboratory data package quality control summary forms, laboratory summaries of sample results and laboratory method blanks, and COCs must be provided to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.

10.0 TOTAL DISSOLVED SOLIDS (EPA 600 SERIES METHOD 160.1)

10.1 Applicability

Method 160.1 is a gravimetric method used to determine filterable residue (total dissolved solids) in domestic and industrial wastes, and drinking, surface and saline waters. The optimum range of total dissolved solids (TDS) determined by this method is 10 to 20,000 mg/L.

Subject DATA VALIDATION - MISCELLANEOUS INORGANICS	Number DV-09	Page 23 of 24
	Revision 0	Effective Date 08/13/01

10.2 Interferences

Interferences during the drying stages of the analytical procedure are observed. Samples containing high concentrations of calcium, magnesium, chloride, sulfate and bicarbonate may require longer desiccation and drying times to minimize interferences. Total residue should be limited to 200 mg to prevent entrapment of water in the evaporating dish.

10.3 Holding Times

Samples should be collected in plastic or glass containers and cooled to 4 C to reduce microbiological decomposition of solid matter. No preservative is needed.

Holding time is defined as the elapsed time period from sample collection to analysis. A 7-day holding time is specified by the method. Chain of Custodies (COCs) and raw data are reviewed to determine if holding times were met for all samples. Positive results and nondetects will be qualified as estimated, (J) and (UJ), respectively, if holding times were exceeded. Gross holding time violations (>2X holding time) will warrant rejection, (R), of nondetects.

10.4 Quality Control

Method 160.1 does not require specific quality control analyses (i.e., blanks, duplicates, etc.). However, if these analyses were performed by the laboratory, the following criteria will be used to evaluate the associated sample data.

10.4.1 Verification

If a verification standard is analyzed, the percent recovery (%R) of the standard should be within a quality control range of 90-110%. Associated sample data will be qualified as estimated, (J) and (UJ), if the verification %R is <90%. Positive sample results will be qualified as estimated, (J), if the verification %R is >110%; nondetects are not impacted.

10.4.2 Blanks

Laboratory method and field quality control blanks, if analyzed, should be evaluated for contamination. Field quality control blanks (field, rinsate, equipment, etc.) can be identified by reviewing the COCs. Positive sample results for TDS < the maximum amount detected in the blanks will be qualified as undetected, (U).

10.4.3 Duplicates

Qualification of sample data based on duplicate precision is left to the professional judgment of the validator. Generally, a quality control limit of 20% is used to evaluate the relative percent difference (RPD) between the sample and duplicate results. An aqueous quality control limit of 30% and a solid quality control limit of 50% is used to evaluate the RPD between field duplicate results; qualification based on field duplicate imprecision for general chemistry parameters is applied to the field duplicate pair only.

10.4.4 Sample Quantitation

The validator should verify that sample results were calculated accurately. The following equation is used to calculate TDS:

Subject DATA VALIDATION - MISCELLANEOUS INORGANICS	Number DV-09	Page 24 of 24
	Revision 0	Effective Date 08/13/01

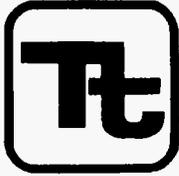
$$\text{TDS (mg/L)} \times \frac{\text{Wt}_{\text{dish+residue}} - \text{Wt}_{\text{dish}}}{\text{VOL}_{\text{sample aliquot used}}} \times \frac{1,000 \text{ mL}}{1 \text{ L}}$$

where: wt = weight (mg)
vol = volume (ml)

10.5 Deliverables Guidance

In addition to any work-request requirements (e.g., data validation memorandum), all laboratory data package quality control summary forms, laboratory summaries of sample results and laboratory method blanks, and COCs must be provided to the Data Validation Quality Assurance Officer (DV/QAO) for quality assurance review.

The validator should ensure that the format of the data validation deliverable is complete and correct and that the validation narrative is free of transcription and typographical errors before submitting all requested items for DV/QAO review.



TETRA TECH NUS, INC.

STANDARD OPERATING PROCEDURES

Number GH-1.2	Page 1 of 9
Effective Date 09/03	Revision 2
Applicability Tetra Tech NUS, Inc.	
Prepared Earth Sciences Department	
Approved D. Senovich <i>[Signature]</i>	

Subject EVALUATION OF EXISTING MONITORING WELLS AND WATER LEVEL MEASUREMENT

TABLE OF CONTENTS

<u>SECTION</u>	<u>PAGE</u>
1.0 PURPOSE.....	2
2.0 SCOPE.....	2
3.0 GLOSSARY	2
4.0 RESPONSIBILITIES	2
5.0 PROCEDURES	2
5.1 PRELIMINARY EVALUATION	3
5.2 FIELD INSPECTION	3
5.3 WATER LEVEL (HYDRAULIC HEAD) MEASUREMENTS	4
5.3.1 General.....	4
5.3.2 Water Level Measuring Techniques.....	5
5.3.3 Methods.....	5
5.3.4 Water Level Measuring Devices	6
5.3.5 Data Recording	6
5.3.6 Specific Quality Control Procedures for Water Level Measuring Devices	7
5.4 EQUIPMENT DECONTAMINATION.....	7
5.5 HEALTH AND SAFETY CONSIDERATIONS	7
6.0 RECORDS	7
 <u>ATTACHMENTS</u>	
A MONITORING WELL INSPECTION SHEET	8
B GROUNDWATER LEVEL MEASUREMENT SHEET	9

Subject EVALUATION OF EXISTING MONITORING WELLS AND WATER LEVEL MEASUREMENT	Number GH-1.2	Page 2 of 9
	Revision 2	Effective Date 09/03

1.0 PURPOSE

The purpose of this procedure is to provide reference information regarding the proper methods for evaluating the physical condition and project utility of existing monitoring wells and determining water levels.

2.0 SCOPE

The procedures described herein are applicable to all existing monitoring wells and, for the most part, are independent of construction materials and methods.

3.0 GLOSSARY

Hydraulic Head - The height to which water will rise in a well.

Water Table - A surface in an unconfined aquifer where groundwater pressure is equal to atmospheric pressure (i.e., the pressure head is zero).

4.0 RESPONSIBILITIES

Site Geologist/Hydrogeologist - Has overall responsibility for the evaluation of existing wells, obtaining water level measurements and developing groundwater contour maps. The site geologist/hydrogeologist (in concurrence with the Project Manager) shall specify the reference point from which water levels are measured (usually a specific point on the upper edge of the inner well casing), the number and location of data points which shall be used for constructing a contour map, and how many complete sets of water levels are required to adequately define groundwater flow directions (e.g., if there are seasonal variations).

Field Personnel - Must have a basic familiarity with the equipment and procedures involved in obtaining water levels and must be aware of any project-specific requirements or objectives.

5.0 PROCEDURES

Accurate, valid and useful groundwater monitoring requires that four important conditions be met:

- Proper characterization of site hydrogeology.
- Proper design of the groundwater monitoring program, including adequate numbers of wells installed at appropriate locations and depths.
- Satisfactory methods of groundwater sampling and analysis to meet the project data quality objectives (DQOs).
- The assurance that specific monitoring well samples are representative of water quality conditions in the monitored interval.

To insure that these conditions are met, adequate descriptions of subsurface geology, well construction methods and well testing results must be available. The following steps will help to insure that the required data are available to permit an evaluation of the utility of existing monitoring wells for collecting additional samples.

Subject EVALUATION OF EXISTING MONITORING WELLS AND WATER LEVEL MEASUREMENT	Number GH-1.2	Page 3 of 9
	Revision 2	Effective Date 09/03

5.1 Preliminary Evaluation

A necessary first step in evaluating existing monitoring well data is the study and review of the original work plan for monitoring well installation (if available). This helps to familiarize the site geologist/hydrogeologist with site-specific condition, and will promote an understanding of the original purpose of the monitoring wells.

The next step of the evaluation should involve a review of all available information concerning borehole drilling and well construction. This will allow interpretation of groundwater flow conditions and area geology, and will help to establish consistency between hydraulic properties of the well and physical features of the well or formation. The physical features which should be identified and detailed, if available, include:

- The well identification number, permit number and location by referenced coordinates, the distance from prominent site features, or the location of the well on a map.
- The installation dates, drilling methods, well development methods, past sampling dates, and drilling contractors.
- The depth to bedrock -- where rock cores were not taken, auger refusal, drive casing refusal or penetration test results (blow counts for split-barrel sampling) may be used to estimate bedrock interface.
- The soil profile and stratigraphy.
- The borehole depth and diameter.
- The elevation of the top of the protective casing, the top of the well riser, and the ground surface.
- The total depth of the well.
- The type of well materials, screen type, slot size, and length, and the elevation/depths of the screen, interval, and/or monitored interval.
- The elevation/depths of the tops and bottom of the filter pack and well seals and the type and size.

5.2 Field Inspection

During the onsite inspection of existing monitoring wells, features to be noted include:

- The condition of the protective casing, cap and lock.
- The condition of the cement seal surrounding the protective casing.
- The presence of depressions or standing water around the casing.
- The presence of and condition of dedicated sampling equipment.
- The presence of a survey mark on the inner well casing.

If the protective casing, cap and lock have been damaged or the cement collar appears deteriorated, or if there are any depressions around the well casing capable of holding water, surface water may have infiltrated into the well. This may invalidate previous sampling results unless the time when leakage started can be precisely determined.

The routine physical inspection must be followed by a more detailed investigation to identify other potential routes of contamination or sampling equipment malfunction. Any of these occurrences may invalidate

Subject EVALUATION OF EXISTING MONITORING WELLS AND WATER LEVEL MEASUREMENT	Number GH-1.2	Page 4 of 9
	Revision 2	Effective Date 09/03

previously-collected water quality data. If the monitoring well is to be used in the future, considerations shown in the steps described above should be rectified to rehabilitate the well.

After disconnecting any wires, cables or electrical sources, remove the lock and open the cap. Check for the presence of organic vapors with a photoionization detector (PID) or flame-ionization detector (FID) to determine the appropriate worker safety level. The following information should be noted:

- Cap function.
- Physical characteristics and composition of the inner casing or riser, including inner diameter and annular space.
- Presence of grout between the riser and outer protective casing and the existence of drain holes in the protective casing.
- Presence of a riser cap, method of attachment to casing, and venting of the riser.
- Presence of dedicated sampling equipment; if possible, remove such equipment and inspect size, materials of construction and condition.

The final step of the field inspection is to confirm previous hydraulic or physical property data and to obtain data not previously available. This includes the determination of static water levels, total well depth and well obstruction. This may be accomplished using a weighted tape measure which can also be used to check for sediment (the weight will advance slowly if sediment is present, and the presence of sediment on the weight upon removal should be noted). If sediment is present and/or the well has not been sampled in 12 or more months, it should be redeveloped before sampling.

Lastly, as a final step, the location, condition and expected water quality of the wells should be reviewed in light of their usefulness for the intended purpose of the investigation.

See Attachment A, Monitoring Well Inspection Sheet.

5.3 Water Level (Hydraulic Head) Measurements

5.3.1 General

Groundwater level measurements can be made in monitoring wells, private or public water wells, piezometers, open boreholes, or test pits (after stabilization). Groundwater measurements should generally not be made in boreholes with drilling rods or auger flights present. If groundwater sampling activities are to occur, groundwater level measurements shall take place prior to well purging or sampling.

All groundwater level measurements shall be made to the nearest 0.01 foot, and recorded in the site geologist/hydrogeologist's field notebook or on the Groundwater Level Measurement Sheet (Attachment B), along with the date and time of the reading. The total depth of the well shall be measured and recorded, if not already known. Weather changes that occur over the period of time during which water levels are being taken, such as precipitation and barometric pressure changes, should be noted.

In measuring groundwater levels, there shall be a clearly-established reference point of known elevation, which is normally identified by a mark on the upper edge of the inner well casing. To be useful, the reference point should be tied in with an established USGS benchmark or other properly surveyed elevation datum. An arbitrary datum could be used for an isolated group of wells, if necessary.

Subject EVALUATION OF EXISTING MONITORING WELLS AND WATER LEVEL MEASUREMENT	Number GH-1.2	Page 5 of 9
	Revision 2	Effective Date 09/03

Cascading water within a borehole or steel well casings can cause false readings with some types of sounding devices (chalked line, electrical). Oil layers may also cause problems in determining the true water level in a well. Special devices (interface probes) are available for measuring the thickness of oil layers and true depth to groundwater, if required.

Water level readings shall be taken regularly, as required by the site geologist/hydrogeologist. Monitoring wells or open-cased boreholes that are subject to tidal fluctuations should be read in conjunction with a tidal chart (or preferably in conjunction with readings of a tide staff or tide level recorder installed in the adjacent water body); the frequency of such readings shall be established by the site hydrogeologist. All water level measurements at a site used to develop a groundwater contour map shall be made in the shortest practical time to minimize affects due to weather changes.

5.3.2 Water Level Measuring Techniques

There are several methods for determining standing or changing water levels in boreholes and monitoring wells. Certain methods have particular advantages and disadvantages depending upon well conditions. A general description of these methods is presented, along with a listing of various advantages and disadvantages of each technique. An effective technique shall be selected for the particular site conditions by the site geologist/hydrogeologist.

In most instances, preparation of accurate potentiometric surface maps require that static water level measurements be obtained to a precision of 0.01 feet. To obtain such measurements in individual accessible wells, electrical water level indicator methods have been found to be best, and thus should be utilized. Other, less precise methods, such as the popper or bell sound, or bailer line methods, should be avoided. When a large number of (or continuous) readings are required, time-consuming individual readings are not usually feasible. In such cases, it is best to use a pressure transducer.

5.3.3 Methods

Water levels can be measured by several different techniques, but the same steps shall be followed in each case. The proper sequence is as follows:

1. Check operation of recording equipment above ground. Prior to opening the well, don personal protective equipment, as required. Never remove an air-tight lock (such as a J-plug) with your face over the well. Pressure changes within the well may explosively force the cap off once loosened.
2. Record all information specified below in the geologist/hydrogeologist's field notebook or on the Groundwater Level Measurement Sheet (Attachment B):
 - Well number.
 - Water level (to the nearest 0.01 foot). Water levels shall be taken from the surveyed reference mark on the top edge of the inner well casing. If the J-plug was on the well very tightly, it may take several minutes for the water level to stabilize.
 - Time and day of the measurement.
 - Thickness of free product if present.

Water level measuring devices with permanently marked intervals shall be used. The devices shall be free of kinks or folds which will affect the ability of the equipment to hang straight in the well pipe.

Subject EVALUATION OF EXISTING MONITORING WELLS AND WATER LEVEL MEASUREMENT	Number GH-1.2	Page 6 of 9
	Revision 2	Effective Date 09/03

5.3.4 Water Level Measuring Devices

Electric Water Level Indicators

These are the most commonly used devices and consist of a spool of small-diameter cable and a weighted probe attached to the end. When the probe comes in contact with the water, an electrical circuit is closed and a meter, light, and/or buzzer attached to the spool will signal the contact.

There are a number of commercial electric sounders available, none of which is entirely reliable under all conditions likely to occur in a contaminated monitoring well. In conditions where there is oil on the water, groundwater with high specific conductance, water cascading into the well, steel well casing, or a turbulent water surface in the well, measuring with an electric sounder may be difficult.

For accurate readings, the probe shall be lowered slowly into the well adjacent to the survey mark on the inner well casing. The electric tape is read (to the nearest 0.01 ft.) at the measuring point and recorded where contact with the water surface was indicated.

Popper or Bell Sounder

A bell- or cup-shaped weight that is hollow on the bottom is attached to a measuring tape and lowered into the well. A "popping" or "plopping" sound is made when the weight strikes the surface of the water. An accurate reading can be determined by lifting and lowering the weight in short strokes, and reading the tape when the weight strikes the water. This method is not sufficiently accurate to obtain water levels to 0.01 feet, and thus is more appropriate for obtaining only approximate water levels quickly.

Pressure Transducer

Pressure transducers can be lowered into a well or borehole to measure the pressure of water and therefore the water elevation above the transducer. The transducer is wired into a recorder at the surface to record changes in water level with time. The recorder digitizes the information and can provide a printout or transfer the information to a computer for evaluation (using a well drawdown/recovery model). The pressure transducer should be initially calibrated with another water level measurement technique to ensure accuracy. This technique is very useful for hydraulic conductivity testing in highly permeable material where repeated, accurate water level measurements are required in a very short period of time. A sensitive transducer element is required to measure water levels to 0.01 foot accuracy.

Borehole Geophysics

Approximate water levels can be determined during geophysical logging of the borehole (although this is not the primary purpose for geophysical logging and such logging is not cost effective if used only for this purpose). Several logging techniques will indicate water level. Commonly-used logs which will indicate saturated/unsaturated conditions include the spontaneous potential (SP) log and the neutron log.

5.3.5 Data Recording

Water level measurements, time, data, and weather conditions shall be recorded in the geologist/hydrogeologist's field notebook or on the Groundwater Level Measurement Sheet. All water level measurements shall be measured from a known reference point. The reference point is generally a marked point on the upper edge of the inner well casing that has been surveyed for an elevation. The exact reference point shall be marked with permanent ink on the casing since the top of the casing may not be entirely level. It is important to note changes in weather conditions because changes in the barometric pressure may affect the water level within the well.

Subject EVALUATION OF EXISTING MONITORING WELLS AND WATER LEVEL MEASUREMENT	Number GH-1.2	Page 7 of 9
	Revision 2	Effective Date 09/03

5.3.6 Specific Quality Control Procedures for Water Level Measuring Devices

All groundwater level measurement devices must be cleaned before and after each use to prevent cross contamination of wells. Manufacturer's instructions for cleaning the device shall be strictly followed. Some devices used to measure groundwater levels may need to be calibrated. These devices shall be calibrated to 0.01 foot accuracy and any adjustments/corrections shall be recorded in the field logbook/notebook. After the corrections/adjustments are made to the measuring device and entered in the field logbook/notebook, the corrected readings shall be entered onto the Groundwater Level Measurement Sheet (Attachment B). Elevations will be entered on the sheet when they become available.

5.4 Equipment Decontamination

Equipment used for water level measurements provide a mechanism for potentially cross contaminating wells. Therefore, all portions of a device which project down the well casing must be decontaminated prior to advancing to the next well. Decontamination procedures vary based on the project objectives but must be defined prior to conducting any field activities including the collection of water level data. Consult the project planning documents and SA-7.1 Decontamination of Field Equipment.

5.5 Health and Safety Considerations

Groundwater contaminated by volatile organic compounds may release toxic vapors into the air space inside the well pipe. The release of this air when the well is initially opened is a health/safety hazard which must be considered. Initial monitoring of the well headspace and breathing zone concentrations using a PID or FID shall be performed to determine required levels of protection. Under certain conditions, air-tight well caps may explosively fly off the well when the pressure is relieved. Never stand directly over a well when uncapping it.

6.0 RECORDS

A record of all field procedures, tests and observations must be recorded in the site logbook or designated field notebook. Entries in the log/notebook should include the individuals participating in the field effort, and the date and time. The use of annotated sketches may help to supplement the evaluation.

Subject EVALUATION OF EXISTING MONITORING WELLS AND WATER LEVEL MEASUREMENT	Number GH-1.2	Page 8 of 9
	Revision 2	Effective Date 09/03

ATTACHMENT A
MONITORING WELL INSPECTION SHEET

Monitoring Well Inspection Sheet

Project Name: _____ Date: _____
Location: _____ Time: _____
Tidally Influenced: Y / N Personnel: _____

Field Measurements				
Well ID	PID Reading PPM	Depth to Water *	Total Depth *	Flush Mt./ Stick-up

Well Construction Details (Taken from construction logs)		
Total Depth *	Ground Elev.	Top/Btm Screen *

Check List:

Riser Pipe Material:
Riser Notched for Surveyors:
Well ID Tag In-place:
Well security:
Photo taken:

Condition of Well:

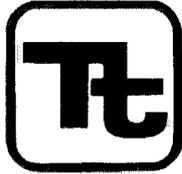
Protective Case:
Riser:
Well Pad:
Other:

Presence/Evidence of:

Standing Water Around Well:
Existing Sampling Equipment:
Sediment build-up in Well Btm:

Comments:

* = Measurements are from the top of the inner case to the nearest 0.01'



TETRA TECH NUS, INC.

STANDARD OPERATING PROCEDURES

Number GH-1.5	Page 1 of 20
Effective Date 06/99	Revision 1
Applicability Tetra Tech NUS, Inc.	
Prepared Earth Sciences Department	
Approved D. Senovich <i>DS</i>	

Subject
BOREHOLE AND SAMPLE LOGGING

TABLE OF CONTENTS

<u>SECTION</u>	<u>PAGE</u>
1.0 PURPOSE	3
2.0 SCOPE	3
3.0 GLOSSARY.....	3
4.0 RESPONSIBILITIES	3
5.0 PROCEDURES	3
5.1 MATERIALS NEEDED	3
5.2 CLASSIFICATION OF SOILS	3
5.2.1 USCS Classification	6
5.2.2 Color	6
5.2.3 Relative Density and Consistency	6
5.2.4 Weight Percentages	7
5.2.5 Moisture	10
5.2.6 Stratification	10
5.2.7 Texture/Fabric/Bedding	10
5.2.8 Summary of Soil Classification	10
5.3 CLASSIFICATION OF ROCKS	13
5.3.1 Rock Type.....	13
5.3.2 Color	16
5.3.3 Bedding Thickness	16
5.3.4 Hardness	16
5.3.5 Fracturing.....	16
5.3.6 Weathering	17
5.3.7 Other Characteristics.....	17
5.3.8 Additional Terms Used in the Description of Rock	18
5.4 ABBREVIATIONS	19
5.5 BORING LOGS AND DOCUMENTATION	19
5.5.1 Soil Classification	19
5.5.2 Rock Classification	23
5.5.3 Classification of Soil and Rock from Drill Cuttings	24
5.6 REVIEW.....	24
6.0 REFERENCES	24
7.0 RECORDS	25

Subject BOREHOLE AND SAMPLE LOGGING	Number GH-1.5	Page 2 of 20
	Revision 1	Effective Date 06/99

TABLE OF CONTENTS (Continued)

FIGURES

<u>NUMBERS</u>		<u>PAGE</u>
1	BORING LOG (EXAMPLE)	4
2	CONSISTENCY FOR COHESIVE SOILS	8
3	BEDDING THICKNESS CLASSIFICATION	10
4	GRAIN SIZE CLASSIFICATION FOR ROCKS	12
5	COMPLETED BORING LOG (EXAMPLE)	17

Subject BOREHOLE AND SAMPLE LOGGING	Number GH-1.5	Page 3 of 20
	Revision 1	Effective Date 06/99

1.0 PURPOSE

The purpose of this document is to establish standard procedures and technical guidance on borehole and sample logging.

2.0 SCOPE

These procedures provide descriptions of the standard techniques for borehole and sample logging. These techniques shall be used for each boring logged to provide consistent descriptions of subsurface lithology. While experience is the only method to develop confidence and accuracy in the description of soil and rock, the field geologist/engineer can do a good job of classification by careful, thoughtful observation and by being consistent throughout the classification procedure.

3.0 GLOSSARY

None.

4.0 RESPONSIBILITIES

Site Geologist. Responsible for supervising all boring activities and assuring that each borehole is completely logged. If more than one rig is being used on site, the Site Geologist must make sure that each field geologist is properly trained in logging procedures. A brief review or training session may be necessary prior to the start up of the field program and/or upon completion of the first boring.

5.0 PROCEDURES

The classification of soil and rocks is one of the most important jobs of the field geologist/engineer. To maintain a consistent flow of information, it is imperative that the field geologist/engineer understand and accurately use the field classification system described in this SOP. This identification is based on visual examination and manual tests.

5.1 Materials Needed

When logging soil and rock samples, the geologist or engineer may be equipped with the following:

- Rock hammer
- Knife
- Camera
- Dilute hydrochloric acid (HCl)
- Ruler (marked in tenths and hundredths of feet)
- Hand Lens

5.2 Classification of Soils

All data shall be written directly on the boring log (Figure 1) or in a field notebook if more space is needed. Details on filling out the boring log are discussed in Section 5.5.

FIGURE 1 (CONTINUED)

SOIL TERMS

UNIFIED SOIL CLASSIFICATION (USCS)											
COARSE-GRAINED SOILS More Than Half of Material is LARGER Than No. 200 Sieve Size					FINE-GRAINED SOILS More Than Half of Material is SMALLER Than No. 200 Sieve Size						
FIELD IDENTIFICATION PROCEDURES (Excluding Field-Layer Thin > 1 inch and Heavy Fracture on Estimated Weights)			GROUP SYMBOL	TYPICAL NAMES		FIELD IDENTIFICATION PROCEDURES (Excluding Field-Layer Thin > 1 inch and Heavy Fracture on Estimated Weights)			GROUP SYMBOL	TYPICAL NAMES	
						Identification Procedure on Fraction Smaller than No. 40 Sieve Size					
						DIAPHRAGM (Counting Characteristics)	ILLIUMIN (Reaction to Spacing)	TOUCHING (Reaction to Near Field-Layer)			
GRAVELS (G) > 40% W _T	CLEAN GRAVELS (Low % Fines)	Wide range in grain size and substantial amounts of all intermediate particle sizes.	GW	Well-graded gravel, gravel-sand mixture, fine or no fines.		SILT AND CLAYS Liquid Limit < 4	None to Slight	Out to Slow	None	ML	Inorganic silts and very fine sands, rock flour, silty or clayey fine sands with slight plasticity.
		Preferably one size or a range of sizes with some intermediate size missing.	GP	Poorly graded gravel, gravel-sand mixture, fine or no fines.			Medium to High	None to Very Slow	Medium	CL	Inorganic clays of low to medium plasticity, gravelly clays, sandy clays, silty clays, lean clays.
	Non-plastic fines (for identification procedures, see ML)	GM	Silty gravel, poorly graded gravel-sand mixture.		Slight to Medium		Slow	Slight	OL	Organic silt and organic silty clays of low plasticity.	
SANDS (S) > 40% W _T	CLEAN SANDS (Low % Fines)	Wide range in grain size and substantial amounts of all intermediate particle sizes.	SW	Well-graded sand, gravelly sand, fine or no fines.		SILT AND CLAYS Liquid Limit > 4	Slight to Medium	Slow to None	Slight to Medium	MH	Inorganic clays, silty clays or clays in which the sandy or silty soils, plastic silts.
		Preferably one size or a range of sizes with some intermediate size missing.	SP	Poorly graded sand, gravelly sand, fine or no fines.			High to Very High	None	High	CH	Inorganic clays of high plasticity, fat clays.
	Non-plastic fines (for identification procedures, see ML)	SM	Silty sand, poorly graded sand-silt mixture.		Medium to High		None to Very Slow	Slight to Medium	OH	Organic clays of medium to high plasticity.	
SANDS (S) > 40% W _T	SANDS WITH FINES (High % Fines)	Wide range in grain size and substantial amounts of all intermediate particle sizes.	SC	Clayey sand, poorly graded sand-clay mixture.		HIGHLY ORGANIC SOILS	Readily disintegrated by color, odor, spongy feel and frequently by blow-stakes.			PT	Peat and other organic soils
		Preferably one size or a range of sizes with some intermediate size missing.	SC	Clayey sand, poorly graded sand-clay mixture.							

Boundary classification: Soils possessing characteristics of two groups are designated by combining group symbols. For example, GW-GC, well-graded gravel-sand mixture with clay binder. All test sizes on this chart are U.S. Standard.

DENSITY OF GRANULAR SOILS	
DESIGNATION	STANDARD PENETRATION TEST VALUE - BLOW/SFOOT
Very Loose	< 4
Loose	4 - 9
Medium Loose	10 - 15
Dense	16 - 30
Very Dense	Over 30

CONSISTENCY OF COHESIVE SOILS			
CONSISTENCY	UNCORRECTED SPT BLOW COUNT (EMSD) (FT)	STANDARD PENETRATION TEST VALUE - BLOW/SFOOT	FIELD IDENTIFICATION TERMS
Very Soft	Less than 2	< 1 to 1	Easily penetrated several inches by hand.
Soft	2 to 4	1 to 4	Easily penetrated several inches by thumb.
Medium Soft	4 to 10	4 to 10	Can be penetrated several inches by thumb.
Stiff	10 to 20	10 to 20	Resists indented by thumb.
Very Stiff	20 to 40	20 to 40	Resists indented by thumb nail.
Hard	More than 40	Over 40	Resists indented by thumb nail.

ROCK TERMS

ROCK HARDNESS (FROM CORE SAMPLES)			ROCK BROKENNESS		
Descriptive Term	Spall/Chips or Knife Edges	Hammer Edges	Descriptive Term	Abbreviation	Spalling
Soft	Easily chipped	Cracks when pressed with hammer	Very Broken	(V.B.)	> 4"
Medium Soft	Can be chipped	Breaks (one block), crumbly edges	Broken	(B.)	1" - 4"
Medium Hard	Can be scratched	Breaks (one block), sharp edges	Blocky	(BL)	1/2" - 1"
Hard	Cannot be scratched	Breaks (one block), sharp edges	Massive	(M)	> 1/2"

LEGEND:

SOIL SAMPLES - TYPES

- 5' x 5" Split-Bar Sample
- 5' x 7" O.D. Undisturbed Sample
- 0 - Other Sample Sizes, Specify in Remarks

ROCK SAMPLES - TYPES

- XNO (Conventional) Core, 2-1/4" O.D.
- ONO (Thin) Core, 1-1/2" O.D.
- Z - Other Core Sizes, Specify in Remarks

TEST LEVELS

- 1 - 1' to 2' depth
- 2 - 2' to 4' depth
- 3 - 4' to 6' depth
- 4 - 6' to 8' depth
- 5 - 8' to 10' depth
- 6 - 10' to 12' depth
- 7 - 12' to 14' depth
- 8 - 14' to 16' depth
- 9 - 16' to 18' depth
- 10 - 18' to 20' depth

Subject BOREHOLE AND SAMPLE LOGGING	Number GH-1.5	Page 6 of 20
	Revision 1	Effective Date 06/99

5.2.1 USCS Classification

Soils are to be classified according to the Unified Soil Classification System (USCS). This method of classification is detailed in Figure 1 (Continued).

This method of classification identifies soil types on the basis of grain size and cohesiveness.

Fine-grained soils, or fines, are smaller than the No. 200 sieve and are of two types: silt (M) and clay (C). Some classification systems define size ranges for these soil particles, but for field classification purposes, they are identified by their respective behaviors. Organic material (O) is a common component of soil but has no size range; it is recognized by its composition. The careful study of the USCS will aid in developing the competence and consistency necessary for the classification of soils.

Coarse-grained soils shall be divided into rock fragments, sand, or gravel. The terms sand and gravel not only refer to the size of the soil particles but also to their depositional history. To insure accuracy in description, the term rock fragments shall be used to indicate angular granular materials resulting from the breakup of rock. The sharp edges typically observed indicate little or no transport from their source area, and therefore the term provides additional information in reconstructing the depositional environment of the soils encountered. When the term "rock fragments" is used it shall be followed by a size designation such as "(1/4 inch Φ -1/2 inch Φ)" or "coarse-sand size" either immediately after the entry or in the remarks column. The USCS classification would not be affected by this variation in terms.

5.2.2 Color

Soil colors shall be described utilizing a single color descriptor preceded, when necessary, by a modifier to denote variations in shade or color mixtures. A soil could therefore be referred to as "gray" or "light gray" or "blue-gray." Since color can be utilized in correlating units between sampling locations, it is important for color descriptions to be consistent from one boring to another.

Colors must be described while the sample is still moist. Soil samples shall be broken or split vertically to describe colors. Samplers tend to smear the sample surface creating color variations between the sample interior and exterior.

The term "mottled" shall be used to indicate soils irregularly marked with spots of different colors. Mottling in soils usually indicates poor aeration and lack of good drainage.

Soil Color Charts shall not be used unless specified by the project manager.

5.2.3 Relative Density and Consistency

To classify the relative density and/or consistency of a soil, the geologist is to first identify the soil type. Granular soils contain predominantly sands and gravels. They are noncohesive (particles do not adhere well when compressed). Finer-grained soils (silts and clays) are cohesive (particles will adhere together when compressed).

The density of noncohesive, granular soils is classified according to standard penetration resistances obtained from split-barrel sampling performed according to the methods detailed in Standard Operating Procedures GH-1.3 and SA-1.3. Those designations are:

Designation	Standard Penetration Resistance (Blows per Foot)
Very loose	0 to 4
Loose	5 to 10
Medium dense	11 to 30
Dense	31 to 50
Very dense	Over 50

Standard penetration resistance is the number of blows required to drive a split-barrel sampler with a 2-inch outside diameter 12 inches into the material using a 140-pound hammer falling freely through 30 inches. The sampler is driven through an 18-inch sample interval, and the number of blows is recorded for each 6-inch increment. The density designation of granular soils is obtained by adding the number of blows required to penetrate the last 12 inches of each sample interval. It is important to note that if gravel or rock fragments are broken by the sampler or if rock fragments are lodged in the tip, the resulting blow count will be erroneously high, reflecting a higher density than actually exists. This shall be noted on the log and referenced to the sample number. Granular soils are given the USCS classifications GW, GP, GM, SW, SP, SM, GC, or SC (see Figure 1).

The consistency of cohesive soils is determined by performing field tests and identifying the consistency as shown in Figure 2.

Cohesive soils are given the USCS classifications ML, MH, CL, CH, OL, or OH (see Figure 1).

The consistency of cohesive soils is determined either by blow counts, a pocket penetrometer (values listed in the table as Unconfined Compressive Strength), or by hand by determining the resistance to penetration by the thumb. The pocket penetrometer and thumb determination methods are conducted on a selected sample of the soil, preferably the lowest 0.5 foot of the sample in the split-barrel sampler. The sample shall be broken in half and the thumb or penetrometer pushed into the end of the sample to determine the consistency. Do not determine consistency by attempting to penetrate a rock fragment. If the sample is decomposed rock, it is classified as a soft decomposed rock rather than a hard soil. Consistency shall not be determined solely by blow counts. One of the other methods shall be used in conjunction with it. The designations used to describe the consistency of cohesive soils are shown in Figure 2.

5.2.4 Weight Percentages

In nature, soils are comprised of particles of varying size and shape, and are combinations of the various grain types. The following terms are useful in the description of soil:

Terms of Identifying Proportion of the Component	Defining Range of Percentages by Weight
Trace	0 - 10 percent
Some	11 - 30 percent
Adjective form of the soil type (e.g., "sandy")	31 - 50 percent

FIGURE 2

CONSISTENCY FOR COHESIVE SOILS

Consistency	Standard Penetration Resistance (Blows per Foot)	Unconfined Compressive Strength (Tons/Sq. Foot by pocket penetration)	Field Identification
Very soft	0 to 2	Less than 0.25	Easily penetrated several inches by fist
Soft	2 to 4	0.25 to 0.50	Easily penetrated several inches by thumb
Medium stiff	4 to 8	0.50 to 1.0	Can be penetrated several inches by thumb with moderate effort
Stiff	8 to 15	1.0 to 2.0	Readily indented by thumb but penetrated only with great effort
Very stiff	15 to 30	2.0 to 4.0	Readily indented by thumbnail
Hard	Over 30	More than 4.0	Indented with difficulty by thumbnail

Subject BOREHOLE AND SAMPLE LOGGING	Number GH-1.5	Page 9 of 20
	Revision 1	Effective Date 06/99

Examples:

- Silty fine sand: 50 to 69 percent fine sand, 31 to 50 percent silt.
- Medium to coarse sand, some silt: 70 to 80 percent medium to coarse sand, 11 to 30 percent silt.
- Fine sandy silt, trace clay: 50 to 68 percent silt, 31 to 49 percent fine sand, 1 to 10 percent clay.
- Clayey silt, some coarse sand: 70 to 89 percent clayey silt, 11 to 30 percent coarse sand.

5.2.5 Moisture

Moisture content is estimated in the field according to four categories: dry, moist, wet, and saturated. In dry soil, there appears to be little or no water. Saturated samples obviously have all the water they can hold. Moist and wet classifications are somewhat subjective and often are determined by the individual's judgment. A suggested parameter for this would be calling a soil wet if rolling it in the hand or on a porous surface liberates water, i.e., dirties or muddies the surface. Whatever method is adopted for describing moisture, it is important that the method used by an individual remains consistent throughout an entire drilling job.

Laboratory tests for water content shall be performed if the natural water content is important.

5.2.6 Stratification

Stratification can only be determined after the sample barrel is opened. The stratification or bedding thickness for soil and rock is depending on grain size and composition. The classification to be used for stratification description is shown in Figure 3.

5.2.7 Texture/Fabric/Bedding

The texture/fabric/bedding of the soil shall be described. Texture is described as the relative angularity of the particles: rounded, subrounded, subangular, and angular. Fabric shall be noted as to whether the particles are flat or bulky and whether there is a particular relation between particles (i.e., all the flat particles are parallel or there is some cementation). The bedding or structure shall also be noted (e.g., stratified, lensed, nonstratified, heterogeneous varved).

5.2.8 Summary of Soil Classification

In summary, soils shall be classified in a similar manner by each geologist/engineer at a project site. The hierarchy of classification is as follows:

- Density and/or consistency
- Color
- Plasticity (Optional)
- Soil types
- Moisture content
- Stratification
- Texture, fabric, bedding
- Other distinguishing features

FIGURE 3

BEDDING THICKNESS CLASSIFICATION

Thickness (metric)	Thickness (Approximate English Equivalent)	Classification
> 1.0 meter	> 3.3'	Massive
30 cm - 1 meter	1.0' - 3.3'	Thick Bedded
10 cm - 30 cm	4" - 1.0'	Medium Bedded
3 cm - 10 cm	1" - 4"	Thin Bedded
1 cm - 3 cm	2/5" - 1"	Very Thin Bedded
3 mm - 1 cm	1/8" - 2/5"	Laminated
1 mm - 3 mm	1/32" - 1/8"	Thinly Laminated
< 1 mm	<1/32"	Micro Laminated

(Weir, 1973 and Ingram, 1954)

Subject BOREHOLE AND SAMPLE LOGGING	Number GH-1.5	Page 11 of 20
	Revision 1	Effective Date 06/99

5.3 Classification of Rocks

Rocks are grouped into three main divisions: sedimentary, igneous and metamorphic. Sedimentary rocks are by far the predominant type exposed at the earth's surface. The following basic names are applied to the types of rocks found in sedimentary sequences:

- Sandstone - Made up predominantly of granular materials ranging between 1/16 to 2 mm in diameter.
- Siltstone - Made up of granular materials less than 1/16 to 1/256 mm in diameter. Fractures irregularly. Medium thick to thick bedded.
- Claystone - Very fine-grained rock made up of clay and silt-size materials. Fractures irregularly. Very smooth to touch. Generally has irregularly spaced pitting on surface of drilled cores.
- Shale - A fissile very fine-grained rock. Fractures along bedding planes.
- Limestone - Rock made up predominantly of calcite (CaCO_3). Effervesces strongly upon the application of dilute hydrochloric acid.
- Coal - Rock consisting mainly of organic remains.
- Others - Numerous other sedimentary rock types are present in lesser amounts in the stratigraphic record. The local abundance of any of these rock types is dependent upon the depositional history of the area. Conglomerate, halite, gypsum, dolomite, anhydrite, lignite, etc. are some of the rock types found in lesser amounts.

In classifying a sedimentary rock the following hierarchy shall be noted:

- Rock type
- Color
- Bedding thickness
- Hardness
- Fracturing
- Weathering
- Other characteristics

5.3.1 Rock Type

As described above, there are numerous types of sedimentary rocks. In most cases, a rock will be a combination of several grain types, therefore, a modifier such as a sandy siltstone, or a silty sandstone can be used. The modifier indicates that a significant portion of the rock type is composed of the modifier. Other modifiers can include carbonaceous, calcareous, siliceous, etc.

Grain size is the basis for the classification of clastic sedimentary rocks. Figure 4 is the Udden-Wentworth classification that will be assigned to sedimentary rocks. The individual boundaries are slightly different than the USCS subdivision for soil classification. For field determination of grain sizes, a scale can be used for the coarse grained rocks. For example, the division between siltstone and claystone may not be measurable in the field. The boundary shall be determined by use of a hand lens. If the grains cannot be seen with the naked eye but are distinguishable with a hand lens, the rock is a siltstone. If the grains are not distinguishable with a hand lens, the rock is a claystone.

FIGURE 4**GRAIN SIZE CLASSIFICATION FOR ROCKS**

Particle Name	Grain Size Diameter
Cobbles	> 64 mm
Pebbles	4 - 64 mm
Granules	2 - 4 mm
Very Coarse Sand	1 - 2 mm
Coarse Sand	0.5 - 1 mm
Medium Sand	0.25 - 0.5 mm
Fine Sand	0.125 - 0.25 mm
Very Fine Sand	0.0625 - 0.125 mm
Silt	0.0039 - 0.0625 mm

After Wentworth, 1922

Subject BOREHOLE AND SAMPLE LOGGING	Number GH-1.5	Page 13 of 20
	Revision 1	Effective Date 06/99

5.3.2 Color

The color of a rock can be determined in a similar manner as for soil samples. Rock core samples shall be classified while wet, when possible, and air cored samples shall be scraped clean of cuttings prior to color classifications.

Rock color charts shall not be used unless specified by the Project Manager.

5.3.3 Bedding Thickness

The bedding thickness designations applied to soil classification (see Figure 3) will also be used for rock classification.

5.3.4 Hardness

The hardness of a rock is a function of the compaction, cementation, and mineralogical composition of the rock. A relative scale for sedimentary rock hardness is as follows:

- Soft - Weathered, considerable erosion of core, easily gouged by screwdriver, scratched by fingernail. Soft rock crushes or deforms under pressure of a pressed hammer. This term is always used for the hardness of the saprolite (decomposed rock which occupies the zone between the lowest soil horizon and firm bedrock).
- Medium soft - Slight erosion of core, slightly gouged by screwdriver, or breaks with crumbly edges from single hammer blow.
- Medium hard - No core erosion, easily scratched by screwdriver, or breaks with sharp edges from single hammer blow.
- Hard - Requires several hammer blows to break and has sharp conchoidal breaks. Cannot be scratched with screwdriver.

Note the difference in usage here of the words "scratch" and "gouge." A scratch shall be considered a slight depression in the rock (do not mistake the scraping off of rock flour from drilling with a scratch in the rock itself), while a gouge is much deeper.

5.3.5 Fracturing

The degree of fracturing or brokenness of a rock is described by measuring the fractures or joint spacing. After eliminating drilling breaks, the average spacing is calculated and the fracturing is described by the following terms:

- Very broken (V. BR.) - Less than 2-inch spacing between fractures
- Broken (BR.) - 2-inch to 1-foot spacing between fractures
- Blocky (BL.) - 1- to 3-foot spacing between fractures
- Massive (M.) - 3 to 10-foot spacing between fractures

Subject BOREHOLE AND SAMPLE LOGGING	Number GH-1.5	Page 14 of 20
	Revision 1	Effective Date 06/99

The structural integrity of the rock can be approximated by calculating the Rock Quality Designation (RQD) of cores recovered. The RQD is determined by adding the total lengths of all pieces exceeding 4 inches and dividing by the total length of the coring run, to obtain a percentage.

Method of Calculating RQD
(After Deere, 1964)

$$RQD \% = r/l \times 100$$

r = Total length of all pieces of the lithologic unit being measured, which are greater than 4 inches length, and have resulted from natural breaks. Natural breaks include slickensides, joints, compaction slicks, bedding plane partings (not caused by drilling), friable zones, etc.

l = Total length of the coring run.

5.3.6 Weathering

The degree of weathering is a significant parameter that is important in determining weathering profiles and is also useful in engineering designs. The following terms can be applied to distinguish the degree of weathering:

- Fresh - Rock shows little or no weathering effect. Fractures or joints have little or no staining and rock has a bright appearance.
- Slight - Rock has some staining which may penetrate several centimeters into the rock. Clay filling of joints may occur. Feldspar grains may show some alteration.
- Moderate - Most of the rock, with exception of quartz grains, is stained. Rock is weakened due to weathering and can be easily broken with hammer.
- Severe - All rock including quartz grains is stained. Some of the rock is weathered to the extent of becoming a soil. Rock is very weak.

5.3.7 Other Characteristics

The following items shall be included in the rock description:

- Description of contact between two rock units. These can be sharp or gradational.
- Stratification (parallel, cross stratified).
- Description of any filled cavities or vugs.
- Cementation (calcareous, siliceous, hematitic).
- Description of any joints or open fractures.
- Observation of the presence of fossils.
- Notation of joints with depth, approximate angle to horizontal, any mineral filling or coating, and degree of weathering.

All information shown on the boring logs shall be neat to the point where it can be reproduced on a copy machine for report presentation. The data shall be kept current to provide control of the drilling program and to indicate various areas requiring special consideration and sampling.

Subject BOREHOLE AND SAMPLE LOGGING	Number GH-1.5	Page 15 of 20
	Revision 1	Effective Date 06/99

5.3.8 Additional Terms Used in the Description of Rock

The following terms are used to further identify rocks:

- Seam - Thin (12 inches or less), probably continuous layer.
- Some - Indicates significant (15 to 40 percent) amounts of the accessory material. For example, rock composed of seams of sandstone (70 percent) and shale (30 percent) would be "sandstone -- some shale seams."
- Few - Indicates insignificant (0 to 15 percent) amounts of the accessory material. For example, rock composed of seam of sandstone (90 percent) and shale (10 percent) would be "sandstone -- few shale seams."
- Interbedded - Used to indicate thin or very thin alternating seams of material occurring in approximately equal amounts. For example, rock composed of thin alternating seams of sandstone (50 percent) and shale (50 percent) would be "interbedded sandstone and shale."
- Interlayered - Used to indicate thick alternating seams of material occurring in approximately equal amounts.

The preceding sections describe the classification of sedimentary rocks. The following are some basic names that are applied to igneous rocks:

- Basalt - A fine-grained extrusive rock composed primarily of calcic plagioclase and pyroxene.
- Rhyolite - A fine-grained volcanic rock containing abundant quartz and orthoclase. The fine-grained equivalent of a granite.
- Granite - A coarse-grained plutonic rock consisting essentially of alkali feldspar and quartz.
- Diorite - A coarse-grained plutonic rock consisting essentially of sodic plagioclase and hornblende.
- Gabbro - A coarse-grained plutonic rock consisting of calcic plagioclase and clinopyroxene. Loosely used for any coarse-grained dark igneous rock.

The following are some basic names that are applied to metamorphic rocks:

- Slate - A very fine-grained foliated rock possessing a well developed slaty cleavage. Contains predominantly chlorite, mica, quartz, and sericite.
- Phyllite - A fine-grained foliated rock that splits into thin flaky sheets with a silky sheen on cleavage surface.
- Schist - A medium to coarse-grained foliated rock with subparallel arrangement of the micaceous minerals which dominate its composition.
- Gneiss - A coarse-grained foliated rock with bands rich in granular and platy minerals.
- Quartzite - A fine- to coarse-grained nonfoliated rock breaking across grains, consisting essentially of quartz sand with silica cement.

Subject BOREHOLE AND SAMPLE LOGGING	Number GH-1.5	Page 16 of 20
	Revision 1	Effective Date 06/99

5.4 Abbreviations

Abbreviations may be used in the description of a rock or soil. However, they shall be kept at a minimum. Following are some of the abbreviations that may be used:

C - Coarse	Lt - Light	Yl - Yellow
Med - Medium	BR - Broken	Or - Orange
F - Fine	BL - Blocky	SS - Sandstone
V - Very	M - Massive	Sh - Shale
Sl - Slight	Br - Brown	LS - Limestone
Occ - Occasional	Bl - Black	Fgr - Fine-grained
Tr - Trace		

5.5 Boring Logs and Documentation

This section describes in more detail the procedures to be used in completing boring logs in the field. Information obtained from the preceding sections shall be used to complete the logs. A sample boring log has been provided as Figure 5.

The field geologist/engineer shall use this example as a guide in completing each boring log. Each boring log shall be fully described by the geologist/engineer as the boring is being drilled. Every sheet contains space for 25 feet of log. Information regarding classification details is provided either on the back of the boring log or on a separate sheet, for field use.

5.5.1 Soil Classification

- Identify site name, boring number, job number, etc. Elevations and water level data to be entered when surveyed data is available.
- Enter sample number (from SPT) under appropriate column. Enter depth sample was taken from (1 block = 1 foot). Fractional footages, i.e., change of lithology at 13.7 feet, shall be lined off at the proportional location between the 13- and 14-foot marks. Enter blow counts (Standard Penetration Resistance) diagonally (as shown). Standard penetration resistance is covered in Section 5.2.3.
- Determine sample recovery/sample length as shown. Measure the total length of sample recovered from the split-spoon sampler, including material in the drive shoe. Do not include cuttings or wash material that may be in the upper portion of the sample tube.
- Indicate any change in lithology by drawing a line at the appropriate depth. For example, if clayey silt was encountered from 0 to 5.5 feet and shale from 5.5 to 6.0 feet, a line shall be drawn at this increment. This information is helpful in the construction of cross-sections. As an alternative, symbols may be used to identify each change in lithology.
- The density of granular soils is obtained by adding the number of blows for the last two increments. Refer to Density of Granular Soils Chart on back of log sheet. For consistency of cohesive soils refer also to the back of log sheet - Consistency of Cohesive Soils. Enter this information under the appropriate column. Refer to Section 5.2.3.

FIGURE 5
COMPLETED BORING LOG (EXAMPLE)



BORING LOG

PROJECT NAME: NSB - SITE BORING NUMBER: SB/MW1
 PROJECT NUMBER: 9594 DATE: 3/8/96
 DRILLING COMPANY: SOILTEST CO. GEOLOGIST: SJ CONTI
 DRILLING RIG: CME-55 DRILLER: R. ROCK

Sample No. and Type or RQD	Depth (Ft.) or Run No.	Blows / 6" or RQD (%)	Sample Recovery / Sample Length	Lithology Change (Depth/Ft.) or Screened Interval	MATERIAL DESCRIPTION			U S C S *	Remarks	PID/FID Reading (ppm)			
					Soil Density/ Consistency or Rock Hardness	Color	Material Classification			Sample	Sampler BZ	Borehole**	Driller BZ**
S-1 e 0800	0.0 2.0	7 6 9 10	1.5/2.0		M DENSE	BRN TO BLK	SILTY SAND - SOME ROCK FR. - TR BRICKS (FILL)	SM	MOIST SL. ORG. ODOR FILL TO 4'±	5	0	0	0
S-2 e 0810	4.0 6.0	5 7 9 8	2.9/2.0	4.0	M DENSE	BRN	SILTY SAND - TR FINE GRAVEL	SM	MOIST - W ODOR NAT. MATL. TOOK SAMPLE SB01-0406 FOR ANALYSIS	10	0	-	-
S-3 e 0820	8.0 10.0	6 8 17 16	1.9/2.0	7.0 8.0	DENSE	TAN BRN	FINE TO COARSE SAND TR. F. GRAVEL	SW	WET HIT WATER @ 7'±	0	0	0	0
S-4 e 0830	12.0 14.0	7 6 5 8	1.6/2.0	12.0	STIFF	GRAY	SILTY CLAY	CL	MOIST → WET	0	5	-	-
	15.0			15.0					AUGER REF @ 15'				
	16.0			16.0	M HARD	BRN	SILTSTONE	VER	WEATHERED				
	17.0			17.0					LO *JNTS @ 15.5 WATER STAINS @ 16.5, 17.1, 17.5	0	0	0	0
	18.0			18.0					LOSING SOME				
	19.0			19.0	HARD	GRAY	SANDSTONE - SOME SILTSTONE	BR	DRILL H ₂ O @ 17'±				
	20.0			20.0					SET TEMP 6" CAS TO 15.5				
	21.0			21.0					SET 2"Ø PVC SCREEN 16-25	0	0	0	0
	22.0			22.0					SAND 14-25				
	23.0			23.0					PELLETS 12-14				

* When rock coring, enter rock brokenness.
 ** Include monitor reading in 6 foot intervals @ borehole. Increase reading frequency if elevated response read.
 Remarks: CME-55 RIG, 4 1/4" ID HSA - 9" OD ± • 1-20Z
2" SPLIT SPOONS - 140 LB HAMMER - 30" DROP 1-80Z Drilling Area
NIX CORE IN BEDROCK RUN (1) = 25 min, RUN (2) = 15 min Background (ppm):
 Converted to Well: Yes No Well I.D. #: MW-1

Subject BOREHOLE AND SAMPLE LOGGING	Number GH-1.5	Page 18 of 20
	Revision 1	Effective Date 06/99

- Enter color of the material in the appropriate column.
- Describe material using the USCS. Limit this column for sample description only. The predominant material is described last. If the primary soil is silt but has fines (clay) - use clayey silt. Limit soil descriptors to the following:
 - Trace: 0 - 10 percent
 - Some: 11 - 30 percent
 - And/Or: 31 - 50 percent
- Also indicate under Material Classification if the material is fill or natural soils. Indicate roots, organic material, etc.
- Enter USCS symbol - use chart on back of boring log as a guide. If the soils fall into one of two basic groups, a borderline symbol may be used with the two symbols separated by a slash. For example ML/CL or SM/SP.
- The following information shall be entered under the "Remarks" column and shall include, but is not limited by, the following:
 - Moisture - estimate moisture content using the following terms - dry, moist, wet and saturated. These terms are determined by the individual. Whatever method is used to determine moisture, be consistent throughout the log.
 - Angularity - describe angularity of coarse grained particles using the terms angular, subangular, subrounded, or rounded. Refer to ASTM D 2488 or Earth Manual for criteria for these terms.
 - Particle shape - flat, elongated, or flat and elongated.
 - Maximum particle size or dimension.
 - Water level observations.
 - Reaction with HCl - none, weak, or strong.
- Additional comments:
 - Indicate presence of mica, caving of hole, when water was encountered, difficulty in drilling, loss or gain of water.
 - Indicate odor and Photoionization Detector (PID) or Flame Ionization Detector (FID) reading if applicable.
 - Indicate any change in lithology by drawing a line through the lithology change column and indicate the depth. This will help when cross-sections are subsequently constructed.
 - At the bottom of the page indicate type of rig, drilling method, hammer size and drop, and any other useful information (i.e., borehole size, casing set, changes in drilling method).

Subject BOREHOLE AND SAMPLE LOGGING	Number GH-1.5	Page 19 of 20
	Revision 1	Effective Date 06/99

- Vertical lines shall be drawn (as shown in Figure 5) in columns 6 to 8 from the bottom of each sample to the top of the next sample to indicate consistency of material from sample to sample, if the material is consistent. Horizontal lines shall be drawn if there is a change in lithology, then vertical lines drawn to that point.
- Indicate screened interval of well, as needed, in the lithology column. Show top and bottom of screen. Other details of well construction are provided on the well construction forms.

5.5.2 Rock Classification

- Indicate depth at which coring began by drawing a line at the appropriate depth. Indicate core run depths by drawing coring run lines (as shown) under the first and fourth columns on the log sheet. Indicate RQD, core run number, RQD percent, and core recovery under the appropriate columns.
- Indicate lithology change by drawing a line at the appropriate depth as explained in Section 5.5.1.
- Rock hardness is entered under designated column using terms as described on the back of the log or as explained earlier in this section.
- Enter color as determined while the core sample is wet; if the sample is cored by air, the core shall be scraped clean prior to describing color.
- Enter rock type based on sedimentary, igneous or metamorphic. For sedimentary rocks use terms as described in Section 5.3. Again, be consistent in classification. Use modifiers and additional terms as needed. For igneous and metamorphic rock types use terms as described in Sections 5.3.8.
- Enter brokenness of rock or degree of fracturing under the appropriate column using symbols VBR, BR, BL, or M as explained in Section 5.3.5 and as noted on the back of the Boring Log.
- The following information shall be entered under the remarks column. Items shall include but are not limited to the following:
 - Indicate depths of joints, fractures and breaks and also approximate to horizontal angle (such as high, low), i.e., 70° angle from horizontal, high angle.
 - Indicate calcareous zones, description of any cavities or vugs.
 - Indicate any loss or gain of drill water.
 - Indicate drop of drill tools or change in color of drill water.
- Remarks at the bottom of Boring Log shall include:
 - Type and size of core obtained.
 - Depth casing was set.
 - Type of rig used.
- As a final check the boring log shall include the following:
 - Vertical lines shall be drawn as explained for soil classification to indicate consistency of bedrock material.
 - If applicable, indicate screened interval in the lithology column. Show top and bottom of screen. Other details of well construction are provided on the well construction forms.

Subject BOREHOLE AND SAMPLE LOGGING	Number GH-1.5	Page 20 of 20
	Revision 1	Effective Date 06/99

5.5.3 Classification of Soil and Rock from Drill Cuttings

The previous sections describe procedures for classifying soil and rock samples when cores are obtained. However, some drilling methods (air/mud rotary) may require classification and borehole logging based on identifying drill cuttings removed from the borehole. Such cuttings provide only general information on subsurface lithology. Some procedures that shall be followed when logging cuttings are:

- Obtain cutting samples at approximately 5-foot intervals, sieve the cuttings (if mud rotary drilling) to obtain a cleaner sample, place the sample into a small sample bottle or "zip lock" bag for future reference, and label the jar or bag (i.e. hole number, depth, date, etc.). Cuttings shall be closely examined to determine general lithology.
- Note any change in color of drilling fluid or cuttings, to estimate changes in lithology.
- Note drop or chattering of drilling tools or a change in the rate of drilling, to determine fracture locations or lithologic changes.
- Observe loss or gain of drilling fluids or air (if air rotary methods are used), to identify potential fracture zones.
- Record this and any other useful information onto the boring log as provided in Figure 1.

This logging provides a general description of subsurface lithology and adequate information can be obtained through careful observation of the drilling process. It is recommended that split-barrel and rock core sampling methods be used at selected boring locations during the field investigation to provide detailed information to supplement the less detailed data generated through borings drilled using air/mud rotary methods.

5.6 Review

Upon completion of the borings logs, copies shall be made and reviewed. Items to be reviewed include:

- Checking for consistency of all logs.
- Checking for conformance to the guideline.
- Checking to see that all information is entered in their respective columns and spaces.

6.0 REFERENCES

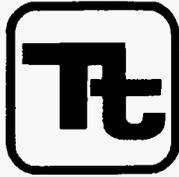
Unified Soil Classification System (USCS).

ASTM D2488, 1985.

Earth Manual, U.S. Department of the Interior, 1974.

7.0 RECORDS

Originals of the boring logs shall be retained in the project files.



TETRA TECH NUS, INC.

STANDARD OPERATING PROCEDURES

Number GH-2.8	Page 1 of 12
Effective Date 09/03	Revision 3
Applicability Tetra Tech NUS, Inc.	
Prepared Earth Sciences Department	
Approved D. Senovich <i>DS</i>	

Subject
GROUNDWATER MONITORING WELL INSTALLATION

TABLE OF CONTENTS

<u>SECTION</u>	<u>PAGE</u>
1.0 PURPOSE.....	2
2.0 SCOPE.....	2
3.0 GLOSSARY	2
4.0 RESPONSIBILITIES.....	2
5.0 PROCEDURES.....	3
5.1 EQUIPMENT/ITEMS NEEDED.....	3
5.2 WELL DESIGN.....	3
5.2.1 Well Depth, Diameter, and Monitored Interval	3
5.2.2 Riser Pipe and Screen Materials.....	5
5.2.3 Annular Materials	6
5.2.4 Protective Casing	6
5.3 MONITORING WELL INSTALLATION	7
5.3.1 Monitoring Wells in Unconsolidated Sediments	7
5.3.2 Confining Layer Monitoring Wells.....	7
5.3.3 Bedrock Monitoring Wells	8
5.3.4 Drive Points.....	8
5.3.5 Innovative Monitoring Well Installation Techniques	8
5.4 WELL DEVELOPMENT METHODS	8
5.4.1 Overpumping and Backwashing	8
5.4.2 Surging with a Surge Plunger.....	9
5.4.3 Compressed Air	9
5.4.4 High Velocity Jetting.....	9
6.0 RECORDS	9
7.0 REFERENCES.....	10
 <u>ATTACHMENTS</u>	
A RELATIVE COMPATIBILITY OF RIGID WELL-CASING MATERIAL (PERCENT) / RELATIVE COMPATIBILITY OF SEMI-RIGID OR ELASTOMERIC MATERIALS (PERCENT).....	11
B COMPARISON OF STAINLESS STEEL AND PVC FOR MONITORING WELL CONSTRUCTION.....	12

Subject GROUNDWATER MONITORING WELL INSTALLATION	Number GH-2.8	Page 2 of 12
	Revision 3	Effective Date 09/03

1.0 PURPOSE

This procedure provides general guidance and information pertaining to proper monitoring well design, installation, and development.

2.0 SCOPE

This procedure is applicable to the construction of monitoring wells. The methods described herein may be modified by project-specific requirements for monitoring well construction. In addition, many regulatory agencies have specific regulations pertaining to monitoring well construction and permitting. These requirements must be determined during the project planning phases of the investigation, and any required permits must be obtained before field work begins. Innovative monitoring well installation techniques, which typically are not used, will be discussed only generally in this procedure.

3.0 GLOSSARY

Monitoring Well - A well which is screened, cased, and sealed which is capable of providing a groundwater level and groundwater sample representative of the zone being monitored. Some monitoring wells may be constructed as open boreholes.

Piezometer - A pipe or tube inserted into the water bearing zone, typically open to water flow at the bottom and to the atmosphere at the top, and used to measure water level elevations. Piezometers may range in size from 1/2-inch-diameter plastic tubes to well points or monitoring wells.

Potentiometric Surface - The surface representative of the level to which water will rise in a well cased to the screened aquifer.

Well Point (Drive Point) - A screened or perforated tube (Typically 1-1/4 or 2 inches in diameter) with a solid, conical, hardened point at one end, which is attached to a riser pipe and driven into the ground with a sledge hammer, drop weight, or mechanical vibrator. Well points may be used for groundwater injection and recovery, as piezometers (i.e., to measure water levels) or to provide groundwater samples for water quality data.

4.0 RESPONSIBILITIES

Driller - The driller provides adequate and operable equipment, sufficient quantities of materials, and an experienced and efficient labor force capable of performing all phases of proper monitoring well installation and construction. The driller may also be responsible for obtaining, in advance, any required permits for monitoring well installation and construction.

Field Geologist - The field geologist supervises and documents well installation and construction performed by the driller, and insures that well construction is adequate to provide representative groundwater data from the monitored interval. Geotechnical engineers, field technicians, or other suitable trained personnel may also serve in this capacity.

Subject GROUNDWATER MONITORING WELL INSTALLATION	Number GH-2.8	Page 3 of 12
	Revision 3	Effective Date 09/03

5.0 PROCEDURES

5.1 Equipment/Items Needed

Below is a list of items that may be needed when installing a monitoring well or piezometer:

- Health and safety equipment (hard hats, safety glasses, etc.) as required by the Site Safety Officer.
- Well drilling and installation equipment with associated materials (typically supplied by the driller).
- Hydrogeologic equipment (weighted engineer's tape, water level indicator, retractable engineers rule, electronic calculator, clipboard, mirror and flashlight - for observing downhole activities, paint and ink marker for marking monitoring wells, sample jars, well installation forms, and a field notebook).
- Drive point installation tools (sledge hammer, drop hammer, or mechanical vibrator; tripod, pipe wrenches, drive points, riser pipe, and end caps).

5.2 Well Design

The objectives and intended use for each monitoring well must be clearly defined before the monitoring system is designed. Within the monitoring system, different monitoring wells may serve different purposes and, therefore, require different types of construction. During all phases of the well design, attention must be given to clearly documenting the basis for design decisions, the details of well construction, and the materials used. The objectives for installing the monitoring wells may include:

- Determining groundwater flow directions and velocities.
- Sampling or monitoring for trace contaminants.
- Determining aquifer characteristics (e.g., hydraulic conductivity).

Siting of monitoring wells shall be performed after a preliminary estimation of the groundwater flow direction. In most cases, groundwater flow directions and potential well locations can be determined by an experienced hydrogeologist through the review of geologic data and the site terrain. In addition, data from production wells or other monitoring wells in the area may be used to determine the groundwater flow direction. If these methods cannot be used, piezometers, which are relatively inexpensive to install, may have to be installed in a preliminary investigative phase to determine groundwater flow direction.

5.2.1 Well Depth, Diameter, and Monitored Interval

The well depth, diameter, and monitored interval must be tailored to the specific monitoring needs of each investigation. Specification of these items generally depends on the purpose of the monitoring system and the characteristics of the hydrogeologic system being monitored. Wells of different depth, diameter, and monitored interval can be employed in the same groundwater monitoring system. For instance, varying the monitored interval in several wells, at the same location (cluster wells) can help to determine the vertical gradient and the depths at which contaminants are present. Conversely, a fully penetrating well is usually not used to quantify or vertically locate a contaminant plume, since groundwater samples collected in wells that are screened over the full thickness of the water-bearing zone will be representative of average conditions across the entire monitored interval. However, fully penetrating wells can be used to establish the existence of contamination in the water-bearing zone. The well diameter desired depends upon the hydraulic characteristics of the water-bearing zone, sampling requirements, drilling method and cost.

Subject GROUNDWATER MONITORING WELL INSTALLATION	Number GH-2.8	Page 4 of 12
	Revision 3	Effective Date 09/03

The decision concerning the monitored interval and well depth is based on the following (and possibly other) information:

- The vertical location of the contaminant source in relation to the water-bearing zone.
- The depth, thickness and uniformity of the water-bearing zone.
- The anticipated depth, thickness, and characteristics (e.g., density relative to water) of the contaminant plume.
- Fluctuation in groundwater levels (due to pumping, tidal influences, or natural recharge/discharge events).
- The presence and location of contaminants encountered during drilling.
- Whether the purpose of the installation is for determining existence or non-existence of contamination or if a particular stratigraphic zone is being investigated.
- The analysis of borehole geophysical logs.

In most situations where groundwater flow lines are horizontal, depending on the purpose of the well and the site conditions, monitored intervals are 20 feet or less. Shorter screen lengths (5 feet or less) are usually required where flow lines are not horizontal, (i.e., if the wells are to be used for accurate measurement of the potentiometric head at a specific point).

Many factors influence the diameter of a monitoring well. The diameter of the monitoring well depends on the application. In determining well diameter, the following needs must be considered:

- Adequate water volume for sampling.
- Drilling methodology.
- Type of sampling device to be used.
- Costs.

Standard monitoring well diameters are 2, 4, 6, or 8 inches. Drive points are typically 1-1/4 or 2 inches in diameter. For monitoring programs which require screened monitoring wells, either a 2-inch or 4-inch-diameter well is preferred. Typically, well diameters greater than 4 inches are used in monitoring programs in which open-hole bedrock monitoring wells are used. With smaller diameter wells, the volume of stagnant water in the well is minimized, and well construction costs are reduced; however, the sampling devices that can be used are limited.

In specifying well diameter, sampling requirements must be considered (up to a total of 4 gallons of water may be required for a single sample to account for full organic and inorganic analyses, and split samples), particularly if the monitored formation is known to be a low-yielding formation. The unit volume of water contained within a monitoring well is dependent on the well diameter as follows:

Casing Inside Diameter (Inch)	Standing Water Length to Obtain 1 Gallon Water (Feet)
2	6.13
4	1.53
6	0.68

If a well recharges quickly after purging, then well diameter may not be an important factor regarding sample volume requirements.

Subject GROUNDWATER MONITORING WELL INSTALLATION	Number GH-2.8	Page 5 of 12
	Revision 3	Effective Date 09/03

Pumping tests for determining aquifer characteristics may require larger diameter wells (for installation of high capacity pumps); however, in small-diameter wells in-situ permeability tests can be performed during drilling or after well installation is completed.

5.2.2 Riser Pipe and Screen Materials

Well materials are specified by diameter, type of material, and thickness of pipe. Well screens require an additional specification of slot size. Thickness of pipe is referred to as "Schedule" for polyvinyl chloride (PVC) casing and is usually Schedule 40 (thinner wall) or 80 (thicker wall). Steel pipe thickness is often referred to as "Strength". Standard Strength is usually adequate for monitoring well purposes. With larger diameter pipe, the wall thickness must be greater to maintain adequate strength. The required thickness is also dependent on the method of installation; risers for drive points require greater strength than wells installed inside drilled borings.

The selection of well screen and riser materials depends on the method of drilling, the type of subsurface materials the well penetrates, the type of contamination expected, and natural water quality and depth. Cost and the level of accuracy required are also important. The materials generally available are Teflon, stainless steel, PVC galvanized steel, and carbon steel. Each has advantages and limitations (see Attachment A of this guideline for an extensive presentation on this topic). The two most commonly used materials are PVC and stainless steel. Properties of these two materials are compared in Attachment B. Stainless steel is a good choice where trace metals or organic sampling is required; however, costs are high. Teflon materials are extremely expensive, but are relatively inert and provide the least opportunity for water contamination due to well materials. PVC has many advantages, including low cost, excellent availability, light weight, ease of manipulation, and widespread acceptance. The crushing strength of PVC may limit the depth of installation, but the use of Schedule 80 materials may overcome some of the problems associated with depth. However, the smaller inside diameter of Schedule 80 pipe may be an important factor when considering the size of bailers or pumps required for sampling or testing. Due to this problem, the minimum well pipe size recommended for Schedule 80 wells is 4-inch I.D.

Screens and risers may have to be decontaminated before use because oil-based preservatives and oil used during thread cutting and screen manufacturing may contaminate samples. Metal pipe may corrode and release metal ions or chemically react with organic constituents, but this is considered a minor issue. Galvanized steel is not recommended where samples may be collected for metals analyses, as zinc and cadmium levels in groundwater samples may become elevated from leaching of the zinc coating.

Threaded, flush-joint casing is most often preferred for monitoring well applications. PVC, Teflon, and steel can all be obtained with threaded joints. Welded-joint steel casing is also acceptable. Glued PVC may release organic contaminants into the well, and therefore, should not be used if the well is to be sampled for organic constituents.

When the water-bearing zone is in consolidated bedrock, such as limestone or fractured granite, a well screen is often not necessary (the well is simply an open hole in bedrock). Unconsolidated materials, such as sands, clay, and silts require a screen. A screen slot size of 0.010 or 0.020 inch is generally used when a screen is necessary, and the annular borehole space around the screened interval is artificially packed with an appropriately sized sand, selected based on formation grain size. The slot size controls the quantity of water entering the well and prevents entry of natural materials or sand pack. The screen shall pass no more than 10 percent of the pack material, or in-situ aquifer material. The site geologist shall specify the combination of screen slot size and sand pack which will be compatible with the water-bearing zone, to maximize groundwater inflow and minimize head losses and movement of fines into the wells. For example, as a standard procedure, a Morie No. 1 or No. 10 to No. 20 U.S. Standard Sieve size filter pack is typically appropriate for a 0.020-inch slot screen; however, a No. 20 to No. 40 U.S. Standard Sieve size filter pack is typically appropriate for a 0.010-inch slot screen.

Subject GROUNDWATER MONITORING WELL INSTALLATION	Number GH-2.8	Page 6 of 12
	Revision 3	Effective Date 09/03

5.2.3 Annular Materials

Materials placed in the annular space between the borehole and riser pipe and screen include a sand pack when necessary, a bentonite seal, and cement-bentonite grout. The sand pack is usually a medium-to coarse-grained poorly graded, silica sand and should relate to the grain size of the aquifer sediments. The quantity of sand placed in the annular space is dependent upon the length of the screened interval, but should always extend at least 1 foot above the top of the screen. At least 1 to 3 feet of bentonite pellets or equivalent shall be placed above the sand pack. Cement-bentonite grout (or equivalent) is then placed to extent from the top of the bentonite pellets to the ground surface.

On occasion, and with the concurrence of the involved regulatory agencies, monitoring wells may be packed naturally (i.e., no artificial sand pack installed). In this case, the natural formation material is allowed to collapse around the well screen after the well is installed. This method has been used where the formation material itself is a relatively uniform grain size, or when artificial sand packing is not possible due to borehole collapse.

Bentonite expands by absorbing water and provides a seal between the screened interval and the overlying portion of the annular space and formation. Cement-bentonite grout is placed on top of the bentonite pellets, extending to the surface. The grout effectively seals the remaining borehole annulus and eliminates the possibility for surface infiltration reaching the screened interval. Grouting also replaces material removed during drilling and prevents hole collapse and subsidence around the well. A tremie pipe should be used to introduce grout from the bottom upward, to prevent bridging, and to provide a better seal. In shallow boreholes that don't collapse, it may be more practical to pour the grout from the surface without a tremie pipe.

Grout is a general term which has several different connotations. For all practical purposes within the monitoring well installation industry, grout refers to the solidified material which is installed and occupies the annular space above the bentonite pellet seal. Grout, most of the time, is made up of one or two assemblages of material, (e.g., cement and/or bentonite). A cement-bentonite grout, which is the most common type of grout used in monitoring well completions, normally is a mixture of cement, bentonite, and water at a ratio of one 90-pound bag of Portland Type I cement, plus 3 to 5 pounds of granular or flake-type bentonite, and 6-7 gallons of water. A neat cement consists of one ninety-pound bag of Portland Type I cement and 6-7 gallons of water. A bentonite slurry (bentonite and water mixed to a thick but pumpable mixture) is sometimes used instead of grout for deep well installations where placement of bentonite pellets is difficult. Bentonite chips are also occasionally used for annular backfill in place of grout.

In certain cases, the borehole may be drilled to a depth greater than the anticipated well installation depth. For these cases, the well shall be backfilled to the desired depth with bentonite pellets/chips or sand. A short (1- to 2-foot) section of capped riser pipe sump is sometimes installed immediately below the screen, as a silt reservoir, when significant post-development silting is anticipated. This will ensure that the entire screen surface remains unobstructed.

5.2.4 Protective Casing

When the well is completed and grouted to the surface, a protective steel casing is typically placed over the top of the well. This casing generally has a hinged cap and can be locked to prevent vandalism. The protective casing has a larger diameter than the well and is set into the wet cement grout over the well upon completion. In addition, one hole is drilled just above the cement collar through the protective casing which acts as a weep hole for the flow of water which may enter the annulus during well development, purging, or sampling.

Subject GROUNDWATER MONITORING WELL INSTALLATION	Number GH-2.8	Page 7 of 12
	Revision 3	Effective Date 09/03

A protective casing which is level with the ground surface (flush-mounted) is used in roadway or parking lot applications where the top of a monitoring well must be below the pavement. The top of the riser pipe is placed 4 to 5 inches below the pavement, and a locking protective casing is cemented in place to 3 inches below the pavement. A large diameter, manhole-type protective collar is set into the wet cement around the well with the top set level with or slightly above the pavement. An appropriately-sized id is placed over the protective sleeve. The cement should be slightly mounded to direct pooled water away from the well head.

5.3 Monitoring Well Installation

Pertinent data regarding monitoring well installation shall be recorded on log sheets as depicted and discussed in SOP SA-6.3. Attachments to this referenced SOP illustrate terms and physical construction of various types of monitoring wells.

5.3.1 **Monitoring Wells in Unconsolidated Sediments**

After the borehole is drilled to the desired depth, well installation can begin. The procedure for well installation will partially be dictated by the stability of the formation in which the well is being placed. If the borehole collapses immediately after the drilling tools are withdrawn, then a temporary casing must be installed and well installation will proceed through the center of the temporary casing, and continue as the temporary casing is withdrawn from the borehole. In the case of hollow-stem auger drilling, the augers will act to stabilize the borehole during well installation.

Before the screen and riser pipe are lowered into the borehole, all pipe and screen sections should be measured with an engineer's rule to ensure proper placement. When measuring sections, the threads on one end of the pipe or screen must be excluded while measuring, since the pipe and screen sections are screwed flush together.

After the screen and riser pipe are lowered through the temporary casing, the sand pack can be installed. A weighted tape measure must be used during the installation procedure to carefully monitor installation progress. The sand is slowly poured into the annulus between the riser pipe and temporary casing, as the casing is withdrawn. Sand should always be kept within the temporary casing during withdrawal in order to ensure an adequate sand pack. However, if too much sand is within the temporary casing (greater than 1 foot above the bottom of the casing) bridging between the temporary casing and riser pipe may occur. Centralizers may be used at the geologist's discretion, one above and one below the screen, to assure enough annular space for sand pack placement.

After the sand pack is installed to the desired depth (at least 1 foot above the top of the screen), then the bentonite pellet seal (or equivalent), can be installed in the same manner as the sand pack. At least 1 to 3 feet of bentonite pellets should be installed above the sand pack. Pellets should be added slowly and their fall monitored closely to ensure that bridging does not occur.

The cement-bentonite grout is then mixed and tremied into the annulus as the temporary casing or augers are withdrawn. Finally, the protective casing can be installed as detailed in Section 5.2.4.

5.3.2 **Confining Layer Monitoring Wells**

When drilling and installing a well in a confined aquifer, proper well installation techniques must be applied to avoid cross contamination between aquifers. Under most conditions, this can be accomplished by installing double-cased wells. This is accomplished by drilling a large-diameter boring through the upper aquifer, 1 to 5 feet into the underlying confining layer, and setting and pressure grouting or tremie grouting a large-diameter casing into the confining layer. The grout material must fill the space between the native material and the outer casing. A smaller diameter boring is then continued through the confining layer for

Subject GROUNDWATER MONITORING WELL INSTALLATION	Number GH-2.8	Page 8 of 12
	Revision 3	Effective Date 09/03

installation of the monitoring well as detailed for overburden monitoring wells. Sufficient time (determined by the field geologist), must be allowed for setting of the grout prior to drilling through the confined layer.

5.3.3 Bedrock Monitoring Wells

When installing bedrock monitoring wells, a large diameter boring is drilled through the overburden and approximately 5 –10 feet into bedrock. A casing (typically steel) is installed and either pressure grouted or tremie grouted in place. After the grout has cured, a smaller diameter boring is continued into bedrock to the desired depth. If the boring does not collapse, the well can be left open, and a screen is not necessary. If the boring collapses, then a screen is required and can be installed as detailed for overburden monitoring wells. If a screen is to be used, then the casing which is installed through the overburden and into the bedrock does not require grouting and can be removed when the final well installation is completed.

5.3.4 Drive Points

Drive points can be installed with either a sledge hammer, drop hammer, or a mechanical vibrator. The screen section is threaded and tightened onto the riser pipe with pipe wrenches. The drive point is simply pounded into the subsurface to the desired depth. If a heavy drop hammer is used, then a tripod and pulley setup is required to lift the hammer. Drive points typically cannot be manually driven to depths exceeding 10 feet.

Direct push sampling/monitoring point installation methods, using a direct push rig or drilling rig, are described in SOP SA-2.5.

5.3.5 Innovative Monitoring Well Installation Techniques

Certain innovative sampling devices have proven advantageous. These devices are essentially screened samplers installed in a borehole with only small-diameter tubes extending to the surface. This reduces drilling costs, decreases the volume of stagnant water, and provides a sampling system that minimizes cross-contamination from sampling equipment. Four manufacturers of these samplers include Timco Manufacturing Company, Inc., of Prairie du Sac, Wisconsin, BARCAD Systems, Inc., of Concord, Massachusetts, Westbay Instruments Ltd. of Vancouver, British Columbia, Canada and the University of Waterloo at Waterloo, Ontario, Canada.. Each manufacturer offers various construction materials.

5.4 Well Development Methods

The purpose of well development is to stabilize and increase the permeability of the gravel pack around the well screen, and to restore the permeability of the formation which may have been reduced by drilling operations. Wells are typically developed until all fine material and drilling water is removed from the well. Sequential measurements of pH, conductivity, turbidity, and temperature taken during development may yield information (stabilized values) regarding whether sufficient development has been performed. The selection of the well development method shall be made by the field geologist and is based on the drilling methods, well construction and installation details, and the characteristics of the formation that the well is screened in. The primary methods of well development are summarized below. A more detailed discussion may be found in Driscoll (1986).

5.4.1 Overpumping and Backwashing

Wells may be developed by alternatively drawing the water level down at a high rate (by pumping or bailing) and then reversing the flow direction (backwashing) so that water is passing from the well into the formation. This back and forth movement of water through the well screen and gravel pack serves to

Subject GROUNDWATER MONITORING WELL INSTALLATION	Number GH-2.8	Page 9 of 12
	Revision 3	Effective Date 09/03

remove fines from the formation immediately adjacent to the well, while preventing bridging (wedging) of sand grains. Backwashing can be accomplished by several methods, including pouring water into the well and then bailing, starting and stopping a pump intermittently to change water levels, or forcing water into the well under pressure through a water-tight fitting ("rawhiding"). Care should be taken when backwashing not to apply too much pressure, which could damage or destroy the well screen.

5.4.2 Surging with a Surge Plunger

A surge plunger (also called a surge block) is approximately the same diameter as the well casing and is aggressively moved up and down within the well to agitate the water, causing it to move in and out of the screens. This movement of water pulls fine materials into the well, where they may be removed by any of several methods, and prevents bridging of sand particles in the gravel pack. There are two basic types of surge plungers; solid and valved surge plungers. In formations with low yields, a valved surge plunger may be preferred, as solid plungers tend to force water out of the well at a greater rate than it will flow back in. Valved plungers are designed to produce a greater inflow than outflow of water during surging.

5.4.3 Compressed Air

Compressed air can be used to develop a well by either of two methods: backwashing or surging. Backwashing is done by forcing water out through the screens, using increasing air pressure inside a sealed well, then releasing the pressurized air to allow the water to flow back into the well. Care should be taken when using this method so that the water level does not drop below the top of the screen, thus introducing air into the formation and reducing well yield. Surging, or the "open well" method, consists of alternately releasing large volumes of air suddenly into an open well below the water level to produce a strong surge by virtue of the resistance of water head, friction, and inertia. Pumping of the well is subsequently done using the air lift method.

5.4.4 High Velocity Jetting

In the high velocity jetting method, water is forced at high velocities from a plunger-type device and through the well screen to loosen fine particles from the sand pack and surrounding formation. The jetting tool is slowly rotated and raised and lowered along the length of the well screen to develop the entire screened area. Jetting using a hose lowered into the well may also be effective. The fines washed into the screen during this process can then be bailed or pumped from the well.

6.0 RECORDS

A critical part of monitoring well installation is recording of all significant details and events in the site logbook or field notebook. The geologist must record the exact depths of significant hydrogeological features, screen placement, gravel pack placement, and bentonite placement.

A Monitoring Well Sheet (see Attachments to SOP SA-6.3) shall be completed, ensuring the uniform recording of data for each installation and rapid identification of missing information. Well depth, length, materials of construction, length and openings of screen, length and type of riser, and depth and type of all backfill materials shall be recorded. Additional information shall include location, installation date, problems encountered, water levels before and after well installation, cross-reference to the geologic boring log, and methods used during the installation and development process. Documentation is very important to prevent problems involving questionable sample validity. Somewhat different information will need to be recorded, depending on whether the well is completed in overburden (single- or double-cased), as a cased well in bedrock, or as an open hole in bedrock.

The quantities of sand, bentonite, and grout placed in the well are also important. The geologist shall calculate the annular space volume and have an idea of the quantity of material needed to fill the annular

Subject GROUNDWATER MONITORING WELL INSTALLATION	Number GH-2.8	Page 10 of 12
	Revision 3	Effective Date 09/03

space. Volumes of backfill significantly higher than the calculated volume may indicate a problem such as a large cavity, while a smaller backfill volume may indicate a cave-in or bridging of the backfill materials. Any problems with rig operation or down-time shall be recorded and may affect the driller's final fee.

7.0 REFERENCES

Scalf, M. R., J. F. McNabb, W. J. Dunlap, R. L. Cosby, and J. Fryberger, 1981. Manual of Groundwater Sampling Procedures. R. S. Kerr Environmental Research Laboratory, Office of Research and Development, U.S. EPA, Ada, Oklahoma.

Barcelona, M. J., P. P. Gibb and R. A. Miller, 1983. A Guide to the selection of Materials for Monitoring Well Construction and Groundwater Sampling. ISWS Contract Report 327, Illinois State Water Survey, Champaign, Illinois.

U.S. EPA, 1980. Procedures Manual for Groundwater Monitoring of Solid Waste Disposal Facilities. Publication SW-611, Office of Solid Waste, U.S. EPA, Washington, D.C.

Driscoll, Fletcher G., 1986. Groundwater and Wells. Johnson Division, St. Paul, Minnesota, 1989.

ATTACHMENT A

RELATIVE COMPATIBILITY OF RIGID WELL CASING MATERIAL (PERCENT)

Potentially-Deteriorating Substance	Type of Casing Material						
	PVC 1	Galvanized Steel	Carbon Steel	Lo-carbon Steel	Stainless Steel 304	Stainless Steel 316	Teflon*
Buffered Weak Acid	100	56	51	59	97	100	100
Weak Acid	98	59	43	47	96	100	100
Mineral Acid/ High Solids Content	100	48	57	60	80	82	100
Aqueous/Organic Mixtures	64	69	73	73	98	100	100
Percent Overall Rating	91	58	56	59	93	96	100

Preliminary Ranking of Rigid Materials:

- | | |
|------------------------|--------------------|
| 1 Teflon [®] | 5 Lo-Carbon Steel |
| 2 Stainless Steel 316 | 6 Galvanized Steel |
| 3. Stainless Steel 304 | 7 Carbon Steel |
| 4 PVC 1 | |

* Trademark of DuPont

RELATIVE COMPATIBILITY OF SEMI-RIGID OR ELASTOMERIC MATERIALS (PERCENT)

Potentially-Deteriorating Substance	Type of Casing Material								
	PVC Flexible	PP	PE Conv.	PE Linear	PMM	Viton ^{®*}	Silicone	Neoprene	Teflon ^{®*}
Buffered Weak Acid	97	97	100	97	90	92	87	85	100
Weak Acid	92	90	94	96	78	78	75	75	100
Mineral Acid/ High Solids Content	100	100	100	100	95	100	78	82	100
Aqueous/Organic Mixtures	62	71	40	60	49	78	49	44	100
Percent Overall Rating	88	90	84	88	78	87	72	72	100

Preliminary Ranking of Semi-Rigid or Elastomeric Materials:

- | | |
|---------------------------|--------------------------|
| 1 Teflon [®] | 5 PE Conventional |
| 2 Polypropylene (PP) | 6 Plexiglas/Lucite (PMM) |
| 3. PVC Flexible/PE Linear | 7 Silicone/Neoprene |
| 4 Viton [®] | |

* Trademark of DuPont

Source: Barcelona et al., 1983

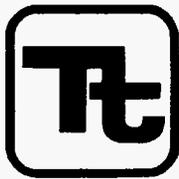
Subject GROUNDWATER MONITORING WELL INSTALLATION	Number GH-2.8	Page 12 of 12
	Revision 3	Effective Date 09/03

ATTACHMENT B

COMPARISON OF STAINLESS STEEL AND PVC FOR MONITORING WELL CONSTRUCTION

Characteristic	Stainless Steel	PVC
Strength	Use in deep wells to prevent compression and closing of screen/riser.	Use when shear and compressive strength are not critical.
Weight	Relatively heavier.	Light-weight; floats in water.
Cost	Relatively expensive.	Relatively inexpensive.
Corrosivity	Deteriorates more rapidly in corrosive water.	Non-corrosive -- may deteriorate in presence of ketones, aromatics, alkyl sulfides, or some chlorinated hydrocarbons.
Ease of Use	Difficult to adjust size or length in the field.	Easy to handle and work with in the field.
Preparation for Use	Should be steam cleaned if organics will be subsequently sampled.	Never use glue fittings -- pipes should be threaded or pressure fitted. Should be steam cleaned when used for monitoring wells.
Interaction with Contaminants*	May sorb organic or inorganic substances when oxidized.	May sorb or release organic substances.

* See also Attachment A.



TETRA TECH NUS, INC.

STANDARD OPERATING PROCEDURES

Number	HS-1.0	Page	1 of 15
Effective Date	12/03	Revision	2
Applicability	Tetra Tech NUS, Inc.		
Prepared	Health & Safety		
Approved	D. Senovich <i>[Signature]</i>		

Subject
UTILITY LOCATING AND EXCAVATION CLEARANCE

TABLE OF CONTENTS

<u>SECTION</u>	<u>PAGE</u>
1.0 PURPOSE	2
2.0 SCOPE	2
3.0 GLOSSARY	2
4.0 RESPONSIBILITIES	3
5.0 PROCEDURES	3
5.1 BURIED UTILITIES	3
5.2 OVERHEAD POWER LINES	5
6.0 UNDERGROUND LOCATING TECHNIQUES	5
6.1 GEOPHYSICAL METHODS	5
6.2 PASSIVE DETECTION SURVEYS	6
6.3 INTRUSIVE DETECTION SURVEYS	6
7.0 INTRUSIVE ACTIVITIES SUMMARY	7
8.0 REFERENCES	8

ATTACHMENTS

1	Listing of Underground Utility Clearance Resources	9
2	Frost Line Penetration Depths by Geographic Location.....	11
3	Utility Clearance Form.....	12
4	OSHA Letter of Interpretation.....	13

Subject UTILITY LOCATING AND EXCAVATION CLEARANCE	Number HS-1.0	Page 2 of 15
	Revision 2	Effective Date 12/03

1.0 PURPOSE

Utilities such as electric service lines, natural or propane gas lines, water and sewage lines, telecommunications, and steam lines are very often in the immediate vicinity of work locations. Contact with underground or overhead utilities can have serious consequences including employee injury/fatality, property and equipment damage, substantial financial impacts, and loss of utility service to users.

The purpose of this procedure is to provide minimum requirements and technical guidelines regarding the appropriate procedures to be followed when performing subsurface and overhead utility locating services. It is the policy of Tetra Tech NUS, Inc. (TtNUS) to provide a safe and healthful work environment for the protection of our employees. The purpose of this Standard Operating Procedure (SOP) is to aid in achieving the objectives of this policy, to present the acceptable procedures pertaining to utility locating and excavation clearance activities, and to present requirements and restrictions relevant to these types of activities. This SOP must be reviewed by any employee potentially involved with underground or overhead utility locating and avoidance activities.

2.0 SCOPE

This procedure applies to all TtNUS field activities where there may be potential contact with underground or overhead utilities. This procedure provides a description of the principles of operation, instrumentation, applicability, and implementability of typical methods used to determine the presence and avoidance of contact with utility services. This procedure is intended to assist with work planning and scheduling, resource planning, field implementation, and subcontractor procurement. Utility locating and excavation clearance requires site-specific information prior to the initiation of any such activities on a specific project. This SOP is not intended to provide a detailed description of methodology and instrument operation. Specialized expertise during both planning and execution of several of the methods presented may also be required.

3.0 GLOSSARY

Electromagnetic Induction (EMI) Survey - A geophysical exploration method whereby electromagnetic fields are induced in the ground and the resultant secondary electromagnetic fields are detected as a measure of ground conductivity.

Magnetometer – A device used for precise and sensitive measurements of magnetic fields.

Magnetic Survey – A geophysical survey method that depends on detection of magnetic anomalies caused by the presence of buried ferromagnetic objects.

Metal Detection – A geophysical survey method that is based on electromagnetic coupling caused by underground conductive objects.

Vertical Gradiometer – A magnetometer equipped with two sensors that are vertically separated by a fixed distance. It is best suited to map near surface features and is less susceptible to deep geologic features.

Ground Penetrating Radar – Ground Penetrating Radar (GPR) involves specialized radar equipment whereby a signal is sent into the ground via a transmitter. Some portion of the signal will be reflected from the subsurface material, which is then recorded with a receiver and electronically converted into a graphic picture.

Subject UTILITY LOCATING AND EXCAVATION CLEARANCE	Number HS-1.0	Page 3 of 15
	Revision 2	Effective Date 12/03

4.0 RESPONSIBILITIES

Project Manager (PM)/Task Order Manager (TOM) - Responsible for ensuring that all field activities are conducted in accordance with this procedure.

Site Manager (SM)/Field Operations Leader (FOL) - Responsible for the onsite verification that all field activities are performed in compliance with approved SOPs or as otherwise directed by the approved project plan(s).

Site Health & Safety Officer (SHSO) – Responsible to provide technical assistance and verify full compliance with this SOP. The SHSO is also responsible for reporting any deficiencies to the Corporate Health and Safety Manager (HSM) and to the PM/TOM.

Health & Safety Manager (HSM) – Responsible for preparing, implementing, and modifying corporate health and safety policy and this SOP.

Site Personnel – Responsible for performing their work activities in accordance with this SOP and the TtNUS Health and Safety Policy.

5.0 PROCEDURES

This procedure addresses the requirements and technical procedures that must be performed to minimize the potential for contact with underground and overhead utility services. These procedures are addressed individually from a buried and overhead standpoint.

5.1 Buried Utilities

Buried utilities present a heightened concern because their location is not typically obvious by visual observation, and it is common that their presence and/or location is unknown or incorrectly known on client properties. This procedure must be followed prior to beginning any subsurface probing or excavation that might potentially be in the vicinity of underground utility services. In addition, the Utility Clearance Form (Attachment 3) must be completed for every location or cluster of locations where intrusive activities will occur.

Where the positive identification and de-energizing of underground utilities cannot be obtained and confirmed using the following steps, the PM/TOM is responsible for arranging for the procurement of a qualified, experienced, utility locating subcontractor who will accomplish the utility location and demarcation duties specified herein.

1. A comprehensive review must be made of any available property maps, blue lines, or as-builts prior to site activities. Interviews with local personnel familiar with the area should be performed to provide additional information concerning the location of potential underground utilities. Information regarding utility locations shall be added to project maps upon completion of this exercise.
- 2., A visual site inspection must be performed to compare the site plan information to actual field conditions. Any findings must be documented and the site plan/maps revised. The area(s) of proposed excavation or other subsurface activities must be marked at the site in white paint or pin flags to identify those locations of the proposed intrusive activities. The site inspection should focus on locating surface indications of potential underground utilities. Items of interest include the presence of nearby area lights, telephone service, drainage grates, fire hydrants, electrical service vaults/panels, asphalt/concrete scars and patches, and topographical depressions. Note the location of any emergency shut off switches. Any additional information regarding utility

Subject UTILITY LOCATING AND EXCAVATION CLEARANCE	Number HS-1.0	Page 4 of 15
	Revision 2	Effective Date 12/03

locations shall be added to project maps upon completion of this exercise and returned to the PM/TOM.

3. If the planned work is to be conducted on private property (e.g., military installations, manufacturing facilities, etc.) the FOL must identify and contact appropriate facility personnel (e.g., public works or facility engineering) before any intrusive work begins to inquire about (and comply with) property owner requirements. It is important to note that private property owners may require several days to several weeks advance notice prior to locating utilities.
4. If the work location is on public property, the state agency that performs utility clearances must be notified (see Attachment 1). State "one-call" services must be notified prior to commencing fieldwork per their requirements. Most one-call services require, by law, 48- to 72-hour advance notice prior to beginning any excavation. Such services typically assign a "ticket" number to the particular site. This ticket number must be recorded for future reference and is valid for a specific period of time, but may be extended by contacting the service again. The utility service will notify utility representatives who then mark their respective lines within the specified time frame. It should be noted that most military installations own their own utilities but may lease service and maintenance from area providers. Given this situation, "one call" systems may still be required to provide location services on military installations.
5. Utilities must be identified and their locations plainly marked using pin flags, spray paint, or other accepted means. The location of all utilities must be noted on a field sketch for future inclusion on project maps. Utility locations are to be identified using the following industry-standard color code scheme, unless the property owner or utility locator service uses a different color code:

white	excavation/subsurface investigation location
red	electrical
yellow	gas, oil, steam
orange	telephone, communications
blue	water, irrigation, slurry
green	sewer, drain
6. Where utility locations are not confirmed with a high degree of confidence through drawings, schematics, location services, etc., the work area must be thoroughly investigated prior to beginning the excavation. In these situations, utilities must be identified using safe and effective methods such as passive and intrusive surveys, or the use of non-conductive hand tools. Also, in situations where such hand tools are used, they should always be used in conjunction with suitable detection equipment, such as the items described in Section 6.0 of this SOP. Each method has advantages and disadvantages including complexity, applicability, and price. It also should be noted that in some states, initial excavation is required by hand to a specified depth.
7. At each location where trenching or excavating will occur using a backhoe or other heavy equipment, and where utility identifications and locations cannot be confirmed prior to groundbreaking, the soil must be probed using a device such as a tile probe which is made of non-conductive material such as fiberglass. If these efforts are not successful in clearing the excavation area of suspect utilities, hand shoveling must be performed for the perimeter of the intended excavation.
8. All utilities uncovered or undermined during excavation must be structurally supported to prevent potential damage. Unless necessary as an emergency corrective measure, TtNUS shall not make any repairs or modifications to existing utility lines without prior permission of the utility owner, property owner, and Corporate HSM. All repairs require that the line be locked-out/tagged-out prior to work.

Subject UTILITY LOCATING AND EXCAVATION CLEARANCE	Number HS-1.0	Page 5 of 15
	Revision 2	Effective Date 12/03

5.2 Overhead Power Lines

If it is necessary to work within the minimum clearance distance of an overhead power line, the overhead line must be de-energized and grounded, or re-routed by the utility company or a registered electrician. If protective measures such as guarding, isolating, or insulating are provided, these precautions must be adequate to prevent employees from contacting such lines directly with any part of their body or indirectly through conductive materials, tools, or equipment.

The following table provides the required minimum clearances for working in proximity to overhead power lines.

<u>Nominal Voltage</u>	<u>Minimum Clearance</u>
0 -50 kV	10 feet, or one mast length; whichever is greater
50+ kV	10 feet plus 4 inches for every 10 kV over 50 kV or 1.5 mast lengths; whichever is greater

6.0 UNDERGROUND LOCATING TECHNIQUES

A variety of supplemental utility locating approaches are available and can be applied when additional assurance is needed. The selection of the appropriate method(s) to employ is site-specific and should be tailored to the anticipated conditions, site and project constraints, and personnel capabilities.

6.1 Geophysical Methods

Geophysical methods include electromagnetic induction, magnetics, and ground penetrating radar. Additional details concerning the design and implementation of electromagnetic induction, magnetics, and ground penetrating radar surveys can be found in one or more of the TtNUS SOPs included in the References (Section 8.0).

Electromagnetic Induction

Electromagnetic Induction (EMI) line locators operate either by locating a background signal or by locating a signal introduced into the utility line using a transmitter. A utility line acts like a radio antenna, producing electrons, which can be picked up with a radiofrequency receiver. Electrical current carrying conductors have a 60HZ signal associated with them. This signal occurs in all power lines regardless of voltage. Utilities in close proximity to power lines or used as grounds may also have a 60HZ signal, which can be picked up with an EM receiver. A typical example of this type of geophysical equipment is an EM-61.

EMI locators specifically designed for utility locating use a special signal that is either indirectly induced onto a utility line by placing the transmitter above the line or directly induced using an induction clamp. The clamp induces a signal on the specific utility and is the preferred method of tracing since there is little chance of the resulting signals being interfered with. A good example of this type of equipment is the Schonstedt® MAC-51B locator. The MAC-51B performs inductively traced surveys, simple magnetic locating, and traced nonmetallic surveys.

When access can be gained inside a conduit to be traced, a flexible insulated trace wire can be used. This is very useful for non-metallic conduits but is limited by the availability of gaining access inside the pipe.

Subject UTILITY LOCATING AND EXCAVATION CLEARANCE	Number HS-1.0	Page 6 of 15
	Revision 2	Effective Date 12/03

Magnetics

Magnetic locators operate by detecting the relative amounts of buried ferrous metal. They are incapable of locating or identifying nonferrous utility lines but can be very useful for locating underground storage tanks (UST's), steel utility lines, and buried electrical lines. A typical example of this type of equipment is the Schonstedt® GA-52Cx locator. The GA-52Cx is capable of locating 4-inch steel pipe up to 8 feet deep.

Non-ferrous lines are often located by using a typical plumbing tool (snake) fed through the line. A signal is then introduced to the snake that is then traced.

Ground Penetrating Radar

Ground Penetrating Radar (GPR) involves specialized radar equipment whereby a signal is sent into the ground via a transmitter. Some portion of the signal will be reflected from the subsurface material, which is then recorded with a receiver and electronically converted into a graphic picture. In general, an object which is harder than the surrounding soil will reflect a stronger signal. Utilities, tunnels, UST's, and footings will reflect a stronger signal than the surrounding soil. Although this surface detection method may determine the location of a utility, this method does not specifically identify utilities (i.e., water vs. gas, electrical vs. telephone); hence, verification may be necessary using other methods. This method is somewhat limited when used in areas with clay soil types or with a high water table.

6.2 Passive Detection Surveys

Acoustic Surveys

Acoustic location methods are generally most applicable to waterlines or gas lines. A highly sensitive Acoustic Receiver listens for background sounds of water flowing (at joints, leaks, etc.) or to sounds introduced into the water main using a transducer. Acoustics may also be applicable to determine the location of plastic gas lines.

Thermal Imaging

Thermal (i.e., infrared) imaging is a passive method for detecting the heat emitted by an object. Electronics in the infrared camera convert subtle heat differentials into a visual image on the viewfinder or a monitor. The operator does not look for an exact temperature; rather they look for heat anomalies (either elevated or suppressed temperatures) characteristic of a potential utility line.

The thermal fingerprint of underground utilities results from differences in temperature between the atmosphere and the fluid present in a pipe or the heat generated by electrical resistance. In addition, infrared scanners may be capable of detecting differences in the compaction, temperature and moisture content of underground utility trenches. High-performance thermal imagery can detect temperature differences to hundredths of a degree.

6.3 Intrusive Detection Surveys

Vacuum Excavation

Vacuum excavation is used to physically expose utility services. The process involves removing the surface material over approximately a 1' x 1' area at the site location. The air-vacuum process proceeds with the simultaneous action of compressed air-jets to loosen soil and vacuum extraction of the resulting

Subject UTILITY LOCATING AND EXCAVATION CLEARANCE	Number HS-1.0	Page 7 of 15
	Revision 2	Effective Date 12/03

debris. This process ensures the integrity of the utility line during the excavation process, as no hammers, blades, or heavy mechanical equipment comes into contact with the utility line, eliminating the risk of damage to utilities. The process continues until the utility is uncovered. Vacuum excavation can be used at the proposed site location to excavate below the "utility window" which is usually 8 feet.

Hand Excavation

When the identification and location of underground utilities cannot be positively confirmed through document reviews and/or other methods, borings and excavations may be cleared via the use of non-conductive hand tools. This should always be done in conjunction with the use of detection equipment. This would be required for all locations where there is a potential to impact buried utilities. The minimum hand-excavation depth that must be reached is to be determined considering the geographical location of the work site. This approach recognizes that the placement of buried utilities is influenced by frost line depths that vary by geographical region. Attachment 2 presents frost line depths for the regions of the contiguous United States. At a minimum, hand excavation depths must be at least to the frost line depth (see Attachment 2) plus two (2) feet, but never less than 4 feet below ground surface (bgs). For hand excavation, the hole created must be reamed large enough to be at least the diameter of the drill rig auger or bit prior to drilling. For soil gas surveys, the survey probe shall be placed as close as possible to the cleared hand excavation. It is important to note that a post-hole digger must not be used in this type of hand excavation activity.

Tile Probe Surveys

For some soil types, site conditions, and excavation requirements, non-conductive tile probes may be used. A tile probe is a "T"-handled rod of varying lengths that can be pushed into the soil to determine if any obstructions exist at that location. Tile probes constructed of fiberglass or other nonconductive material are readily-available from numerous vendors. Tile probes must be performed to the same depth requirements as previously specified. As with other types of hand excavating activities, the use of a non-conductive tile probe, should always be in conjunction with suitable utility locating detection equipment.

7.0 INTRUSIVE ACTIVITIES SUMMARY

The following list summarizes the activities that must be performed prior to beginning subsurface activities:

1. Map and mark all subsurface locations and excavation boundaries using white paint or markers specified by the client or property owner.
2. Notify the property owner and/or client that the locations are marked. At this point, drawings of locations or excavation boundaries shall be provided to the property owner and/or client so they may initiate (if applicable) utility clearance.

Note: Drawings with confirmed locations should be provided to the property owner and/or client as soon as possible to reduce potential time delays.

3. Notify "One Call" service. If possible, arrange for an appointment to show the One Call representative the surface locations or excavation boundaries in person. This will provide a better location designation to the utilities they represent. You should have additional drawings should you need to provide plot plans to the One Call service.
4. Implement supplemental utility detection techniques as necessary and appropriate to conform utility locations or the absence thereof.

Subject UTILITY LOCATING AND EXCAVATION CLEARANCE	Number HS-1.0	Page 8 of 15
	Revision 2	Effective Date 12/03

5. Complete Attachment 3, Utility Clearance Form. This form should be completed for each excavation location. In situations where multiple subsurface locations exist within the close proximity of one another, one form may be used for multiple locations provided those locations are noted on the Utility Clearance Form. Upon completion, the Utility Clearance Form and revised/annotated utility location map becomes part of the project file.

8.0 REFERENCES

OSHA Letter of Interpretation, Mr. Joseph Caldwell, Attachment 4
 OSHA 29 CFR 1926(b)(2)
 OSHA 29 CFR 1926(b)(3)
 TtNUS Utility Locating and Clearance Policy
 TtNUS SOP GH-3.1; Resistivity and Electromagnetic Induction
 TtNUS SOP GH-3.2; Magnetic and Metal Detection Surveys
 TtNUS SOP GH-3.4; Ground-penetrating Radar Surveys

Subject UTILITY LOCATING AND EXCAVATION CLEARANCE	Number HS-1.0	Page 9 of 15
	Revision 2	Effective Date 12/03

**ATTACHMENT 1
LISTING OF UNDERGROUND UTILITY CLEARANCE RESOURCES**



American Public Works Association
2345 Grand Boulevard, Suite 500, Kansas City, MO 64108-2625
Phone (816) 472-6100 • Fax (816) 472-1610
Web www.apwa.net • E-mail apwa@apwa.net

**ONE-CALL SYSTEMS INTERNATIONAL
CONDENSED DIRECTORY**

- | | | |
|---|---|--|
| <p>Alabama
Alabama One-Call
1-800-292-8525</p> | <p>Iowa
Iowa One-Call
1-800-292-8989</p> | <p>New Jersey
New Jersey One Call
1-800-272-1000</p> |
| <p>Alaska
Locate Call Center of Alaska, Inc.
1-800-478-3121</p> | <p>Kansas
Kansas One-Call System, Inc.
1-800-344-7233</p> | <p>New Mexico
New Mexico One Call System, Inc.
1-800-321-2537
Las Cruces- Dona Ana Blue Stakes
1-888-526-0400</p> |
| <p>Arizona
Arizona Blue Stake
1-800-782-5348</p> | <p>Kentucky
Kentucky Underground Protection Inc.
1-800-752-6007</p> | <p>New York
Dig Safely New York
1-800-862-7962
New York City- Long Island One Call
Center
1-800-272-4480</p> |
| <p>Arkansas
Arkansas One Call System, Inc.
1-800-482-8998</p> | <p>Louisiana
Louisiana One Call System, Inc.
1-800-272-3020</p> | <p>North Carolina
The North Carolina One-Call Center,
Inc.
1-800-632-4949</p> |
| <p>California
Underground Service Alert North
1-800-227-2600
Underground Service Alert of Southern
California
1-800-227-2600</p> | <p>Maine
Dig Safe System, Inc.
1-888-344-7233</p> | <p>North Dakota
North Dakota One-Call
1-800-795-0555</p> |
| <p>Colorado
Utility Notification Center of Colorado
1-800-922-1987</p> | <p>Maryland
Miss Utility
1-800-257-7777
Miss Utility of Delmarva
1-800-282-8555</p> | <p>Ohio
Ohio Utilities Protection Service
1-800-362-2764
Oil & Gas Producers Underground
Protect'n Svc
1-800-925-0988</p> |
| <p>Connecticut
Call Before You Dig
1-800-922-4455</p> | <p>Massachusetts
Dig Safe System, Inc.
1-888-344-7233</p> | <p>Oklahoma
Call Okie
1-800-522-6543</p> |
| <p>Delaware
Miss Utility of Delmarva
1-800-282-8555</p> | <p>Michigan
Miss Dig System, Inc.
1-800-482-7171</p> | <p>Oregon
Oregon Utility Notification Center/One
Call Concepts
1-800-332-2344</p> |
| <p>Florida
Sunshine State One-Call of Florida, Inc.
1-800-432-4770</p> | <p>Minnesota
Gopher State One Call
1-800-252-1168</p> | <p>Pennsylvania
Pennsylvania One Call System, Inc.
1-800-242-1776</p> |
| <p>Georgia
Underground Protection Center, Inc.
1-800-282-7411</p> | <p>Mississippi
Mississippi One-Call System, Inc
1-800-227-6477</p> | <p>Rhode Island
Dig Safe System, Inc.
1-888-344-7233</p> |
| <p>Hawaii
Underground Service Alert North
1-800-227-2600</p> | <p>Missouri
Missouri One-Call System, Inc.
1-800-344-7483</p> | <p>South Carolina
Palmetto Utility Protection Service Inc.
1-888-721-7877</p> |
| <p>Idaho
Dig Line Inc.
1-800-342-1585
Kootenai County One-Call
1-800-428-4950
Shoshone - Benewah One-Call
1-800-398-3285</p> | <p>Montana
Utilities Underground Protection Center
1-800-424-5555
Montana One Call Center
1-800-551-8344</p> | <p>South Dakota
South Dakota One Call
1-800-781-7474</p> |
| <p>Illinois
JULIE, Inc.
1-800-892-0123
Digger (Chicago Utility Alert Network)
312-744-7000</p> | <p>Nebraska
Diggers Hotline of Nebraska
1-800-331-5666</p> | <p>Tennessee
Tennessee One-Call System, Inc.
1-800-351-1111</p> |
| <p>Indiana
Indiana Underground Plant Protection
Service
1-800-382-5544</p> | <p>Nevada
Underground Service Alert North
1-800-227-2600</p> | |
| | <p>New Hampshire
Dig Safe System, Inc.
1-888-344-7233</p> | |

Subject UTILITY LOCATING AND EXCAVATION CLEARANCE	Number HS-1.0	Page 10 of 15
	Revision 2	Effective Date 12/03

ATTACHMENT 1 (Continued)

Texas

Texas One Call System
1-800-245-4545
Texas Excavation Safety System, Inc.
1-800-344-8377
Lone Star Notification Center
1-800-669-8344

Utah

Blue Stakes of Utah
1-800-662-4111

Vermont

Dig Safe System, Inc.
1-888-344-7233

Virginia

Miss Utility of Virginia
1-800-552-7001
Miss Utility (Northern Virginia)
1-800-257-7777

Washington

Utilities Underground Location Center
1-800-424-5555
Northwest Utility Notification Center
1-800-553-4344
Inland Empire Utility Coordinating
Council
509-456-8000

West Virginia

Miss Utility of West Virginia, Inc.
1-800-245-4848

Wisconsin

Diggers Hotline, Inc.
1-800-242-8511

Wyoming

Wyoming One-Call System, Inc.
1-800-348-1030
Call Before You Dig of Wyoming
1-800-849-2476

District of Columbia

Miss Utility
1-800-257-7777

Alberta

Alberta One-Call Corporation
1-800-242-3447

British Columbia

BC One Call
1-800-474-6886

Ontario

Ontario One-Call System
1-800-400-2255

Quebec

Info-Excavation
1-800-663-9228

Subject

UTILITY LOCATING AND
EXCAVATION CLEARANCE

Number

HS-1.0

Revision

2

Page

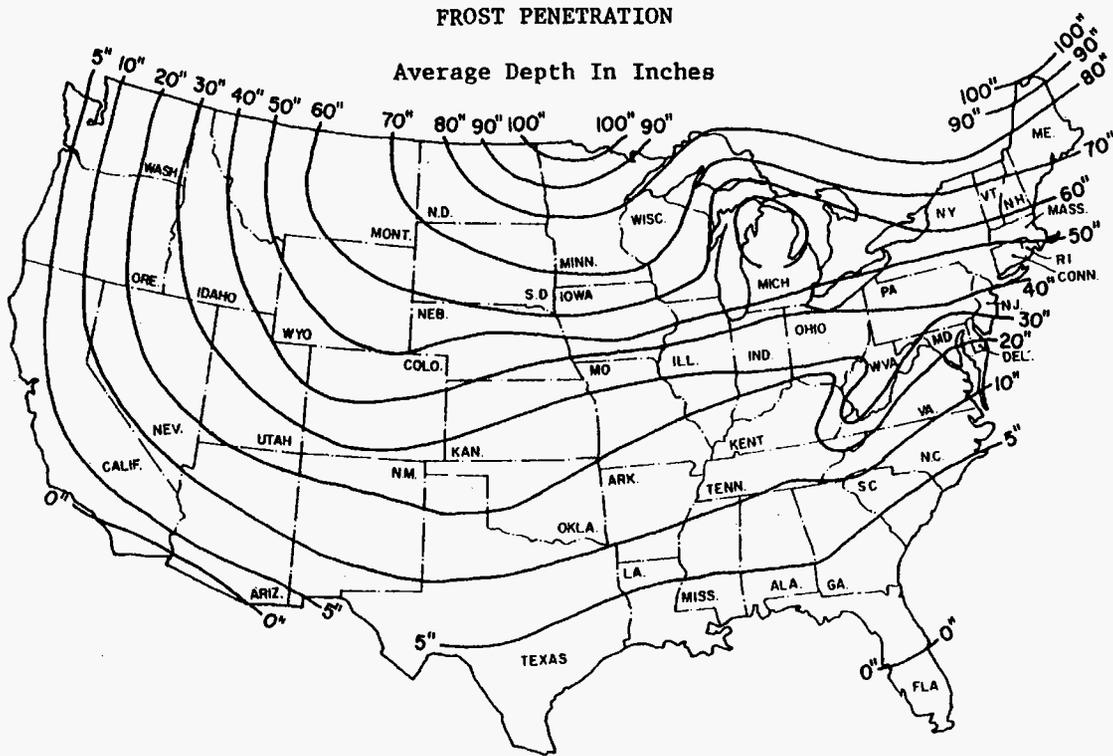
11 of 15

Effective Date

12/03

ATTACHMENT 2

FROST LINE PENETRATION DEPTHS BY GEOGRAPHIC LOCATION



Courtesy U.S. Department Of Commerce

Subject UTILITY LOCATING AND EXCAVATION CLEARANCE	Number HS-1.0	Page 12 of 15
	Revision 2	Effective Date 12/03

**ATTACHMENT 3
UTILITY CLEARANCE FORM**

Client: _____ Project Name: _____
 Project No.: _____ Completed By: _____
 Location Name: _____ Work Date: _____
 Excavation Method/Overhead Equipment: _____

1. Underground Utilities Circle One
- a) Review of existing maps? yes no N/A
 - b) Interview local personnel? yes no N/A
 - c) Site visit and inspection? yes no N/A
 - d) Excavation areas marked in the field? yes no N/A
 - e) Utilities located in the field? yes no N/A
 - f) Located utilities marked/added to site maps? yes no N/A
 - g) Client contact notified yes no N/A
 Name _____ Telephone: _____ Date: _____
 - g) State One-Call agency called? yes no N/A
 Caller: _____
 Ticket Number: _____ Date: _____
 - h) Geophysical survey performed? yes no N/A
 Survey performed by: _____
 Method: _____ Date: _____
 - i) Hand excavation performed (with concurrent use of utility
 detection device)? yes no N/A
 Completed by: _____
 Total depth: _____ feet Date: _____
 - j) Trench/excavation probed? yes no N/A
 Probing completed by: _____
 Depth/frequency: _____ Date: _____

2. Overhead Utilities Present Absent
- a) Determination of nominal voltage yes no N/A
 - b) Marked on site maps yes no N/A
 - c) Necessary to lockout/insulate/re-route yes no N/A
 - d) Document procedures used to lockout/insulate/re-route yes no N/A
 - e) Minimum acceptable clearance (SOP Section 5.2): _____

3. Notes:

Approval:

 Site Manager/Field Operations Leader Date

c: PM/Project File
 Program File

Subject UTILITY LOCATING AND EXCAVATION CLEARANCE	Number HS-1.0	Page 13 of 15
	Revision 2	Effective Date 12/03

**ATTACHMENT 4
OSHA LETTER OF INTERPRETATION**

Mr. Joseph Caldwell
Consultant
Governmental Liaison
Pipeline Safety Regulations
211 Wilson Boulevard
Suite 700
Arlington, Virginia 22201

Re: Use of hydro-vacuum or non-conductive hand tools to locate underground utilities.

Dear Mr. Caldwell:

In a letter dated July 7, 2003, we responded to your inquiry of September 18, 2002, regarding the use of hydro-vacuum equipment to locate underground utilities by excavation. After our letter to you was posted on the OSHA website, we received numerous inquiries that make it apparent that aspects of our July 7 letter are being misunderstood. In addition, a number of industry stakeholders, including the National Utility Contractors Association (NUCA), have provided new information regarding equipment that is available for this work.

To clarify these issues, we are withdrawing our July 7 letter and issuing this replacement response to your inquiry.

***Question:** Section 1926.651 contains several requirements that relate to the safety of employees engaged in excavation work. Specifically, paragraphs (b)(2) and (b)(3) relate in part to the safety of the means used to locate underground utility installations that, if damaged during an uncovering operation, could pose serious hazards to employees.*

Under these provisions, what constitutes an acceptable method of uncovering underground utility lines, and further, would the use of hydro-vacuum excavation be acceptable under the standard?

Answer

Background

Two sections of 29 CFR 1926 Subpart P (Excavations), 1926.651 (Specific excavation requirements), govern methods for uncovering underground utility installations. Specifically, paragraph (b)(2) states:

When utility companies or owners cannot respond to a request to locate underground utility installations within 24 hours * * * or cannot establish the exact location of these installations, the employer may proceed, provided the employer does so with caution, and provided detection equipment or other acceptable means to locate utility installations are used. (emphasis added).

Paragraph (b)(3) provides:

Subject UTILITY LOCATING AND EXCAVATION CLEARANCE	Number HS-1.0	Page 14 of 15
	Revision 2	Effective Date 12/03

ATTACHMENT 4 (Continued)

When excavation operations approach the estimated location of underground installations, the exact location of the installations shall be determined by safe and acceptable means. (emphasis added).

Therefore, “acceptable means” must be used where the location of the underground utilities have not been identified by the utility companies and detection equipment is not used.

Subpart P does not contain a definition of either “other acceptable means” or “safe and acceptable means.” The preambles to both the proposed rule and the final rule discussed the rationale behind the wording at issue. For example, the preamble to the proposed rule, 52 Fed. Reg. 12301 (April 15, 1987), noted that a 1972 version of this standard contained language that specified “careful probing or hand digging” as the means to uncover utilities. The preamble then noted that an amendment to the 1972 standard later deleted that language “to allow other, *equally effective means* of locating such installations.” The preamble continued that in the 1987 proposed rule, OSHA again proposed using language in section (b)(3) that would provide another example of an acceptable method of uncovering utilities that could be used where the utilities have not been marked and detection equipment is not being used – “probing with hand-held tools.” This method was rejected in the final version of 29 CFR 1926. As OSHA explained in the preamble to the final rule, 54 Fed. Reg. 45916 (October 31, 1989):

OSHA received two comments * * * and input from ACCSH [OSHA’s Advisory Committee on Construction Safety and Health] * * * on this provision. All commenters recommended dropping ‘such as probing with hand-held tools’ from the proposed provision, because this could create a hazard to employees by damaging the installation or its insulation.

In other words, the commenters objected to the use of hand tools being used unless detection equipment was used in conjunction with them. OSHA then concluded its discussion relative to this provision by agreeing with the commentators and ultimately not including any examples of “acceptable means” in the final provision.

Non-conductive hand tools are permitted

This raises the question of whether the standard permits the use of hand tools alone -- without also using detection equipment. NUCA and other industry stakeholders have recently informed us that non-conductive hand tools that are appropriate to be used to locate underground utilities are now commonly available.

Such tools, such as a “shooter” (which has a non-conductive handle and a snub nose) and non-conductive or insulated probes were not discussed in the rulemaking. Since they were not considered at that time, they were not part of the class of equipment that was thought to be unsafe for this purpose. Therefore, we conclude that the use of these types of hand tools, when used with appropriate caution, is an “acceptable means” for locating underground utilities.

Subject UTILITY LOCATING AND EXCAVATION CLEARANCE	Number HS-1.0	Page 15 of 15
	Revision 2	Effective Date 12/03

ATTACHMENT 4 (Continued)

Hydro-vacuum excavation

It is our understanding that some hydro-vacuum excavation equipment can be adjusted to use a minimum amount of water and suction pressure. When appropriately adjusted so that the equipment will not damage underground utilities (especially utilities that are particularly vulnerable to damage, such as electrical lines), use of such equipment would be considered a "acceptable means" of locating underground utilities. However, if the equipment cannot be sufficiently adjusted, then this method would not be acceptable under the standard.

Other technologies

We are not suggesting that these are the only devices that would be "acceptable means" under the standard. Industry stakeholders have informed us that there are other types of special excavation equipment designed for safely locating utilities as well.

We apologize for any confusion our July 7 letter may have caused. If you have further concerns or questions, please feel free to contact us again by fax at: U.S. Department of Labor, OSHA, Directorate of Construction, Office of Construction Standards and Compliance Assistance, fax # 202-693-1689. You can also contact us by mail at the above office, Room N3468, 200 Constitution Avenue, N.W., Washington, D.C. 20210, although there will be a delay in our receiving correspondence by mail.

Sincerely,

Russell B. Swanson, Director
Directorate of Construction

NOTE: OSHA requirements are set by statute, standards and regulations. Our interpretation letters explain these requirements and how they apply to particular circumstances, but they cannot create additional employer obligations. This letter constitutes OSHA's interpretation of the requirements discussed. Note that our enforcement guidance may be affected by changes to OSHA rules. Also, from time to time we update our guidance in response to new information. To keep apprised of such developments, you can consult OSHA's website at <http://www.osha.gov>.



TETRA TECH

STANDARD OPERATING PROCEDURES

Number	SA-1.1	Page	1 of 35
Effective Date	04/07/2008	Revision	7
Applicability	Tetra Tech NUS, Inc.		
Prepared	Earth Sciences Department		
Approved	Tom Johnston <i>T. Johnston</i>		

Subject
GROUNDWATER SAMPLE ACQUISITION AND
ONSITE WATER QUALITY TESTING

TABLE OF CONTENTS

<u>SECTION</u>	<u>PAGE</u>
1.0 PURPOSE.....	2
2.0 SCOPE	2
3.0 GLOSSARY	2
4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS	3
5.0 HEALTH AND SAFETY	4
6.0 PROCEDURES.....	5
6.1 General	5
6.2 Sampling, Monitoring, and Evacuation Equipment.....	7
6.3 Calculations of Well Volume	8
6.4 Evacuation of Static Water – Purging.....	9
6.4.1 General	9
6.4.2 Evacuation Devices	9
6.5 Onsite Water Quality Testing.....	12
6.5.1 Measurement of pH	13
6.5.2 Measurement of Specific Conductance.....	15
6.5.3 Measurement of Temperature	16
6.5.4 Measurement of Dissolved Oxygen.....	17
6.5.5 Measurement of Oxidation-Reduction Potential.....	19
6.5.6 Measurement of Salinity	20
6.5.7 Measurement of Turbidity	21
6.6 Sampling.....	22
6.6.1 Sampling Plan.....	22
6.6.2 Sampling Methods as Related to Low-Flow Sampling	23
6.7 Low-Flow Purging and Sampling.....	25
6.7.1 Scope and Application	25
6.7.2 Equipment.....	25
6.7.3 Purging and Sampling Procedure.....	26
7.0 REFERENCES.....	28
 <u>ATTACHMENTS</u>	
A PURGING EQUIPMENT SELECTION	29
B GROUNDWATER SAMPLE LOG SHEET	32
C EQUIPMENT CALIBRATION LOG.....	33
D LOW FLOW PURGE DATA SHEET	34

Subject GROUNDWATER SAMPLE ACQUISITION AND ONSITE WATER QUALITY TESTING	Number SA-1.1	Page 2 of 35
	Revision 7	Effective Date 04/07/2008

1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the process to be used for purging groundwater monitoring wells prior to sampling, for collecting groundwater samples, and for measuring groundwater quality parameters.

2.0 SCOPE

This document provides information on proper sampling equipment, onsite water quality testing, safety measures to ensure the safety of the field technician(s), and techniques for groundwater sampling. All personnel are encouraged to review the information contained herein to facilitate planning of the field sampling effort. The techniques described shall be followed whenever applicable, noting that site-specific conditions or project-specific plans may require modifications to methodology.

3.0 GLOSSARY

Conductivity – Conductivity is a numerical expression of the ability of an aqueous solution to carry an electric current. This ability depends on the presence of ions and their total concentration, mobility, valence, and relative concentrations and on temperature. Conductivity is highly dependent on temperature and should be reported at a particular temperature, i.e., 20.2 microSiemens per centimeter (mS/cm) at 14°C.

Dissolved Oxygen (DO) – DO levels in natural and wastewater depend on the physical, chemical, and biochemical activities in the water sample.

Groundwater Sample – A quantity of water removed from the ground, usually via a monitoring well that may or may not be lined with a well casing.

Oxidation-Reduction Potential (ORP) - A measure of the activity ratio of oxidizing and reducing species as determined by the electromotive force developed by a noble metal electrode immersed in water, as referenced against a reference electrode. A reference electrode commonly used in the field is the silver/silver chloride electrode, which has a voltage offset of about 210 mV from the standard hydrogen electrode (SHE). To convert field ORP measurements to equivalent SHE values, approximately 210 mV must be added to the ORP values obtained using the silver/silver chloride electrode. The actual offset depends on the concentration of the potassium chloride (KCl) in the field reference electrode and the temperature. Offsets typically range from 199 (saturated KCl) to 205 (3.5 Molar KCl) to 222 mV (1 Molar KCl) at 25°C and are greater at lower temperatures.

pH - The negative logarithm (base 10) of the hydrogen ion activity. The hydrogen ion activity is related to the hydrogen ion concentration, and, in a relatively weak solution, the two are nearly equal. Thus, for all practical purposes, pH is a measure of the hydrogen ion concentration.

pH Paper - Indicator paper that turns different colors depending on the pH of the solution to which it is exposed. Comparison with color standards supplied by the manufacturer will then give an indication of the solution's pH.

Representativeness – A qualitative description of the degree to which an individual sample accurately reflects population characteristics or parameter variations at a sampling point. It is therefore an important characteristic not only of assessment and quantification of environmental threats posed by the site, but also for providing information for engineering design and construction. Proper sample location selection and proper sample collection methods are important to ensure that a truly representative sample has been collected.

Subject GROUNDWATER SAMPLE ACQUISITION AND ONSITE WATER QUALITY TESTING	Number SA-1.1	Page 3 of 35
	Revision 7	Effective Date 04/07/2008

Salinity – The measurement of dissolved salts in a given mass of solution. Note: most field meters determined salinity automatically from conductivity and temperature. The value will be displayed in either parts per thousand (ppt) or percent (e.g., 35 ppt equals 3.5 percent). The parts per thousand symbol ($^{\circ}/_{00}$) is not the same as the percent symbol (%).

Turbidity – Turbidity in water is caused by suspended matter such as clay, silt, and fine organic and inorganic matter. Turbidity is an expression of the optical property that causes light to be scattered and absorbed rather than transmitted in a straight line through the sample.

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager - The Project Manager is responsible for determining the sampling objectives, initial sampling locations, and field procedures used in the collection of groundwater samples. Additionally, in consultation with other project personnel (geologist, hydrogeologist, etc.), the Project Manager identifies sampling locations.

Site Safety Officer (SSO) - The SSO (or a qualified designee) is responsible for providing the technical support necessary to implement the project Health and Safety Plan (HASP). This includes but is not limited to performing air quality monitoring during sampling, boring and excavation activities, and ensuring that workers and offsite (downwind) individuals are not exposed to hazardous levels of airborne contaminants. The SSO or SSO designee may also be required to advise the FOL on other safety-related matters regarding sampling, such as mitigative measures to address potential hazards from hazardous objects or conditions.

Project Geologist/Sampler - The project geologist/sampler is responsible for the proper acquisition of samples in accordance with this SOP or other project-specific documents. In addition, this individual is responsible for the completion of all required paperwork (e.g., sample log sheets, field notebook, boring logs, container labels, custody seals, and chain-of-custody forms) associated with the collection of those samples.

Project Hydrogeologist – This individual is responsible for selecting and detailing the specific groundwater sampling techniques, onsite water quality testing (type, frequency, and location), equipment to be used, and providing detailed input in this regard to the project planning documents. The project hydrogeologist is also responsible for properly briefing and overseeing the performance of site sampling personnel.

Field Operations Leader (FOL) – This individual is primarily responsible for the execution of the planning document containing the Sampling and Analysis Plan (SAP). This is accomplished through management of a field sampling team for the proper acquisition of samples. He or she is responsible for the supervision of onsite analyses; ensuring proper instrument calibration, care, and maintenance; sample collection and handling; the completion and accuracy of all field documentation; and making sure that custody of all samples obtained is maintained according to proper procedures. When appropriate and as directed by the FOL, such responsibilities may be performed by other qualified personnel (e.g., field technicians) where credentials and time permit. The FOL is ultimately responsible for adherence to Occupational Safety and Health Administration (OSHA) regulations during these operations through self acquisition or through the management of a field team of samplers.

General personnel qualifications for groundwater sample collection and onsite water quality testing include the following:

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.
- Capability of performing field work under the expected physical and environmental (i.e., weather) conditions.

Subject GROUNDWATER SAMPLE ACQUISITION AND ONSITE WATER QUALITY TESTING	Number SA-1.1	Page 4 of 35
	Revision 7	Effective Date 04/07/2008

- Familiarity with appropriate procedures for sample documentation, handling, packaging, and shipping.

5.0 HEALTH AND SAFETY

Specific safety and health precautions are identified throughout this SOP. In addition to those precautions, the following general hazards may be incurred during sampling activities:

- Knee injuries from kneeling on hard surfaces
- Slips, trips, and falls
- Cuts and lacerations
- Traffic hazards associated with sampling in parking areas and roadways and along highways.

Methods of avoiding these hazards are provided below.

Knee injuries – Many monitoring wells are installed as flush mounts. Personnel are required to kneel to open these wells and to take groundwater level measurements, etc. This could result in knee injuries from kneeling on stones/foreign objects and general damage due to stress on the joints. To combat this hazard:

- Clear any foreign objects from the work area.
- Wear hard-sided knee pads.

Slips, Trips, and Falls – These hazards exist while traversing varying terrains carrying equipment to sample wells. To minimize these hazards:

- Pre-survey well locations. Eliminate, barricade, or otherwise mark physical hazards leading to the locations.
- Carry small loads that do not restrict the field of vision.
- Travel the safest and clearest route (not necessarily the shortest).

Cuts and Lacerations – To prevent cuts and lacerations associated with groundwater sampling, the following provisions are required:

- Always cut away from yourself and others when cutting tubing or rope. This will prevent injury to yourself and others if the knife slips.
- Do not place items to be cut in your hand or on your knee.
- Change blades as necessary to maintain a sharp cutting edge. Many accidents result from struggling with dull cutting attachments.
- Whenever practical, wear cut-resistant gloves (e.g., leather or heavy cotton work gloves) at least on the hand not using the knife.

Subject GROUNDWATER SAMPLE ACQUISITION AND ONSITE WATER QUALITY TESTING	Number SA-1.1	Page 5 of 35
	Revision 7	Effective Date 04/07/2008

- Keep cutting surfaces clean and smooth.
- Secure items to be cut -- do not hold them against the opposing hand, a leg, or other body part.
- When transporting glassware, keep it in a hard-sided container such as a cooler so that if there is a fall, you will be less likely to get cut by broken glass.
- DO NOT throw broken glass or glass ampoules into garbage bags. Place broken glass and glass ampoules in hard-sided containers such as a cardboard box or directly into a dumpster. DO NOT reach into garbage bags to retrieve any item accidentally thrown away. Empty the contents onto a flat surface to avoid punctures and lacerations from reaching where you cannot see.

Vehicular and Foot Traffic Hazards – When sampling along the roadway or near traffic patterns, follow the following precautions:

- Motorists may be distracted by onsite activities – ASSUME THEY DO NOT SEE YOU OR MEMBERS OF YOUR FIELD CREW.
- DO NOT place obstructions (such as vehicles) along the sides of the road that may cause site personnel to move into the flow of traffic to avoid your activities or equipment or that will create a blind spot.
- **Provide a required free space of travel.** Maintain at least 6 feet of space between you and moving traffic. Where this is not possible, use flaggers and/or signs to warn oncoming traffic of activities near or within the travel lanes.
- **Face Traffic.** Whenever feasible, if you must move within the 6 feet of the required free space or into traffic, attempt to face moving traffic at all times. Always leave yourself an escape route.
- Wear high-visibility vests to increase visual recognition by motorists.
- Do not rely on the vehicle operator's visibility, judgment, or ability. Make eye contact with the driver. Carefully and deliberately use hand signals so they will not startle or confuse motorists or be mistaken for a flagger's direction before moving into traffic.
- Your movements may startle a motorist and cause an accident, so move deliberately. Do not make sudden movements that might confuse a motorist.

6.0 PROCEDURES

6.1 General

For information derived from a groundwater sample to be useful and accurate, the sample must be representative of the particular zone being sampled. The physical, chemical, and bacteriological integrity of the sample must be maintained from the time of sampling to the time of analysis to keep any changes in water quality parameters to a minimum.

CAUTION

A closed well may generate and accumulate gases due to biological degradation, evolution of volatile chemicals from groundwater into the air, or other chemical actions. These gases may also be artificially generated, such as in the case of air sparging or

Subject GROUNDWATER SAMPLE ACQUISITION AND ONSITE WATER QUALITY TESTING	Number SA-1.1	Page 6 of 35
	Revision 7	Effective Date 04/07/2008

extraction wells, which may take several days to depressurize. See Section 6.6.2 for safety measures to be employed to protect sampling personnel.

Methods for withdrawing samples from completed wells include the use of pumps, compressed air or nitrogen, bailers, and various types of samplers. The primary considerations in obtaining a representative sample of groundwater are to avoid collection of stagnant (standing) water in the well and to avoid physical or chemical alteration of the water sample due to external influences of the sampling technique(s). In a non-pumping well, there will be little or no vertical mixing of water in the well pipe or casing, and stratification will occur. The well water in the screened section will mix with groundwater due to normal flow patterns, but the well water above the screened section will remain isolated and become stagnant. Concentration gradients resulting from mixing and dispersion processes, layers of variable geologic permeability, and the presence of separate-phase product (e.g., floating hydrocarbons) may cause stratification. Excessive pumping or improper sampling methods can dilute or increase contaminant concentrations in the collected sample compared to what is representative of the integrated water column as it naturally occurs at that point, resulting in the collection of a non-representative sample. To safeguard against collecting non-representative samples, the following approach shall be followed prior to sample acquisition:

CAUTION

Mechanical agitation of well water may cause off-gas generation of volatile contaminants, creating an inhalation exposure to the sampler(s). Where avoiding an inhalation exposure is not possible and mechanical agitation is possible, pump into closed-top containers to control potential air emissions.

1. If possible, position yourself (and the sampling equipment) upwind of the well head.
2. Purge the monitoring well to be sampled prior to obtaining any samples from it. Evacuation of three to five well volumes is recommended prior to sampling, unless low-flow purging and sampling methods are utilized as described in Section 6.7 (Consult the site-specific SAP for exact purging parameters). In a high-yielding groundwater formation and where there is no stagnant water in the well above the screened section, extensive evacuation prior to sample withdrawal is not as critical as it is in a low-yielding well or in wells containing stagnant water.
3. For wells with low yields that are purged dry during sampling, evacuate the well and allow it to recover to 75 percent of full capacity prior to sample acquisition. If the recovery rate is fairly rapid (generally 300 mL per minute or greater), attempt to continue evacuation until the number of well volumes specified in the SAP is achieved. If this cannot be accomplished, allow recovery to 75 percent of capacity and begin sampling.

CAUTION

For moderate to high-yielding monitoring wells, an evacuation rate that does not cause excessive turbulence in the well should be selected. There is no absolute safeguard against contaminating the sample with stagnant water; hence, special techniques are required for purging to minimize the potential for sample contamination (see below).

4. For moderate to high-yielding monitoring wells, use one of the following purge techniques:
 - Place a submersible pump or the intake line of a surface pump or bailer just below the water surface when removing the stagnant water.

Subject GROUNDWATER SAMPLE ACQUISITION AND ONSITE WATER QUALITY TESTING	Number SA-1.1	Page 7 of 35
	Revision 7	Effective Date 04/07/2008

- While purging and as the water level decreases, lower the pump or intake line as the water level drops in the well. Three to five volumes of water shall be removed to provide reasonable assurance that all stagnant water has been evacuated. After this is accomplished, a bailer or other approved device may be used to collect the sample for analysis.
- Unless otherwise directed, place the intake line of the sampling pump (or the submersible pump itself) near the center of the screened section, and pump approximately one casing volume of water from the well at a low purge rate equal to the well's recovery rate (low-flow sampling).

6.2 Sampling, Monitoring, and Evacuation Equipment

Sample containers shall conform to the guidelines in SOP SA-6.1.

The following equipment shall be on hand when sampling groundwater wells (reference SOPs SA-6.1 and SA-7.1):

- Sample packaging and shipping equipment – Coolers for sample shipping and cooling, chemical preservatives, appropriate sampling containers and filler materials, ice, labels, and chain-of-custody documents.
- Field tools and instrumentation
 - Multi-parameter water quality meter with an in-line sample chamber capable of measuring ORP, pH, temperature, DO, specific conductance, turbidity, and salinity, or individual meters (as applicable)
 - pH Paper
 - Camera and film (if appropriate)
 - Appropriate keys (for locked wells)
 - Water level indicator and/or oil-water interface probe if separate-phase product is expected
- Pumps
 - Shallow-well pumps: Centrifugal, bladder, suction, or peristaltic pumps with drop lines and air-lift apparatus (compressor and tubing) where applicable.
 - Deep-well pumps: Submersible pump and electrical power-generating unit, or bladder pumps where applicable.
- Other sampling equipment – Bailers, graduated cylinder, stopwatch, and inert line with tripod-pulley assembly (if necessary).
- Pails – Plastic, graduated.
- Clean paper or cotton towels for cleaning equipment.
- Buckets with lids for collecting purge water.

Subject GROUNDWATER SAMPLE ACQUISITION AND ONSITE WATER QUALITY TESTING	Number SA-1.1	Page 8 of 35
	Revision 7	Effective Date 04/07/2008

- Decontamination solutions – Deionized water, potable water, phosphate-free laboratory-grade detergent, and analytical-grade solvent (e.g., pesticide-grade isopropanol), as required.

Ideally, sample withdrawal equipment shall be completely inert, economical, easily cleaned, cleaned prior to use, reusable, able to operate at remote sites in the absence of power sources, and capable of delivering variable rates for well purging and sample collection.

6.3 Calculations of Well Volume

To ensure that the proper volume of water has been removed from the well prior to sampling, it is first necessary to know the volume of standing water in the well pipe (including well screen where applicable). This volume can be easily calculated by the following method. Calculations shall be entered in the site logbook or field notebook or on a sample log sheet form or equivalent electronic form(s) (see SOP SA-6.3):

1. Obtain all available information on well construction (location, casing, screen, etc.).
2. Determine well or inner casing diameter.
3. Measure and record static water level (depth below ground level or top of casing reference point).
4. Determine depth of well by sounding using a clean, decontaminated, weighted tape measure or water level indicator.
5. Calculate number of linear feet of static water (total depth or length of well pipe minus the depth to static water level).
6. Calculate one static well volume in gallons $V = (0.163)(T)(r^2)$

where: V = Static volume of well in gallons.
T = Linear feet of water in the well.
r = Inside radius of well casing in inches.
0.163 = Conversion factor (compensates for conversion of casing radius from inches to feet and cubic feet to gallons and pi.

7. Per evacuation volumes discussed above, determine the minimum amount to be evacuated before sampling.

Measuring devices may become contaminated when gathering the above information if they are submerged in contaminated water. Decontamination of the tape or water level indicator must be conducted between measurements in different wells as follows:

1. Saturate a paper towel or clean cotton towel with deionized water.
2. As the measuring device is extracted, wipe the tape, changing the cleaning surface frequently.
3. After it is extracted, rinse the probe or tape using a spray bottle of deionized water over a bucket or similar collection container.

Based on the contaminant (oily, etc), it may be necessary to use a soap and water wash and rinse to remove contaminants. Isopropanol can be used on the probe/tape. However, it is recommended that the use of solvents on the tape be minimized because they could degrade the protective covering or possibly

Subject GROUNDWATER SAMPLE ACQUISITION AND ONSITE WATER QUALITY TESTING	Number SA-1.1	Page 9 of 35
	Revision 7	Effective Date 04/07/2008

remove the scale designations. If isopropanol (or some other solvent) is used, assure that the manufacturer/supplier Material Safety Data Sheet (MSDS) is obtained, kept on site at a readily available location with other MSDSs, and reviewed by personnel prior to the first usage of the solvent. Also, add the substance to the site-specific Hazardous Chemical Inventory list (see Section 5 of the TtNUS Health and Safety Guidance Manual [HSGM], Hazard Communication Program and OSHA Standard 29 CFR 1910.1200).

6.4 Evacuation of Static Water – Purging

6.4.1 General

The amount to be purged from each well will be determined prior to sample collection. This amount will depend on the intent of the monitoring program and the hydrogeologic conditions. Programs to determine overall quality of water resources may require long pumping periods to obtain a sample that is representative of a large volume of the aquifer. The pumped volume may be specified prior to sampling so that the sample can be a composite of a known volume of the aquifer. Alternately, the well can be pumped until parameters such as temperature, specific conductance, pH, and turbidity (as applicable) have stabilized. Onsite measurements of these parameters shall be recorded in the site logbook or field notebook or on standardized data sheets or an equivalent electronic form(s).

6.4.2 Evacuation Devices

The following discussion is limited to those devices commonly used at hazardous waste sites. Attachment A provides guidance on the proper evacuation device to use for given sampling situations. All of these techniques involve equipment that is portable and readily available.

Bailers

Bailers are the simplest evacuation devices used and have many advantages. They generally consist of a length of tubing equipped with a base plate and ball check-valve at the bottom. Bailers are comprised of stainless steel and plastic. They come in a variety of sizes, but the two most often used are 2 inches and 4 inches in diameter. An inert non-absorbent line such as polyethylene rope is used to lower and then raise the bailer to retrieve the sample. As the bailer is lowered into the water column, the ball is pushed up allowing the tube to be filled. When the bailer is pulled upward, the ball seats in the base plate preventing water from escaping.

Advantages of bailers include the following:

- There are few limitations on size and materials used.
- No external power source is needed.
- Bailers are inexpensive and can be dedicated and hung in a well to reduce the chances of cross-contamination.
- Bailers are relatively easy to decontaminate.

Limitations on the use of bailers include the following:

- It is time consuming to remove stagnant water using a bailer.

Subject GROUNDWATER SAMPLE ACQUISITION AND ONSITE WATER QUALITY TESTING	Number SA-1.1	Page 10 of 35
	Revision 7	Effective Date 04/07/2008

- Splashing the bailer into the water or transfer of sample may cause aeration.
- The use of a bailer does not permit constant in-line monitoring of groundwater parameters.
- Use of bailers is physically demanding, especially in warm temperatures at personal protection equipment (PPE) levels above Level D.

Safety concerns using a bailer include the following:

- Muscle stress and strain, especially when using 4-inch bailers and when pulling from excessively deep wells.
- Entanglement, possible hand/finger injuries, and rope burns during a sudden release of the bailer back down the well.
- Direct contact with contaminants of concern and sample preservatives when discharging the bailer contents because there is not a high level of control during a direct pour, and splashing and indirect contact with contaminants/preservatives could occur.

Control measures for these hazards are provided in Section 6.6.2.

Suction Pumps

There are many different types of inexpensive suction pumps including centrifugal, diaphragm, and peristaltic pumps. Centrifugal and diaphragm pumps can be used for well evacuation at a fast pumping rate and for sampling at a low pumping rate. The peristaltic pump is a low-volume pump that uses rollers to squeeze flexible tubing to create suction. This tubing can be dedicated to a well to prevent cross-contamination from well to well. Suction pumps are all portable, inexpensive, and readily available. However, because they are based on suction, their use is restricted to areas with water levels within 20 to 25 feet of the ground surface. A significant limitation is that the vacuum created by these pumps can cause loss of dissolved gases and volatile organics. Another limitation of these pumps is that they require a secondary energy source to drive them. Electrically driven pumps may require portable generators as energy sources. Air diaphragm pumps require air compressors and/or compressed gas cylinders to drive them. The advantage of the peristaltic pump is that it will operate from a portable battery source. Safety measures associated with these pumps are provided below.

Air-Lift and Gas-Lift Samplers

This group of pump samplers uses gas pressure either in the annulus of the well or in a venturi to force groundwater up a sampling tube. These pumps are also relatively inexpensive. Air- or gas-lift samplers are more suitable for well development than for sampling because the samples may be aerated as a result of pump action. Aeration can cause pH changes and subsequent trace metal precipitation or loss of volatile organics.

Submersible Pumps

Submersible pumps take in water and push the sample up a sample tube to the surface. The power sources for these samplers may be compressed gas or electricity. Operation principles vary, and displacement of the sample can be by an inflatable bladder, sliding piston, gas bubble, or impeller. Pumps are available for 2-inch-diameter wells and larger. These pumps can lift water from considerable depths (several hundred feet).

Subject GROUNDWATER SAMPLE ACQUISITION AND ONSITE WATER QUALITY TESTING	Number SA-1.1	Page 11 of 35
	Revision 7	Effective Date 04/07/2008

Limitations of this class of pumps include the following:

- They may have low delivery rates.
- Many models are expensive.
- Compressed gas or electric power is needed.
- Sediment in water may cause clogging of the valves or eroding of the impellers with some of these pumps.
- Decontamination of internal components can be difficult and time consuming.

Compressed Gases

Safety concerns using compressed gases as an energy source in these pumps are numerous. The nitrogen gas or compressed air is provided in a compressed gas cylinder at a pressure of approximately 2,000 psi. If damaged, these cylinders can become dangerous projectiles. Additionally, a sudden release of a cylinder's contents can involve considerable force that could cause significant damage to the eyes and/or skin. Protective measures include the following:

- Always wear safety impact glasses when handling compressed gases.
- Always administer compressed gases through an appropriate pressure-reducing regulator.
- When clearing the cylinder connection port, open the cylinder valve only enough to clear foreign debris. During this process, always position the cylinder valve so that it faces away from you and others.
- If the cylinder is designed to accept a valve protection cap, always keep that protection cap in place, except the cylinder is connected for use.
- When using the cylinder, lay the cylinder on its side to avoid the potential of it falling and knocking the valve off (and becoming a missile).
- DO NOT use the compressed nitrogen or air to clean clothing or to spray off the skin. Small cuts in the protective layer of the skin may permit the gas to enter into the bloodstream, presenting the potential danger of an embolism.

See the project-specific HASP for additional direction concerning cylinder safe handling procedures pertaining to the safe handling, transportation, and storage of compressed gas cylinders.

Electrical Shock

Even in situations where portable batteries are used, the potential for electrical shock exists. This potential risk is increased in groundwater sampling activities because of the presence of groundwater near the batteries. This potential is also increased in (prohibited) situations where jury-rigging of electrical connections is performed. Other potential hazards occur when field samplers open the hood of a running car to access the battery as a power source. To control these hazards:

Subject GROUNDWATER SAMPLE ACQUISITION AND ONSITE WATER QUALITY TESTING	Number SA-1.1	Page 12 of 35
	Revision 7	Effective Date 04/07/2008

- If you are unfamiliar with electrical devices, do not experiment, get help, and get the proper equipment necessary to power your device.
- Use the proper portable power inverters for cigarette lighter connections to minimize the need to access the battery under the hood of your vehicle.
- Use of electrical generators may pose a number of hazards including noise, those associated with fueling, and indirect sample influence.

To minimize or eliminate electrical generator hazards:

- Inspect the generator before use. Ensure that the generator and any extension cords are rated for the intended operation and have a Ground Fault Circuit Interrupter (GFCI) in line to control potential electrical shock.
- Fuel the generator before purging and sampling to avoid loss of power during sampling.
- Fuel engines only when they are turned OFF and have cooled sufficiently to prevent a fire hazard.
- Place the generator and any fuel source at least 50 feet from the well to be sampled to avoid indirect influence to the sample from fuel vapors or emission gases.

Lifting Hazards

This hazard may be experienced when moving containers of purge water, equipment, cylinders, etc. To control these potential hazards:

- Do not fill purge buckets to more than 80 percent of their capacity.
- Obtain a gas cylinder of sufficient size to complete the designated task but not too large to handle. K-size cylinders weigh approximately 135 pounds and are difficult to handle. M-size cylinders weigh approximately 50 pounds and are easier to handle and move.
- When necessary, get help lifting and moving gas cylinders and other heavy objects. Minimize twisting and turning while lifting. If it is necessary to move these cylinders or generators over significant distance, use mechanical means (carts, etc.).
- Use proper lifting techniques as described in Section 4.4 of the HSGM.

6.5 Onsite Water Quality Testing

This section describes the procedures and equipment required to measure the following parameters of an aqueous sample in the field:

- pH
- Specific conductance
- Temperature
- DO
- ORP
- Turbidity
- Salinity

Subject GROUNDWATER SAMPLE ACQUISITION AND ONSITE WATER QUALITY TESTING	Number SA-1.1	Page 13 of 35
	Revision 7	Effective Date 04/07/2008

This section is applicable for use in an onsite groundwater quality monitoring program to be conducted at a hazardous or nonhazardous waste site. The procedures and equipment described are applicable to groundwater samples and are not, in general, subject to solution interferences from color, turbidity, or colloidal material or other suspended matter.

This section provides general information for measuring the parameters listed above with instruments and techniques in common use. Because instruments from different manufacturers may vary, review of the manufacturer's literature pertaining to the use of a specific instrument is required before use. Most meters used to measure field parameters require calibration on a daily basis. Refer to SOP SA-6.3 for an example equipment calibration log.

6.5.1 Measurement of pH

6.5.1.1 General

Measurement of pH is one of the most important and frequently used tests in water chemistry. Practically every phase of water supply and wastewater treatment such as acid-base neutralization, water softening, and corrosion control is pH dependent. Likewise, the pH of leachate can be correlated with other chemical analyses to determine the probable source of contamination. It is therefore important that reasonably accurate pH measurements be taken and recorded on the groundwater sample log sheet (Attachment B) or equivalent electronic form.

Two methods are given for pH measurement: the pH meter and pH indicator paper. Indicator paper is used when only an approximation of the pH is required or when pH meter readings need to be verified, and the pH meter is used when a more accurate measurement is needed. The response of a pH meter can be affected by high levels of colloidal or suspended solids, but the effect is generally of little significance. Consequently, specific methods to overcome this interference are not described. The response of pH paper is unaffected by solution interferences from color, turbidity, or colloidal or suspended materials unless extremely high levels capable of coating or masking the paper are encountered. In such cases, use of a pH meter is recommended.

6.5.1.2 Principles of Equipment Operation

Use of pH papers for pH measurement relies on a chemical reaction caused by the acidity or alkalinity of the solution created by the addition of the water sample reacting with the indicator compound on the paper. Various types of pH papers are available, including litmus (for general acidity or alkalinity determination) and specific, or narrower range, pH range paper.

Use of a pH meter relies on the same principle as other ion-specific electrodes. Measurement relies on establishment of a potential difference across a glass or other type of membrane in response to (in this instance, hydrogen) ion activity (which is usually similar to concentration) across that membrane. The membrane is conductive to ionic species and, in combination with a standard or reference electrode, a potential difference proportional to the ion concentration is generated and measured.

6.5.1.3 Equipment

The following equipment is to be used for obtaining pH measurements:

- A stand-alone portable pH meter or combination meter equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).

Subject GROUNDWATER SAMPLE ACQUISITION AND ONSITE WATER QUALITY TESTING	Number SA-1.1	Page 14 of 35
	Revision 7	Effective Date 04/07/2008

- Combination electrode with polymer body to fit the above meter. Alternately, a pH electrode and a reference electrode can be used if the pH meter is equipped with suitable electrode inputs.
- Buffer solutions, as specified by the manufacturer. If the buffer solutions are considered hazardous per 29 Code of Federal Regulations (CFR) 1910.1200 (Hazard Communication) or the volumes used are greater than consumer commodity levels, the SSO shall obtain MSDSs from the manufacturer for the specific buffer solutions (see Section 4 of the HSGM regarding the Hazard Communication Program)
- pH indicator paper to cover the pH range 2 through 12.
- Manufacturer's operation manual. All personnel must be familiar with the equipment operation to ensure that the integrity of samples is preserved and that the equipment is operated safely.

6.5.1.4 Measurement Techniques for Field Determination of pH

pH Meter

The following procedure shall be used for measuring pH with a pH meter (meter standardization is according to manufacturer's instructions):

1. Inspect the instrument and batteries prior to initiation of the field effort.
2. Check the integrity of the buffer solutions used for field calibration. Buffer solutions need to be changed often as a result of degradation upon exposure to the atmosphere.
3. If applicable, make sure all electrolyte solutions within the electrode(s) are at their proper levels and that no air bubbles are present within the electrode(s).
4. Calibrate the meter and electrode(s) on a daily use basis (or as recommended by manufacturer) following manufacturer's instructions. Record calibration data on a water quality meter calibration log sheet (Attachment C) or equivalent electronic form.
5. Immerse the electrode(s) in the sample. Stabilization may take several seconds to minutes. If the pH continues to drift, the sample temperature may not be stable, a physical reaction (e.g., degassing) may be taking place in the sample, or the meter or electrode may be malfunctioning. The failure of the measurements to stabilize must be clearly noted in the logbook or equivalent electronic form.
6. Read and record the pH of the sample. pH shall be recorded to the nearest 0.01 pH standard unit. Also record the sample temperature (unless otherwise specified in the SAP, record temperatures to the nearest whole degree Fahrenheit or 0.5 degree Celsius).
7. Rinse the electrode(s) with deionized water.
8. Store the electrode(s) in an accordance with manufacturer's instructions when not in use.

Any visual observation of conditions that may interfere with pH measurement, such as oily materials or turbidity, shall be noted and avoided as much as possible.

Subject GROUNDWATER SAMPLE ACQUISITION AND ONSITE WATER QUALITY TESTING	Number SA-1.1	Page 15 of 35
	Revision 7	Effective Date 04/07/2008

pH Paper

Use of pH paper is very simple and requires no sample preparation, standardization, etc. pH paper is available in several ranges, including wide-range (indicating approximately pH 1 to 12), mid-range (approximately pH 0 to 6, 6 to 9, 8 to 14) and narrow-range (many available, with ranges as narrow as 1.5 pH units). The appropriate range of pH paper shall be selected. If the pH is unknown the investigation shall start with wide-range paper and proceed with successively narrower range paper until the sample pH is determined. To measure the pH with pH paper:

1. Collect a small portion of sample into a clean container.
2. Dip the pH paper into this small portion of sample.
3. Compare the color of the paper to the color chart that is provided with the pH paper and read the corresponding pH from the chart.
4. Record the pH value from the chart on the sampling log sheet.
5. Discard the used pH paper as trash.
6. Discard the small volume of sample that was used for the pH measurement with the other investigative derived waste.

6.5.2 Measurement of Specific Conductance

6.5.2.1 General

Conductance provides a measure of dissolved ionic species in water and can be used to identify the direction and extent of migration of contaminants in groundwater or surface water. It can also be used as a measure of subsurface biodegradation or to indicate alternate sources of groundwater contamination.

Conductivity is a numerical expression of the ability of a water sample to carry an electric current. This value depends on the total concentration of ionized substances dissolved in the water and the temperature at which the measurement is made. The mobility of each of the various dissolved ions, their valences, and their actual and relative concentrations affect conductivity.

It is important to obtain a specific conductance measurement soon after taking a sample because temperature changes, precipitation reactions, and absorption of carbon dioxide from the air all affect specific conductance. Most conductivity meters in use today display specific conductance in units of mS/cm, which is the conductivity normalized to a temperature of 25°C. These are the required units to be recorded on the groundwater sample log field form or equivalent electronic form.

6.5.2.2 Principles of Equipment Operation

An aqueous system containing ions will conduct an electric current. In a direct-current field, the positive ions migrate toward the negative electrode, and the negatively charged ions migrate toward the positive electrode. Most inorganic acids, bases, and salts such as hydrochloric acid, sodium carbonate, and sodium chloride, respectively, are relatively good conductors. Conversely, organic compounds such as sucrose or benzene, which do not dissociate in aqueous solution, conduct a current very poorly if at all.

Subject GROUNDWATER SAMPLE ACQUISITION AND ONSITE WATER QUALITY TESTING	Number SA-1.1	Page 16 of 35
	Revision 7	Effective Date 04/07/2008

A conductance cell and a Wheatstone Bridge (for the measurement of potential difference) may be used for measurement of electrical resistance. The ratio of current applied to voltage across the cell may also be used as a measure of conductance. The core element of the apparatus is the conductivity cell containing the solution of interest. Depending on the ionic strength of the aqueous solution to be tested, a potential difference is developed across the cell, which can be converted directly or indirectly (depending on instrument type) to a measurement of specific conductance.

6.5.2.3 Equipment

The following equipment is needed for taking specific conductance measurements:

- Stand-alone portable conductivity meter or combination meter equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).
- Calibration solution, as specified by the manufacturer.
- Manufacturer's operation manual.

A variety of conductivity meters are available that may also be used to monitor salinity and temperature. Probe types and cable lengths vary, so equipment must be obtained to meet the specific requirements of the sampling program.

6.5.2.4 Measurement Techniques for Specific Conductance

The steps involved in taking specific conductance measurements are as follows (calibration shall be conducted according to manufacturer's instructions):

1. Check batteries and calibrate instrument before going into the field.
2. Calibrate on a daily use basis (or as recommended by manufacturer), according to the manufacturer's instructions and record all pertinent information on a water quality meter calibration log sheet or equivalent electronic form. Potassium chloride solutions with a specific conductance closest to the values expected in the field shall be used for calibration.
3. Rinse the cell with one or more portions of the sample to be tested or with deionized water and shake excess water from the cell.
4. Immerse the electrode in the sample and measure the conductivity.
5. Read and record the results in a field logbook or on a sample log sheet or equivalent electronic form.
6. Rinse the electrode with deionized water.

If the specific conductance measurements become erratic, recalibrate the instrument and see the manufacturer's instructions for troubleshooting assistance.

6.5.3 Measurement of Temperature

6.5.3.1 General

In combination with other parameters, temperature can be a useful indicator of the likelihood of biological action in a water sample. It can also be used to trace the flow direction of contaminated groundwater.

Subject GROUNDWATER SAMPLE ACQUISITION AND ONSITE WATER QUALITY TESTING	Number SA-1.1	Page 17 of 35
	Revision 7	Effective Date 04/07/2008

Temperature measurements shall be taken in situ, or as quickly as possible in the field because collected water samples may rapidly equilibrate with the temperature of their surroundings.

6.5.3.2 Equipment

Temperature measurements may be taken with alcohol-toluene, mercury-filled, dial-type thermometers or combination meters equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22). In addition, various meters such as specific conductance or DO meters that have temperature measurement capabilities may also be used. Using such instrumentation along with suitable probes and cables, in-situ measurements of temperature at great depths can be performed.

6.5.3.3 Measurement Techniques for Water Temperature

If a thermometer is used to determine the temperature for a water sample, use the following procedure:

1. Immerse the thermometer in the sample until temperature equilibrium is obtained (1 to 3 minutes). To avoid the possibility of cross-contamination, the thermometer shall not be inserted into samples that will undergo subsequent chemical analysis.
2. Record values in a field logbook or on a sample log sheet or equivalent electronic form.

If a temperature meter or probe is used:

1. Calibrate the instrument according to manufacturer's recommendations prior to use.
2. Immerse the meter/probe in the sample until temperature equilibrium is obtained (1 to 3 minutes). To avoid the possibility of cross-contamination, the meter/probe shall not be inserted into samples that will undergo subsequent chemical analysis.
3. Record values in a field logbook or on a sample log sheet or equivalent electronic form.

6.5.4 Measurement of Dissolved Oxygen

6.5.4.1 General

DO levels in natural water and wastewater depend on the physical, chemical and biochemical activities in the water body. In addition, the growth of many aquatic organisms and the rate of corrosivity are dependent on DO concentrations. Thus, analysis for DO is a key test in water pollution and waste treatment process control. If at all possible, DO measurements shall be taken in situ because concentrations may show a large change in a short time if the sample is not adequately preserved.

The monitoring method discussed herein is limited to the use of DO meters. Chemical methods of analysis (i.e., Winkler methods) are available but require more equipment and greater sample manipulation. Furthermore, DO meters using a membrane electrode are suitable for highly polluted waters because the probe is completely submersible and is not susceptible to interference caused by color, turbidity, or colloidal material or suspended matter.

Subject GROUNDWATER SAMPLE ACQUISITION AND ONSITE WATER QUALITY TESTING	Number SA-1.1	Page 18 of 35
	Revision 7	Effective Date 04/07/2008

6.5.4.2 Principles of Equipment Operation

DO probes are normally electrochemical cells that have two solid metal electrodes of different nobility immersed in an electrolyte. The electrolyte is retained by an oxygen-permeable membrane. The metal of highest nobility (the cathode) is positioned at the membrane. When a suitable potential exists between the two metals, reduction of oxygen to hydroxide ion (OH⁻) occurs at the cathode surface. An electrical current is developed that is directly proportional to the rate of arrival of oxygen molecules at the cathode. This rate is proportional to the oxygen concentration in the water being measured.

Because the current produced in the probe is directly proportional to the rate of arrival of oxygen at the cathode, it is important that a fresh supply of sample always be in contact with the membrane. Otherwise, the oxygen in the aqueous layer along the membrane is quickly depleted and false low readings are obtained. It is therefore necessary to stir the sample (or the probe) constantly to maintain fresh solution near the membrane interface. Stirring, however, shall not be so vigorous that additional oxygen is introduced through the air-water interface at the sample surface. To avoid this possibility, some probes are equipped with stirrers to agitate the solution near the probe, leaving the surface of the solution undisturbed.

DO probes are relatively unaffected by interferences. Interferences that can occur are reactions with oxidizing gases such as chlorine or with gases such as hydrogen sulfide that are not easily depolarized from the indicating electrode. If a gaseous interference is suspected, it shall be noted in the field logbook and checked if possible. Temperature variations can also cause interference because probes exhibit temperature sensitivity. Automatic temperature compensation is normally provided by the manufacturer. This compensation can counteract some of the temperature effects but not all of them.

6.5.4.3 Equipment

The following equipment is needed to measure DO concentrations:

- A stand-alone portable DO meter or combination meter equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).
- Sufficient cable to allow the probe to contact the sample.
- Manufacturer's operation manual.

6.5.4.4 Measurement Techniques for Dissolved Oxygen Determination

DO probes differ as to instructions for use. Follow the manufacturer's instructions to obtain an accurate reading. The following general steps shall be used to measure DO concentrations:

1. Check the DO meter batteries before going to the field.
2. Condition the probe in a water sample for as long a period as practical before use in the field. Long periods of dry storage followed by short periods of use in the field may result in inaccurate readings.
3. Calibrate the instrument in the field according to manufacturer's recommendations or in a freshly air-saturated water sample of known temperature.
4. Record all pertinent information on a water quality meter calibration log sheet or equivalent electronic form.

Subject GROUNDWATER SAMPLE ACQUISITION AND ONSITE WATER QUALITY TESTING	Number SA-1.1	Page 19 of 35
	Revision 7	Effective Date 04/07/2008

5. Rinse the probe with deionized water.
6. Immerse the probe in the sample. Be sure to provide for sufficient flow past the membrane by stirring the sample. Probes without stirrers placed in wells may be moved up and down to achieve the required mixing.
7. Record the DO content and temperature of the sample in a field logbook or on a sample log sheet or equivalent electronic form.
8. Rinse the probe with deionized water.
9. Recalibrate the probe when the membrane is replaced, or as needed. Follow the manufacturer's instructions.

Note that in-situ placement of the probe is preferable because sample handling is not involved. This however may not always be practical.

Special care shall be taken during sample collection to avoid turbulence that can lead to increased oxygen solubilization and positive test interferences.

6.5.5 Measurement of Oxidation-Reduction Potential

6.5.5.1 General

ORP provides a measure of the tendency of organic or inorganic chemicals to exist in an oxidized state. The ORP parameter therefore provides evidence of the likelihood of anaerobic degradation of biodegradable organics or the ratio of activities of reduced to oxidized species in the sample.

6.5.5.2 Principles of Equipment Operation

When an inert metal electrode, such as platinum, is immersed in a solution, a potential is developed at that electrode depending on the ions present in the solution. If a reference electrode is placed in the same solution, an ORP electrode pair is established. This electrode pair allows the potential difference between the two electrodes to be measured and is dependent on the concentration of the ions in solution. By this measurement, the ability to oxidize or reduce species in solution may be determined. Supplemental measurements, such as DO, may be correlated with ORP to provide knowledge of the quality of the solution, water, or wastewater.

6.5.5.3 Equipment

The following equipment is needed for measuring the ORP of a solution:

- A combination meter with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).
- Reference solution as specified by the manufacturer.
- Manufacturer's operation manual.

Subject GROUNDWATER SAMPLE ACQUISITION AND ONSITE WATER QUALITY TESTING	Number SA-1.1	Page 20 of 35
	Revision 7	Effective Date 04/07/2008

6.5.5.4 Measurement Techniques for Oxidation-Reduction Potential

The following procedure is used for measuring ORP:

1. Check the equipment using the manufacturer's recommended reference solution and check its batteries before going to the field.
2. Thoroughly rinse the electrode with deionized water.
3. If the probe does not respond properly to the recommended reference solution, verify the sensitivity of the electrodes by noting the change in millivolts when the pH of a test solution is altered. The ORP will increase when the pH of a test solution decreases, and the ORP will decrease when the test solution pH is increased. Place the sample in a clean container and agitate the sample. Insert the electrodes and note that the ORP drops sharply when the caustic is added (i.e., pH increases) thus indicating that the electrodes are sensitive and operating properly. If the ORP increases sharply when the caustic is added, the polarity is reversed and must be corrected in accordance with the manufacturer's instructions or the probe should be replaced.
4. Record all pertinent information on a water quality meter calibration log sheet or equivalent electronic form.

6.5.6 Measurement of Salinity

6.5.6.1 General

Salinity is a unitless property of industrial and natural waters. It is the measurement of dissolved salts in a given mass of solution. Most field meters determine salinity automatically from conductivity and temperature. The displayed value will be displayed in either parts per thousand (ppt) or percent (e.g., 35 ppt equals 3.5 percent).

6.5.6.2 Principles of Equipment Operation

Salinity is determined automatically from the meter's conductivity and temperature readings according to algorithms (such as are found in Standard Methods for the Examination of Water and Wastewater). Depending on the meter, the results are displayed in either ppt or percent. The salinity measurements are carried out in reference to the conductivity of standard seawater (corrected to salinity = 35 ppt).

6.5.6.3 Equipment

The following equipment is needed for salinity measurements:

- A multi-parameter water quality meter capable of measuring conductivity and temperature and converting them to salinity (e.g., Horiba U-22 or YSI 600 series).
- Calibration solution as specified by the manufacturer.
- Manufacturer's operation manual.

Subject GROUNDWATER SAMPLE ACQUISITION AND ONSITE WATER QUALITY TESTING	Number SA-1.1	Page 21 of 35
	Revision 7	Effective Date 04/07/2008

6.5.6.4 Measurement Techniques for Salinity

The steps involved in taking salinity measurements are as follows (standardization shall be conducted according to manufacturer's instructions):

1. Check the expiration date of the solutions used for field calibration and replace them if they are expired.
2. Check batteries and calibrate the meter before going into the field.
3. Calibrate on a daily use basis, according to the manufacturer's instructions and record all pertinent information on a water quality meter calibration log sheet or equivalent electronic form.
4. Rinse the cell with the sample to be tested. This is typically accomplished as the probe is placed in line during the collection of the purge water up to the time of sample acquisition.
5. Immerse the multi-probe in the sample and measure the salinity. Read and record the results in a field logbook or on a sample log sheet or equivalent electronic form.
6. Rinse the probes with deionized water.

6.5.7 Measurement of Turbidity

6.5.7.1 General

Turbidity is an expression of the optical property that causes light to be scattered and absorbed rather than transmitted in a straight line through the sample. Turbidity in water is caused by suspended matter such as clay, silt, or other finely divided organic and inorganic matter and microscopic organisms including plankton.

It is important to obtain a turbidity reading immediately after taking a sample because irreversible changes in turbidity may occur if the sample is stored too long.

6.5.7.2 Principles of Equipment Operation

Turbidity is measured by the Nephelometric Method, which is based on a comparison of the intensity of light scattered by the sample under defined conditions with the intensity of light scattered by a standard reference suspension under the same conditions. The higher the scattered light intensity, the higher the turbidity.

Formazin polymer is used as the reference turbidity standard suspension because of its ease of preparation combined with a higher reproducibility of its light-scattering properties than clay or turbid natural water. The turbidity of a specified concentration of formazin suspension is defined as 40 nephelometric units. This same suspension has an approximate turbidity of 40 Jackson units when measured on the candle turbidimeter. Therefore, nephelometric turbidity units (NTUs) based on the formazin preparation will approximate units derived from the candle turbidimeter but will not be identical to them.

Subject GROUNDWATER SAMPLE ACQUISITION AND ONSITE WATER QUALITY TESTING	Number SA-1.1	Page 22 of 35
	Revision 7	Effective Date 04/07/2008

6.5.7.3 Equipment

The following equipment is needed for turbidity measurements:

- A turbidity meter (e.g., LaMotte 2020) that calibrates easily using test cells with standards of 0.0, 1.0, and 10 NTUs, or a combination meter equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).
- Calibration solution and sample tubes, as specified by the manufacturer.
- Manufacturer's operation manual.

6.5.7.4 Measurement Techniques for Turbidity

The steps involved in taking turbidity measurements utilizing an electrode (e) or light meter (l) are listed below (standardization shall be done according to manufacturer's instructions):

1. Check the expiration date of the solutions used for field calibration and replace them if they are expired.
2. Check batteries and calibrate the instrument before going into the field.
3. Calibrate on a daily basis according to the manufacturer's instructions, and record all pertinent information on a turbidity meter calibration log sheet (Attachment C) or equivalent electronic form.
4. When using the YSI and/or Horiba U-22, rinse the electrode with one or more portions of the sample to be tested or with deionized water.
5. When using the Lamotte 2020, fill the light meter's glass test cell with approximately 5 mL of sample, screw on the cap, wipe off glass to remove all residue that could intercept the instrument's light beam, place the test cell in the light meter, and close the lid.
6. Immerse the electrode in the sample and measure the turbidity.
7. The reading must be taken immediately because suspended solids will settle over time resulting in a lower, inaccurate turbidity reading.
8. Read and record the results in a field logbook or on a sample log sheet or equivalent electronic form. Include a physical description of the sample, including color, qualitative estimate of turbidity, etc.
9. Rinse the electrode or test cell with deionized water.

6.6 Sampling

6.6.1 Sampling Plan

The sampling approach consisting of the following shall be developed as part of the project planning documents approved prior to beginning work in the field:

- Background and objectives of sampling.

Subject GROUNDWATER SAMPLE ACQUISITION AND ONSITE WATER QUALITY TESTING	Number SA-1.1	Page 23 of 35
	Revision 7	Effective Date 04/07/2008

- Brief description of area and waste characterization.
- Identification of sampling locations, with map or sketch, and applicable well construction data (well size, depth, screened interval, reference elevation).
- Intended number, sequence, volumes, and types of samples. If the relative degree of contamination between wells is insignificant, a sampling sequence that facilitates sampling logistics may be followed. Where some wells are known or strongly suspected of being highly contaminated, these shall be sampled last to reduce the risk of cross-contamination between wells. In situations where the well is not well-characterized and the nature or extent of airborne contamination is unknown, it is recommended that head space analysis using a photoionization detector (PID) or flame ionization detector (FID) is performed to rate the wells, sampling from least contaminated to most contaminated. Refer to the project-specific HASP for appropriate information and direction on air monitoring requirements.
- Sample preservation requirements.
- Work schedule.
- List of team members.
- List of observers and contacts.
- Other information, such as the necessity for a warrant or permission of entry, requirements for split samples, access problems, location of keys, etc.
- The FOL shall ensure that the sampling method(s) to be employed is accurately represented in the HASP, indicating the types of sampling to be employed and the hazards. If the methods are not accurately represented, the FOL should rectify this with the HASP author.
- The FOL shall ensure that sampling teams understand the sampling approach that they are to follow. Where sampling teams are made up of personnel from multiple locations, personal sampling experiences may vary. Therefore the FOL shall review project-specific requirements, SOPs, and protocol to be followed. The FOL will conduct periodic surveys to ensure that these methods are being completed per his/her direction.

6.6.2 Sampling Methods as Related to Low-Flow Sampling

The collection of a groundwater sample consists of the following steps:

1. Ensure the safety of the sample location. Take a few minutes to evaluate the area for physical hazards (trip hazards, uneven ground, overhanging branches, etc.) and natural hazards (snakes, bees, spiders, etc.) that may exist in the area or that may have constructed nests in the well head. Snakes often like to sun themselves on concrete well pads. Follow provisions in the project-specific HASP and/or HSGM for addressing natural hazards.
2. As indicated earlier, some monitoring wells have the potential to contain pressurized headspace (e.g., through the generation of gases from contaminated groundwater, due to biological processes, degradation of contaminants, or simply based on location such as near a landfill or in areas that intersect lithological abnormalities) or through intentional artificial means such as those associated with air sparging systems. Injection or extraction wells may be artificially pressurized and may remain so for several days after the system has been turned off. This presents a hazard to people opening

Subject GROUNDWATER SAMPLE ACQUISITION AND ONSITE WATER QUALITY TESTING	Number SA-1.1	Page 24 of 35
	Revision 7	Effective Date 04/07/2008

these wells. The Field Sampling Technician shall employ the following practices to minimize these hazards:

- Wear safety glasses to protect the eyes. If site-specific observations and conditions indicate that the wells may be pressurized, wear a full-face shield over the safety impact eye protection.
- DO NOT place your face or any other part of your body over the well when opening because this may place you in a strike zone.
- Open the well cover at arms length, then step away and allow the well to off gas and stabilize.

Follow directions provided in the project-specific HASP, Work Plan and/or Sampling Plan pertaining to the use of volatile chemical detection equipment (PID or FID) within the breathing zone of the sampler during sampling to determine the need to retreat from the work area and/or for the use of respiratory protection (as specified in the HASP).

3. When proper respiratory protection has been donned, sound the well for total depth and water level (using clean equipment) and record these data on a groundwater sampling log sheet or equivalent electronic form; then calculate the fluid volume in the well pipe (as previously described in this SOP). It is imperative that downhole equipment be adequately decontaminated between wells to prevent cross-contamination. Just as sampling occurs from the least contaminated to the most contaminated, it is also recommended that groundwater level measurements be taken in this manner.
4. Calculate volume of well water to be removed as described in Section 6.3.
5. Select the appropriate purging equipment (see Attachment A to this SOP) or as designated within your Work Plan/Sampling Plan. If an electric submersible pump with packer is chosen, go to Step 10.
6. Lower the purging equipment or intake into the well to a short distance below the water level or mid-screen as indicated in project-specific documentation and begin water removal. Remember that some contaminants are "bottom dwellers," and in these cases, project-specific direction may specify placing the intake just above (1 to 2 feet) the well bottom. Secure the pump intake at the well and secure the effluent at the collection container and begin pumping. The pumping rate will be determined based on the decrease in the water level (see Section 6.7) or as directed in your project-specific documents or this SOP. Purge water is generally collected in a 5-gallon bucket or similar open- or closed-top container. To minimize the potential for spills and back injuries, do not fill 5-gallon buckets beyond approximately 80 percent of their capacity. Dispose of purge water as indicated in the planning document(s). Where necessary, slow the pumping rate or lower the pump intake as required to maintain submergence.
7. Estimate the approximate rate of discharge frequently and record it on the Low Flow Purge Data Sheet (see Attachment D). Estimate flow rate by noting the amount of discharge in a bucket or graduated cylinder per unit time using a watch with a second hand or a stopwatch.
8. Observe the peristaltic pump tubing intake for degassing "bubbles." If bubbles are abundant and the intake is fully submerged, this pump is not suitable for collecting samples for volatile organics.
9. Purge a minimum of three to five casing volumes before sampling (or as directed by the site-specific SAP). In low-permeability strata (i.e., if the well is pumped to dryness), one volume will suffice. Allow the well to recover to 75 percent of initial water level before sampling. Do not overfill purge containers because this increases the potential for spills and lifting injuries.

Subject GROUNDWATER SAMPLE ACQUISITION AND ONSITE WATER QUALITY TESTING	Number SA-1.1	Page 25 of 35
	Revision 7	Effective Date 04/07/2008

10. If sampling using a submersible pump, lower the pump intake to mid-screen (or the middle of the open section in uncased wells) and collect the sample. If sampling with a bailer, lower the bailer to just below the water surface.
11. For pump and packer assemblies only: Lower the assembly into the well so that the packer is positioned just above the screen or open section. Inflate the packer. Purge a volume equal to at least twice the screened interval (or unscreened open section volume below the packer) before sampling. Packers shall always be tested in a casing section above ground to determine proper inflation pressures for good sealing.
12. If the recovery time of the well is very slow (e.g., 24 hours or greater), sample collection can be delayed until the following day. If the well has been purged early in the morning, sufficient water may be standing in the well by the day's end to permit sample collection. If the well is incapable of producing a sufficient volume of sample at any time, take the largest quantity available and record this occurrence in the site logbook or equivalent electronic form. When this occurs, contact the analytical laboratory to alert them that a reduced sample volume(s) will be submitted for analysis.
13. Fill sample containers and preserve and label them as described in SOP SA-6.1. Many sample bottles will contain preservative when they are shipped to the field. In those cases, do not add preservative.
14. Replace the well cap and lock it as appropriate. Make sure the well is readily identifiable as the source of the sample.
15. Process sample containers as described in SOP SA-6.1.
16. Decontaminate equipment as described in SOP SA-7.1.

6.7 Low-Flow Purging and Sampling

6.7.1 Scope and Application

Low-flow purging and sampling techniques may be required for groundwater sampling activities. The purpose of low-flow purging and sampling is to collect groundwater samples that contain "representative" amounts of mobile organic and inorganic constituents in the vicinity of the selected open well interval, at or near natural flow conditions. This minimum-stress procedure emphasizes negligible water level drawdown and low pumping rates to collect samples with minimal alterations in water chemistry. This procedure is designed primarily to be used in wells with a casing diameter of 1 inch or more and a saturated screen length, or open interval, of 10 feet or less. Samples obtained are suitable for analyses of common types of groundwater contaminants (volatile and semivolatile organic compounds, pesticides, polychlorinated biphenyls [PCBs], metals and other inorganic ions [cyanide, chloride, sulfate, etc.]). This low-flow procedure is not designed for collection of non-aqueous phase liquid samples from wells containing light or dense non-aqueous phase liquids (LNAPLs or DNAPLs).

This procedure is flexible for various well construction types and groundwater yields. The goal of the procedure is to obtain a turbidity level of less than 10 NTUs and to achieve a water level drawdown of less than 0.3 foot during purging and sampling. If these goals cannot be achieved, sample collection can take place provided that the remaining criteria in this procedure are met.

6.7.2 Equipment

The following equipment is required (as applicable) for low-flow purging and sampling:

Subject GROUNDWATER SAMPLE ACQUISITION AND ONSITE WATER QUALITY TESTING	Number SA-1.1	Page 26 of 35
	Revision 7	Effective Date 04/07/2008

- Adjustable rate submersible pump (e.g., centrifugal or bladder pump constructed of stainless steel or Teflon).
- Disposable clear plastic bottom-filling bailers to be used to check for and obtain samples of LNAPLs or DNAPLs.
- Tubing – Teflon, Teflon-lined polyethylene, polyethylene, polyvinyl chloride (PVC), Tygon, or stainless steel tubing can be used to collect samples for analysis, depending on the analyses to be performed and regulatory requirements.
- Water level measuring device with 0.01-foot accuracy (electronic devices are preferred for tracking water level drawdown during all pumping operations).
- Interface probe.
- Flow measurement supplies.
- Power source (generator, nitrogen tank, etc.). If a gasoline generator is used, it must be located downwind and at a safe distance from the well so that the exhaust fumes do not contaminate the samples.
- Indicator parameter monitoring instruments – pH, turbidity, specific conductance, and temperature. Use of a flow-through cell is recommended. Optional indicators - ORP, salinity, and DO. A flow-through cell (also referred to as an in-line sample chamber) is required.
- Standards to perform field calibration of instruments.
- Decontamination supplies.
- Logbook(s) and other forms (see Attachments B through D) or equivalent electronic form(s).
- Sample bottles.
- Sample preservation supplies (as required by the analytical methods).
- Sample tags and/or labels.
- Well construction data, location map, field data from last sampling event (if available).
- Field Sampling Plan.
- PID or FID instrument for measuring volatile organic compounds (VOCs) per the HASP.

6.7.3 Purging and Sampling Procedure

1. Open the monitoring well as stated earlier and step away. Prepare sampling equipment while allowing 3 to 5 minutes to allow the water level to reach equilibrium. In situations where VOCs are the primary contaminants of concern, air monitoring of the samplers' breathing zone areas may be required by the HASP (typically with a PID or FID).

Subject GROUNDWATER SAMPLE ACQUISITION AND ONSITE WATER QUALITY TESTING	Number SA-1.1	Page 27 of 35
	Revision 7	Effective Date 04/07/2008

2. Measure the water level immediately prior to placing the pump in the well and record the water level on the Low-Flow Purge Data Form or equivalent electronic form immediately prior to placing the pump or tubing into the well.
3. Lower the measuring device further into the well to collect the total depth measurement. Again wait 3 to 5 minutes to allow the well to equilibrate to the initial water level prior to placing the pump or pump intake in the well.
4. Record the total well depth on the Low-Flow Purge Data Form or equivalent electronic form immediately prior to placing the pump or tubing into the well
5. Lower the pump or tubing slowly into the well so that the pump intake is located at the center of the saturated screen length of the well. If possible, keep the pump intake at least 2 feet above the bottom of the well to minimize mobilization of sediment that may be present in the bottom of the well. Collection of turbidity-free water samples may be difficult if there is 3 feet or less of standing water in the well.
6. Start with the initial pump rate set at approximately 0.1 liter per minute. Use a graduated cylinder and stopwatch to measure the pumping rate. Adjust the pumping rates as necessary to prevent drawdown from exceeding 0.3 foot during purging. If no drawdown is noted, the pump rate may be increased (to a maximum of 0.4 liter per minute) to expedite the purging and sampling event. The pump rate will be reduced if turbidity is greater than 10 NTUs after all other field parameters have stabilized. If groundwater is drawn down below the top of the well screen, purging shall cease or the well shall be pumped to dryness and then allowed to recover before purging continues. Well recovery to 75 percent is necessary prior to sampling. Slow-recovering wells should be identified and purged at the beginning of the workday to maximize field work efficiency. If possible, samples should be collected from these wells within the same workday and no later than 24 hours after the end of purging.
7. Measure the water level in the well every 5 to 10 minutes using the water level meter. Record the well water level on the Low Flow Purge Data Form (Attachment D) or equivalent electronic form.
8. Record on the Low Flow Purge Data Form every 5 to 10 minutes the water quality parameters (pH, specific conductance, temperature, turbidity, ORP, DO, and salinity or as specified by the approved site-specific planning document) measured by the water quality meter and turbidity meter. If the cell needs to be cleaned during purging operations, continue pumping (allow the pump to discharge into a container) and disconnect the cell. Rinse the cell with distilled/deionized water. After cleaning is completed, reconnect the flow-through cell and continue purging. Document the cell cleaning on the Low-Flow Purge Data Form or equivalent electronic form.
9. Estimate the flow rate by noting the amount of discharge in a graduated cylinder per unit time using a watch with a second hand. Remeasure the flow rate any time the pump rate is adjusted and periodically during purging. This will determine if a reduction in rate has occurred due to possible battery depletion.
10. During purging, check for the presence of bubbles in the flow-through cell. The presence of bubbles is an indication that connections are not tight. If bubbles are observed, check for loose connections and tighten, repair, or replace them as necessary to achieve a tight connection.
11. Wait until stabilization is achieved, or a minimum of two saturated screen volumes have been removed and three consecutive readings, taken at 5 to 10 minute intervals, are within the following limits, then begin sampling:
 - pH ± 0.2 standard units

Subject GROUNDWATER SAMPLE ACQUISITION AND ONSITE WATER QUALITY TESTING	Number SA-1.1	Page 28 of 35
	Revision 7	Effective Date 04/07/2008

- Specific conductance $\pm 10\%$
- Temperature $\pm 10\%$
- Turbidity less than 10 NTUs
- DO $\pm 10\%$

12. If the above conditions have not been met after the well has been purged for 4 hours, purging will be considered complete and sampling can begin. Record the final well stabilization parameters from the Low-Flow Purge Data Form onto the Groundwater Sample Log Form or equivalent electronic form.

NOTE: VOC samples are preferably collected first, directly into pre-preserved sample containers. Fill all sample containers by allowing the pump discharge to flow gently down the inside of the container with minimal turbulence.

13. If the water column in the pump tubing collapses (water does not completely fill the tubing) before exiting the tubing, use one of the following procedures to collect VOC samples:

- Collect samples for non-VOC analyses first, then increase the flow rate incrementally until the water column completely fills the tubing, collect the sample for VOCs, and record the new flow rate.
- Reduce the diameter of the existing tubing until the water column fills the tubing either by adding a connector (Teflon or stainless steel) or clamp, which should reduce the flow rate by constricting the end of the tubing. Proceed with sample collection.
- Insert a narrow-diameter Teflon tube into the pump's tubing so that the end of the tubing is in the water column and the other end of the tubing protrudes beyond the pump's tubing, then collect the sample from the narrow diameter tubing.
- Prepare samples for shipping as per SOP SA-6.1.

7.0 REFERENCES

American Public Health Association, 1989. Standard Methods for the Examination of Water and Wastewater, 17th Edition, APHA, Washington, D.C.

Barcelona, M. J., J. P. Gibb and R. A. Miller, 1983. A Guide to the Selection of Materials for Monitoring Well Construction and Groundwater Sampling. ISWS Contract Report 327, Illinois State Water Survey, Champaign, Illinois.

Johnson Division, UOP, Inc. 1975. Ground Water and Wells, A Reference Book for the Water Well Industry. Johnson Division, UOP, Inc., Saint Paul, Minnesota.

Nielsen, D. M. and G. L. Yeates, 1985. A Comparison of Sampling Mechanisms Available for Small-Diameter Ground Water Monitoring Wells. Ground Water Monitoring Review 5:83-98.

Scalf, M. R., J. F. McNabb, W. J. Dunlap, R. L. Crosby and J. Fryberger, 1981. Manual of Ground Water Sampling Procedures. R. S. Kerr Environmental Research Laboratory, Office of Research and Development, U.S. EPA, Ada, Oklahoma.

U.S. EPA, 1979. Methods for Chemical Analysis of Water and Wastes. EPA-600/4-79-020.

Subject GROUNDWATER SAMPLE ACQUISITION AND ONSITE WATER QUALITY TESTING	Number SA-1.1	Page 29 of 35
	Revision 7	Effective Date 04/07/2008

U.S. EPA, 1980. Procedures Manual for Ground Water Monitoring at Solid Waste Disposal Facilities. Office of Solid Waste, United States Environmental Protection Agency, Washington, D.C.

U.S. EPA, 1994. Groundwater Sampling Procedure - Low Flow Purge and Sampling (Draft Final). U.S. Environmental Protection Agency, Region I.

U.S. Geological Survey, 1984. National Handbook of Recommended Methods for Water Data Acquisition, Chapter 5: Chemical and Physical Quality of Water and Sediment. U.S. Department of the Interior, Reston, Virginia.

Subject GROUNDWATER SAMPLE ACQUISITION AND ONSITE WATER QUALITY TESTING	Number SA-1.1	Page 30 of 35
	Revision 7	Effective Date 04/07/2008

**ATTACHMENT A
PURGING EQUIPMENT SELECTION**

Diameter Casing		Bailer	Peristaltic Pump	Vacuum Pump	Air-lift	Diaphragm "Trash" Pump	Submersible Diaphragm Pump	Submersible Electric Pump	Submersible Electric Pump w/Packer
1.25-Inch	Water level <25 feet	X	X	X	X	X			
	Water Level >25 feet	X			X				
2-Inch	Water level <25 feet	X	X	X	X	X	X		
	Water Level >25 feet	X			X		X		
4-Inch	Water level <25 feet	X	X	X	X	X	X	X	X
	Water Level >25 feet	X			X		X	X	X
6-Inch	Water level <25 feet				X	X		X	X
	Water Level >25 feet				X			X	X
8-Inch	Water level <25 feet				X	X		X	X
	Water Level >25 feet				X			X	X

ATTACHMENT A
PURGING EQUIPMENT SELECTION
PAGE 2

Manufacturer	Model Name/Number	Principle of Operation	Maximum Outside Diameter/L ength (Inches)	Construction Materials (w/Lines and Tubing)	Lift Range (ft)	Delivery Rates or Volumes	1982 Price (Dollars)	Comments
BarCad Systems, Inc.	BarCad Sampler	Dedicated; gas drive (positive displacement)	1.5/16	PE, brass, nylon, aluminum oxide	0-150 with std. tubing	1 liter for each 10-15 feet of submergence	\$220-350	Requires compressed gas; custom sizes and materials available; acts as piezometer.
Cole-Parmer Inst. Co.	Master Flex 7570 Portable Sampling Pump	Portable; peristaltic (suction)	<1.0/NA	(not submersible) Tygon®, silicone Viton®	0-30	670 mL/min with 7015-20 pump head	\$500-600	AC/DC; variable speed control available; other models may have different flow rates.
ECO Pump Corp.	SAMPLifier	Portable; venturi	<1.5 or <2.0/NA	PP, PE, PVC, SS, Teflon®, Tefzel®	0-100	0-500 mL/min depending on lift	\$400-700	AC, DC, or gasoline-driven motors available; must be primed.
Geltek Corp.	Bailer 219-4	Portable; grab (positive displacement)	1.66/38	Teflon®	No limit	1,075 mL	\$120-135	Other sizes available.
GeoEngineering, Inc.	GEO-MONITOR	Dedicated; gas drive (positive displacement)	1.5/16	PE, PP, PVC, Viton®	Probably 0-150	Approximately 1 liter for each 10 feet of submergence	\$185	Acts as piezometer; requires compressed gas.
Industrial and Environmental Analysts, Inc. (IEA)	Aquarius	Portable; bladder (positive displacement)	1.75/43	SS, Teflon®, Viton®	0-250	0-2,800 mL/min	\$1,500-3,000	Requires compressed gas; other models available; AC, DC, manual operation possible.
IEA	Syringe Sampler	Portable; grab (positive displacement)	1.75/43	SS, Teflon®	No limit	850 mL sample volume	\$1,100	Requires vacuum and/or pressure from hand pump.
Instrument Specialties Co. (ISCO)	Model 2600 Well Sampler	Portable; bladder (positive displacement)	1.75/50	PC, silicone, Teflon®, PP, PE, Detrin®, acetal	0-150	0-7,500 mL/min	\$990	Requires compressed gas (40 psi minimum).
Keck Geophysical Instruments, Inc.	SP-81 Submersible Sampling Pump	Portable; helical rotor (positive displacement)	1.75/25	SS, Teflon®, PP, EPDM, Viton®	0-160	0-4,500 mL/min	\$3,500	DC operated.
Leonard Mold and Die Works, Inc.	GeoFilter Small Diameter Well Pump (#0500)	Portable; bladder (positive displacement)	1.75/38	SS, Teflon®, PC, Neoprene®	0-400	0-3,500 mL/min	\$1,400-1,500	Requires compressed gas (55 psi minimum); pneumatic or AC/DC control module.
Oil Recovery Systems, Inc.	Surface Sampler	Portable; grab (positive displacement)	1.75/12	acrylic, Detrin®	No limit	Approximately 250 mL	\$125-160	Other materials and models available; for measuring thickness of "floating" contaminants.
Q.E.D. Environmental Systems, Inc.	Well Wizard® Monitoring System (P-100)	Dedicated; bladder (positive displacement)	1.66/36	PVC	0-230	0-2,000 mL/min	\$300-400	Requires compressed gas; piezometric level indicator; other materials available.

Subject
GROUNDWATER SAMPLE
ACQUISITION AND ONSITE
WATER QUALITY TESTING

Number
SA-1.1
Revision
7

Page
31 of 35
Effective Date
04/07/2008

ATTACHMENT A
PURGING EQUIPMENT SELECTION
PAGE 3

Manufacturer	Model Name/Number	Principle of Operation	Maximum Outside Diameter/Length (Inches)	Construction Materials (w/Lines and Tubing)	Lift Range (ft)	Delivery Rates or Volumes	1982 Price (Dollars)	Comments
Randolph Austin Co.	Model 500 Vari-Flow Pump	Portable; peristaltic (suction)	<0.5/NA	(Not submersible) Rubber, Tygon®, or Neoprene®	0-30	See comments	\$1,200-1,300	Flow rate dependent on motor and tubing selected; AC operated; other models available.
Robert Bennett Co.	Model 180	Portable; piston (positive displacement)	1.8/22	SS, Teflon®, Delrin® PP, Viton®, acrylic, PE	0-500	0-1,800 mL/min	\$2,600-2,700	Requires compressed gas; water level indicator and flow meter; custom models available.
Slope Indicator Co. (SINCO)	Model 514124 Pneumatic Water Sampler	Portable; gas drive (positive displacement)	1.9/18	PVC, nylon	0-1,100	250 mL/flushing cycle	\$250-350	Requires compressed gas; SS available; piezometer model available; dedicated model available.
Solinst Canada Ltd.	5W Water Sampler	Portable; grab (positive displacement)	1.9/27	PVC, brass, nylon, Neoprene®	0-330	500 mL	\$1,300-1,800	Requires compressed gas; custom models available.
TIMCO Mfg. Co., Inc.	Std. Bailer	Portable; grab (positive displacement)	1.66/Custom	PVC, PP	No limit	250 mL/ft of bailer	\$20-60	Other sizes, materials, models available; optional bottom-emptying device available; no solvents used.
TIMCO	Air or Gas Lift Sampler	Portable; gas drive (positive displacement)	1.66/30	PVC, Tygon®, Teflon®	0-150	350 mL/flushing cycle	\$100-200	Requires compressed gas; other sizes, materials, models available; no solvents used.
Tole Devices Co.	Sampling Pump	Portable; bladder (positive displacement)	1.38/48	SS, silicone, Delrin®, Tygon®	0-125	0-4,000 mL/min	\$800-1,000	Compressed gas required; DC control module; custom built.

Construction Material Abbreviations:

PE	Polyethylene
PP	Polypropylene
PVC	Polyvinyl chloride
SS	Stainless steel
PC	Polycarbonate
EPDM	Ethylene-propylene diene (synthetic rubber)

Other Abbreviations:

AC	Alternating current	NA	Not applicable
DC	Direct current		

NOTE: Other manufacturers market pumping devices which could be used for groundwater sampling, though not expressly designed for this purpose. The list is not meant to be all-inclusive and listing does not constitute endorsement for use. Information in the table is from sales literature and/or personal communication. No skimmer, scavenger-type, or high-capacity pumps are included.

Source: Barcelona et al., 1983.

Subject
GROUNDWATER SAMPLE
ACQUISITION AND ONSITE
WATER QUALITY TESTING

Number
SA-1.1
Revision
7

Page
32 of 35
Effective Date
04/07/2008



STANDARD OPERATING PROCEDURES

Number SA-1.3	Page 1 of 31
Effective Date 04/072008	Revision 9
Applicability Tetra Tech NUS, Inc.	
Prepared Earth Sciences Department	
Approved Tom Johnston <i>T.E. Johnston</i>	

Subject
SOIL SAMPLING

TABLE OF CONTENTS

<u>SECTION</u>	<u>PAGE</u>
1.0 PURPOSE.....	2
2.0 SCOPE.....	2
3.0 GLOSSARY	2
4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS	3
5.0 HEALTH AND SAFETY	4
6.0 PROCEDURES.....	5
6.1 Overview	6
6.2 Soil Sample Collection	6
6.2.1 Procedure for Preserving and Collecting Soil Samples for Volatile Organic Compound Analysis	6
6.2.2 Procedure for Collecting Soil Samples for Non-Volatile Analyses	9
6.2.3 Procedure for Collecting Undisturbed Soil Samples	10
6.3 Surface Soil Sampling	13
6.4 Near-Surface Soil Sampling	14
6.5 Subsurface Soil Sampling With a Hand Auger	15
6.6 Subsurface Soil Sampling with a Split-Barrel Sampler.....	17
6.7 Subsurface Soil Sampling Using Direct-Push Technology	18
6.8 Excavation and Sampling of Test Pits and Trenches.....	18
6.8.1 Applicability	18
6.8.2 Test Pit and Trench Excavation.....	19
6.8.3 Sampling in Test Pits and Trenches.....	21
6.8.4 Backfilling of Trenches and Test Pits	25
6.9 Records.....	25
7.0 REFERENCES.....	26
 <u>ATTACHMENTS</u>	
A SOIL & SEDIMENT SAMPLE LOG SHEET.....	28
B SPLIT-SPOON SAMPLER	29
C TEST PIT LOG.....	30
D REMOTE SAMPLE HOLDER FOR TEST PIT/TRENCH SAMPLING.....	31

Subject SOIL SAMPLING	Number SA-1.3	Page 2 of 31
	Revision 9	Effective Date 04/07/2008

1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the procedures to be used to collect surface, near-surface, and subsurface soil samples. Additionally, it describes the methods for sampling of test pits and trenches to determine subsurface soil and rock conditions and for recovery of small-volume or bulk samples from pits.

2.0 SCOPE

This document applies to the collection of surface, near-surface, and subsurface soil samples exposed through hand digging, hand augering, drilling, or machine excavating at hazardous substance sites for laboratory testing, onsite visual examination, and onsite testing.

3.0 GLOSSARY

Composite Sample - A composite sample is a combination of more than one grab sample from various locations and/or depths and times that is homogenized and treated as one sample. This type of sample is usually collected when determination of an average waste concentration for a specific area is required. Composite samples shall not be collected for volatile organics analysis.

Confined Space - As stipulated in 29 Code of Federal Regulations (CFR) 1910.146, a confined space means a space that: (1) is large enough and so configured that an employee can bodily enter and perform assigned work; (2) has limited or restricted means for entry or exit (e.g., tanks, vessels, silos, storage bins, hoppers, vaults, pits, and excavations); and (3) is not designed for continuous employee occupancy. TtNUS considers all confined space as permit-required confined spaces.

Grab Sample - One sample collected at one location and at one specific time.

Hand Auger - A sampling device used to extract soil from the ground.

Representativeness – A qualitative description of the degree to which an individual sample accurately reflects population characteristics or parameter variations at a sampling point. It is therefore an important characteristic not only of assessment and quantification of environmental threats posed by the site, but also for providing information for engineering design and construction. Proper sample location selection and proper sample collection methods are important to ensure that a truly representative sample has been collected.

Sample for Non-Volatile Analyses - Includes all chemical parameters other than volatile organics (e.g., semivolatiles, pesticides/PCBs, metals, etc.) and those engineering parameters that do not require undisturbed soil for their analysis.

Split-Barrel Sampler - A steel tube, split in half lengthwise, with the halves held together by threaded collars at either end of the tube. Also called a split-spoon sampler, this device can be driven into resistant materials using a drive weight mounted in the drilling string. A standard split-barrel sampler is typically available in two common lengths, providing either 20-inch or 26-inch longitudinal clearance for obtaining 18-inch or 24-inch-long samples, respectively. These split-barrel samplers commonly range in size from 2 to 3.5 inches OD. The larger sizes are commonly used when a larger volume of sample material is required (see Attachment B).

Test Pit and Trench - Open, shallow excavations, typically rectangular (if a test pit) or longitudinal (if a trench), excavated to determine shallow subsurface conditions for engineering, geological, and soil chemistry exploration and/or sampling purposes. These pits are excavated manually or by machine (e.g., backhoe, clamshell, trencher, excavator, or bulldozer).

Subject SOIL SAMPLING	Number SA-1.3	Page 3 of 31
	Revision 9	Effective Date 04/07/2008

Thin-Walled Tube Sampler - A thin-walled metal tube (also called a Shelby tube) used to recover relatively undisturbed soil samples. These tubes are available in various sizes, ranging from 2 to 5 inches outside diameter (OD) and from 18 to 54 inches in length.

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager - The Project Manager is responsible for determining the sampling objectives, selecting proposed sampling locations, and selecting field procedures used in the collection of soil samples. Additionally, in consultation with other project personnel (geologist, hydrogeologist, etc.), the Project Manager establishes the need for test pits or trenches and determines their approximate locations and dimensions.

Site Safety Officer (SSO) - The SSO (or a qualified designee) is responsible for providing the technical support necessary to implement the project Health and Safety Plan. This will include (but not be limited to) performing air quality monitoring during sampling, boring, and excavation activities and to ensure that workers and offsite (downwind) individuals are not exposed to hazardous levels of airborne contaminants. The SSO/designee may also be required to advise the FOL on other safety-related matters regarding boring, excavation, and sampling, such as mitigative measures to address potential hazards from unstable trench walls, puncturing of drums or other hazardous objects, etc.

Field Operations Leader (FOL) - This individual is primarily responsible for the execution of the planning document containing the Sampling and Analysis Plan (SAP). This is accomplished through management of a field sampling team for the proper acquisition of samples. He or she is responsible for the supervision of onsite analyses; ensuring proper instrument calibration, care, and maintenance; sample collection and handling; the completion and accuracy of all field documentation; and making sure that custody of all samples obtained is maintained according to proper procedures. When appropriate and as directed by the FOL, such responsibilities may be performed by other qualified personnel (e.g., field technicians) where credentials and time permit. The FOL is responsible for finalizing the locations for collection of surface, near-surface, and subsurface (hand and machine borings, test pits/trenches) soil samples. He/she is ultimately responsible for the sampling and backfilling of boreholes, test pits, and trenches and for adherence to Occupational Safety and Health Administration (OSHA) regulations during these operations through self acquisition or through the management of a field team of samplers.

Project Geologist/Sampler - The project geologist/sampler is responsible for the proper acquisition of samples in accordance with this SOP and/or other project-specific documents. In addition, this individual is responsible for the completion of all required paperwork (e.g., sample log sheets, field notebook, boring logs, test pit logs, container labels, custody seals, and chain-of-custody forms) associated with the collection of those samples.

Competent Person - A Competent Person, as defined in 29 CFR 1929.650 of Subpart P - Excavations, means one who is capable of identifying existing and predictable hazards in the surroundings, or working conditions that are unsanitary, hazardous, or dangerous to employees, and who has authorization to take prompt corrective measures to eliminate them.

General personnel qualifications for groundwater sample collection and onsite water quality testing include the following:

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.
- Capability of performing field work under the expected physical and environmental (i.e., weather) conditions.

Subject SOIL SAMPLING	Number SA-1.3	Page 4 of 31
	Revision 9	Effective Date 04/07/2008

- Familiarity with appropriate procedures for sample documentation, handling, packaging, and shipping.

5.0 HEALTH AND SAFETY

Health and safety precautions are identified for individual sample collection procedures throughout this SOP. In addition to those precautions, the following general hazards may be incurred during sampling activities:

- Knee injuries from kneeling on hard or uneven surfaces
- Slips, trips, and falls
- Cuts and lacerations
- Traffic hazards associated with sampling in parking areas, along roadways and highways.

Methods of avoiding these hazards are provided below.

Knee injuries – If kneeling is required during soil sampling, this could result in knee injuries from stones/foreign objects and general damage due to stress on the joints. To minimize this hazard:

- Clear any foreign objects from the work area.
- Wear hard-sided knee pads.
- Stretch ligaments, tendons and muscles before, during and after. Take breaks as frequently as necessary.
- Report pre-existing conditions to the SSO if you feel this activity will aggravate an existing condition.

Slips, Trips, and Falls – These hazards exist while traversing varying terrains carrying equipment to sample locations. To minimize these hazards:

- Pre-survey sampling locations. Eliminate, barricade, or otherwise mark physical hazards leading to the locations.
- Carry small loads that do not restrict the field of vision.
- Travel the safest and clearest route (not necessarily the shortest).

Cuts and Lacerations - To prevent cuts and lacerations associated with soil sampling, the following provisions are required:

- Always cut away from yourself and others when cutting tubing or rope. This will prevent injury to yourself and others if the knife slips.
- Do not place items to be cut in your hand or on your knee.
- Change blades as necessary to maintain a sharp cutting edge. Many accidents result from struggling with dull cutting attachments.

Subject SOIL SAMPLING	Number SA-1.3	Page 5 of 31
	Revision 9	Effective Date 04/07/2008

- Whenever practical, wear cut-resistant gloves (e.g., leather or heavy cotton work gloves) at least on the hand not using the knife.
- Keep cutting surfaces clean and smooth.
- Secure items to be cut – do not hold them against the opposing hand, a leg, or other body part.
- When transporting glassware, keep it in a hard-sided container such as a cooler so that if there is a fall, you will be less likely to get cut by broken glass.
- DO NOT throw broken sample jars or glass ampoules into garbage bags. Place broken glass and glass ampoules in hard-sided containers such as a cardboard box or directly into a dumpster. DO NOT reach into garbage bags to retrieve any item accidentally thrown away. Empty the contents onto a flat surface to avoid punctures and lacerations from reaching where you cannot see.

Vehicular and Foot Traffic Hazards – When sampling along the roadway or near traffic patterns, follow the following precautions:

- Motorists may be distracted by onsite activities – ASSUME THEY DO NOT SEE YOU OR MEMBERS OF YOUR FIELD CREW.
- DO NOT place obstructions (such as vehicles) along the sides of the road that may cause site personnel to move into the flow of traffic to avoid your activities or equipment or that will create a blind spot.
- **Provide a required free space of travel.** Maintain at least 6 feet of space between you and moving traffic. Where this is not possible, use flaggers and/or signs to warn oncoming traffic of activities near or within the travel lanes.
- **Face Traffic.** Whenever feasible, if you must move within the 6 feet of the required free space or into traffic, attempt to face moving traffic at all times. Always leave yourself an escape route.
- Wear high-visibility vests to increase visual recognition by motorists.
- Do not rely on the vehicle operator's visibility, judgment, or ability. Make eye contact with the driver. Carefully and deliberately use hand signals so they will not startle or confuse motorists or be mistaken for a flagger's direction before moving into traffic.
- Your movements may startle a motorist and cause an accident, so move deliberately. Do not make sudden movements that might confuse a motorist.

6.0 PROCEDURES

The following procedures address surface and subsurface sampling.

CAUTION

Each situation must be evaluated individually to determine the applicability and necessity for obtaining a utility clearance ticket/dig permit. Common sense dictates, prior to digging or boring with power equipment, no matter what the depth, or digging by hand in a manner that could damage unprotected underground utilities, that a dig permit is required. See SOP HS-1.0, Utility Locating and Excavation Clearance, for additional

Subject SOIL SAMPLING	Number SA-1.3	Page 6 of 31
	Revision 9	Effective Date 04/07/2008

clarification. If you do not know or are unsure as to whether a ticket is necessary – **Get the Ticket.**

6.1 Overview

Soil sampling is an important adjunct to groundwater monitoring. Sampling of the soil horizons above the groundwater table can detect contaminants before they migrate to the water table, and can establish the amount of contamination absorbed or adsorbed on aquifer solids that have the potential of contributing to groundwater contamination.

Soil types can vary considerably on a hazardous waste site. These variations, along with vegetation, can affect the rate of contaminant migration through the soil. It is important, therefore, that a detailed record be maintained during sampling operations, particularly noting sampling locations, depths, and such characteristics as grain size, color, and odor. Subsurface conditions are often stable on a daily basis and may demonstrate only slight seasonal variation especially with respect to temperature, available oxygen and light penetration. Changes in any of these conditions can radically alter the rate of chemical reactions or the associated microbiological community, thus further altering specific site conditions. Certain vegetation species can create degradation products that can alter contaminant concentrations in soil. This is why vegetation types and extent of degradation of this foliage must be recorded. To prevent degradation, samples must be kept at their at-depth temperature or lower, protected from direct light, sealed tightly in approved glass containers, and be analyzed as soon as possible after collection. In addition, to the extent possible, vegetation should be removed from the sample.

The physical properties of the soil, its grain size, cohesiveness, associated moisture, and such factors as depth to bedrock and water table, will limit the depth from which samples can be collected and the method required to collect them. It is the intent of this document to present the most commonly employed soil sampling methods used at hazardous waste sites.

6.2 Soil Sample Collection

6.2.1 Procedure for Preserving and Collecting Soil Samples for Volatile Organic Compound Analysis

Samples collected using traditional methods such as collection in a jar with no preservation have been known to yield non-representative samples due to loss of volatile organic compounds (VOCs). To prevent such losses, preservation of samples with methanol or sodium bisulfate may be used to minimize volatilization and biodegradation. This preservation may be performed either in the field or laboratory, depending on the sampling methodology employed. Because of the large number of sampling methods and associated equipment required, careful coordination between field and laboratory personnel is needed.

Soil samples to be preserved by the laboratory are currently being collected using Method SW-846, 5035. For samples preserved in the field, laboratories are currently performing low-level analyses (sodium bisulfate preservation) and high- to medium-level analyses (methanol preservation) depending on the needs of the end user.

The following procedures outline the necessary steps for collecting soil samples to be preserved at the laboratory, and for collecting soil samples to be preserved in the field with methanol or sodium bisulfate.

Subject SOIL SAMPLING	Number SA-1.3	Page 7 of 31
	Revision 9	Effective Date 04/07/2008

6.2.1.1 Soil Samples to be Preserved at the Laboratory

Soil samples collected for volatile organic analysis that are to be preserved at the laboratory shall be obtained using a hermetically sealed sample vial such as an EnCore™ sampler. Each sample shall be obtained using a reusable sampling handle (T-handle) that can be provided with the EnCore™ sampler when requested and purchased. Collect the sample in the following manner for each EnCore™ sampler:

1. Scene Safety - Evaluate the area where sampling will occur. Ensure that the area is safe from physical, chemical, and natural hazards. Clear or barricade those hazards that have been identified.
2. Wear the appropriate personal protective equipment (PPE). This will include, at a minimum, safety glasses and nitrile surgeon's gloves. If you must kneel on the ground or place equipment on the surface being sampled, cover the ground surface with plastic to minimize surface contamination of your equipment and clothing. Wear knee pads to protect your knees from kneeling on hard or uneven surfaces.
3. Load the Encore™ sampler into the T-handle with the plunger fully depressed.
4. Expose the area to be sampled using a hand trowel or similar device to remove surface debris.
5. Press the T-handle against the freshly exposed soil surface, forcing soil into the sampler. The plunger will be forced upward as the cavity fills with soil.
6. When the sampler is full, rotate the plunger and lock it into place. If the plunger does not lock, the sampler is not full. This method ensures there is no headspace. Soft soil may require several plunges or forcing soil against a hard surface such as a sample trowel to ensure that headspace is eliminated.
7. Use a paper towel to remove soil from the side of the sampler so a tight seal can be made between the sample cap and the rubber O-ring.
8. With soil slightly piled above the rim of the sampler, force the cap on until the catches hook the side of the sampler.
9. Remove any surface soil from the outside of the sampler and place in the foil bag provided with the sampler. Good work hygiene practices and diligent decontamination procedures prevents the spread of contamination even on the outside of the containers.
10. Label the bag with appropriate information in accordance with SOP SA-6.3.
11. Place the full sampler inside a lined cooler with ice and cool to $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$. Make sure any required trip blanks and temperature blanks are also in the cooler. Secure custody of the cooler in accordance with SOP SA-6.3.
12. Typically, collect three Encore™ samplers at each location. Consult the SAP or laboratory to determine the required number of Encore™ samplers to be collected.
13. The T-handle shall be decontaminated before moving to the next interval or location using a soap and water wash and rinse, and where applicable, the selected solvent as defined in the project planning documents.

Using this type of sampling device eliminates the need for field preservation and the shipping restrictions associated with preservatives. A complete set of instructions is included with each Encore™ sampler.

Subject SOIL SAMPLING	Number SA-1.3	Page 8 of 31
	Revision 9	Effective Date 04/07/2008

After the Encore™ samples are collected, they should be placed on ice immediately and delivered to the laboratory within 48 hours (following the chain-of-custody and documentation procedures outlined in SOP SA-6.1). Samples must be preserved by the laboratory within 48 hours of sample collection.

6.2.1.2 Soil Samples to be Preserved in the Field

Soil samples preserved in the field may be prepared for analyses using both the low-level (sodium bisulfate preservation) and high- to medium-level (methanol preservation) methods.

Safety Reminder

When using chemicals in the field to preserve samples, the FOL and/or SSO must ensure that Materials Safety Data Sheets (MSDSs) have been provided with the chemicals to be used. They also must ensure that these chemicals have been added to the Chemical Inventory List contained within Section 5.0, Hazard Communication, of your Health and Safety Guidance Manual (HSGM). Lastly, but most importantly, the FOL and/or SSO must review the hazards with personnel using these chemicals and ensure that provisions are available for recommended PPE and emergency measures (e.g., eyewash, etc.).

Methanol Preservation (High to Medium Level):

Bottles may be pre-spiked with methanol in the laboratory or prepared in the field. Soil samples to be preserved in the field with methanol shall utilize 40 to 60 mL glass vials with septum-lined lids. Each sample bottle shall be filled with 25 mL of demonstrated analyte-free purge-and-trap grade methanol. The preferred method for adding methanol to the sample bottle is by removing the lid and using a pipette or scaled syringe to add the methanol directly to the bottle.

CAUTION

NEVER attempt to pipette by mouth

In situations where personnel are required to spike the septum using a hypodermic needle, the following provisions for handling sharps must be in place:

- Training of personnel regarding methods for handling of sharps
- Hard-sided containers for the disposal of sharps
- Provisions for treatment in cases where persons have received a puncture wound

Soil shall be collected with the use of a decontaminated (or disposable), small-diameter coring device such as a disposable tube/plunger-type syringe with the tip cut off. The outside diameter of the coring device must be smaller than the inside diameter of the sample bottle neck.

A small electronic balance or manual scale will be necessary for measuring the volume of soil to be added to the methanol-preserved sample bottle. Calibration of the scale shall be performed prior to use and intermittently throughout the day according to the manufacturer's requirements.

The sample should be collected as follows:

1. Weigh the unused syringe and plunger to the nearest 0.01 gram.
2. Pull the plunger back and insert the syringe into the soil to be sampled.

Subject SOIL SAMPLING	Number SA-1.3	Page 9 of 31
	Revision 9	Effective Date 04/07/2008

3. Collect 8 to 12 grams of soil by pushing the syringe barrel into the soil.
4. Weigh the sample and adjust until obtaining the required amount of sample.
5. Record the sample weight to the nearest 0.01 gram in the field logbook and/or on the sample log sheet.
6. Extrude the weighed soil sample into the methanol-preserved sample bottle taking care not to contact the sample container with the syringe.
7. If dirty, wipe soil particles from the threads of the bottle and cap. Cap the bottle tightly.
8. After capping the bottle, swirl the sample (do not shake) in the methanol and break up the soil such that all of the soil is covered with methanol.
9. Place the sample on ice immediately and prepare for shipment to the laboratory as described in SOP SA-6.1.

Sodium Bisulfate Preservation (Low Level):

CAUTION

Care should be taken when adding the soil to the sodium bisulfate solution. A chemical reaction of soil containing carbonates (limestone) may cause the sample to effervesce or the vial to possibly explode. To avoid this hazard or hazards of this type, a small sample aliquot should be subjected to the sample preservative. If it effervesces in an open air environment, utilize an alternative method such as Encore™ or 2-ounce jar.

Bottles may be prepared in the laboratory or in the field with sodium bisulfate solution. Samples to be preserved in the field using the sodium bisulfate method are to be prepared and collected as follows:

1. Add 1 gram of sodium bisulfate to 5 mL of laboratory-grade deionized water in a 40 to 60 mL glass vial with septum-lined lid.
2. Collect the soil sample and record the sample weight to the nearest 0.01 gram in the field logbook or on the sample log sheet as described for methanol preservation
3. Add the weighed sample to the sample vial.
4. Collect duplicate samples using the methanol preservation method on a one-for-one sample basis because it is necessary for the laboratory to perform both low-level and medium-level analyses.
5. Place the samples on ice immediately and prepare for shipment to the laboratory as described in SOP SA-6.1.

NOTE

If lower detection limits are necessary, an option to field preserving with sodium bisulfate may be to collect EnCore™ samplers at a given sample location. Consult the planning documents to determine whether this is required. If it is, collect samples in accordance with the Encore™ sampling procedure above and then send all samplers to the laboratory to perform the required preservation and analyses.

Subject SOIL SAMPLING	Number SA-1.3	Page 10 of 31
	Revision 9	Effective Date 04/07/2008

6.2.2 Procedure for Collecting Soil Samples for Non-Volatile Analyses

Samples collected for non-volatile analyses may be collected as either grab or composite samples as follows:

1. With a stainless steel trowel or other approved tool, transfer a portion of soil to be sampled to a stainless steel bowl or disposable inert plastic tray.
2. Remove roots, vegetation, sticks, and stones larger than the size of a green pea.
3. Thoroughly mix the soil in the bowl or tray to obtain as uniform a texture and color as practicable. The soil type, moisture content, amount of vegetation, and other factors may affect the amount of time required to obtain a properly mixed sample. In some cases, it may be impossible to obtain a uniform sample appearance. Use the field logbook to describe any significant difficulties encountered in obtaining a uniform mixture.
4. Transfer the mixed soil to the appropriate sample containers and close the containers.
5. Label the sample containers in accordance with SOP SA-6.3.
6. Place the containers in a cooler of ice as soon after collection as possible.
7. Prepare the sample shipment and ship the samples in accordance with SOP SA-6.1.

NOTE

Cooling may not be required for some samples depending on the scheduled analyses. Consult the planning documents if in doubt regarding correct sample preservation conditions. When in doubt – Cool to 4 °C.

NOTE

Head space is permitted in soil sample containers for non-volatile analyses to allow for sample expansion.

6.2.3 Procedure for Collecting Undisturbed Soil Samples

NOTE

Use of thin-walled undisturbed tube samplers is restricted by the consistency of the soil to be sampled. Often, very loose and/or wet samples cannot be retrieved by the samplers, and soil with a consistency in excess of very stiff cannot be penetrated by the sampler. Devices such as Dennison or Pitcher core samplers can be used to obtain undisturbed samples of stiff soil. Using these devices normally increases sampling costs, and therefore their use should be weighed against the need for acquiring an undisturbed sample. These devices are not discussed in this SOP because they are not commonly used.

When it is necessary to acquire undisturbed samples of soil for purposes of engineering parameter analysis (e.g., permeability), a thin-walled, seamless tube sampler (Shelby tube) shall be employed using the following collection procedure:

1. In preparation for sampling utilizing a drill rig, field personnel must complete the following activities:

Subject SOIL SAMPLING	Number SA-1.3	Page 11 of 31
	Revision 9	Effective Date 04/07/2008

- Ensure that all subsurface drilling activities are preceded by a utility clearance for the area to be investigated. This includes activities described in SOP HS-1.0, Utility Location and Excavation Clearance, as well as any location-specific procedures that may apply.

REMEMBER

If you are digging near a marked utility (within the diameter of an underground utility that has been marked plus 18 inches), you must first locate the utility through vacuum extraction or hand digging to ensure that your activities will not damage the utility.

- Complete an Equipment Inspection Checklist for the drill rig or direct-push technology (DPT) rig. This checklist will be provided in the HASP.
 - Review the Safe Work Permit prior to conducting the activity.
 - Review the activity to be conducted.
2. Remove all surface debris (e.g., vegetation, roots, twigs, etc.) from the specific sampling location and drill and/or clean out the borehole to the desired sampling depth. Be careful to minimize potential disturbance of the material to be sampled. In saturated material, withdraw the drill bit slowly to prevent loosening of the soil around the borehole and to maintain the water level in the hole at or above groundwater level.

CAUTION

The use of bottom-discharge bits or jetting through an open-tube sampler to clean out the borehole shall not be allowed. Only the use of side-discharge bits is permitted.

3. Determine whether a stationary piston-type sampler is required to limit sample disturbance and aid in retaining the sample. Either the hydraulically operated or control rod activated-type of stationary piston sampler may be used.
4. Prior to inserting the tube sampler into the borehole, check to ensure that the sampler head contains a check valve. The check valve is necessary to keep water in the rods from pushing the sample out the tube sampler during sample withdrawal. In addition, the check valve maintains a positive suction within the tube to help retain the sample.
5. A stainless steel tube sampler is typically used to minimize chemical reaction between the sample and the sampling tube.
6. With the sampling tube resting on the bottom of the hole and the water level in the boring at groundwater level or above, push the tube into the soil with a continuous and rapid motion, without impacting or twisting. If the soil is too hard to penetrate by pushing alone, careful hammering may be used by minimizing drop distance (tapping) of the hammer. Before pulling the tube, turn it at least one revolution to shear the sample off at the bottom. In no case shall the tube be pushed farther than the length provided for the soil sample. Allow about 3 inches in the tube for cuttings and sludge.
7. Upon removal of the sampling tube from the hole, measure the length of sample in the tube and also the length penetrated.
8. Remove disturbed material in the upper end of the tube and measure the length of sample again.

Subject SOIL SAMPLING	Number SA-1.3	Page 12 of 31
	Revision 9	Effective Date 04/07/2008

9. After removing at least 1 inch of soil from the lower end, place enough packing material (clean inert material such as paper or cloth) tightly in each end of the Shelby tube and then pour melted wax into each end to make at least a ½-inch wax plug and then add more packing material to fill the voids at both ends.
10. Place plastic caps on the ends, tape the caps in place, and dip the ends in wax to prevent loss of soil.
11. Affix label(s) to the tube as required and record sample number, depth, penetration, and recovery length on the label.
12. Mark the "up" direction on the side and upper end of the tube with indelible ink.
13. Complete a chain-of-custody form (see SOP SA-6.3) and other required forms (including Attachment A of this SOP).
14. Ship samples protected with suitable resilient packing material to reduce shock, vibration, and disturbance.

CAUTION

To preserve sample integrity do not allow tubes to freeze, and store the samples vertically with the same orientation they had in the ground, (i.e., top of sample is up) in a cool place out of the sun at all times.

CAUTION

A primary concern in the preparation of the wax plugs is the potential for the heat source and melted wax to cause a fire and/or burns. Follow the directions below to prevent injury or fire.

Electrical Heating

Using hot plates to melt the wax is acceptable. In an outdoor setting, make sure a Ground Fault Circuit Interrupter (GFCI) is employed within the electrical circuit. If a portable generator is used, ensure that the generator is an adequate distance from the sampling operation (at least 50 feet). Ensure that the extension cord is rated for the intended load and for outdoor use and is free from recognizable damage. Ensure flammable preservatives are not employed or stored near the hot plate. Although a Hot Work Permit is not required, scene safety evaluation by site personnel of the above elements is. As always, if a fire potential exists, the provisions for extinguishing must be immediately accessible as well as any provisions for first aid measures.

Open Flame

If an open flame is used, the following provisions are necessary:

- Complete a Hot Work Permit and any local permit required for elevated temperature applications. The Hot Work Permit, provided in your HASP, will aid the FOL and/or the SSO in ensuring that fire protection provisions (extinguishers, fire watches, etc.) are in place as well as ensuring that local requirements have been addressed.

Subject SOIL SAMPLING	Number SA-1.3	Page 13 of 31
	Revision 9	Effective Date 04/07/2008

- Ensure that water is available to address any wax splashes or contact. If possible, immerse the contacted area. Where this is not possible, run water over the area and apply cold compresses. The need for medical attention or first aid shall be determined on site under the direction of the SSO.

6.3 Surface Soil Sampling

The simplest, most direct method of collecting surface soil samples for subsequent analysis is by use of a stainless steel shovel, hand auger, soil corer, or stainless steel or disposable plastic trowel.

NOTE

Multiple depth intervals are used to describe surface soil. Sometimes surface soil is defined as soil from 0 to 2 inches below ground surface (bgs), and sometimes it is defined as soil from other depths such as 0 to 2 feet bgs. Ensure that the definition of surface soil depth is clear before collecting surface soil samples.

For the purposes of instruction, the terms "surface soil" and "near-surface soil" are used in this SOP as follows:

- Surface soil - 0 to 6 inches bgs
- Near-surface soil - 6 to 18 inches bgs

If these intervals are defined differently in the planning documents, substitute the appropriate depth ranges.

In general, the following equipment is necessary for obtaining surface soil samples:

- Stainless steel or pre-cleaned disposable trowel.
- Stainless steel hand auger, soil corer, or shovel.
- Real-time air monitoring instrument (e.g., PID, FID) as directed in project planning document.
- Required PPE.
 - Nitrile surgeon's or latex gloves may be used, layered as necessary.
 - Safety glasses
 - Other – Items identified on the Safe Work Permit may be required based on location-specific requirements such as hearing protection, steel-toed work boots, and a hard hat when working near a drill rig. These provisions will be listed in the HASP or directed by the FOL and/or SSO.

Safety Reminder

The use of latex products may elicit an allergic reaction in some people. Should this occur, remove the latex gloves, treat for an allergic reaction, and seek medical attention as necessary.

- Required paperwork (see SOP SA-6.3 and Attachment A of this SOP)

Subject SOIL SAMPLING	Number SA-1.3	Page 14 of 31
	Revision 9	Effective Date 04/07/2008

- Required decontamination equipment
- Required sample container(s)
- Wooden stakes or pin flags
- Sealable polyethylene bags (e.g., Ziploc® baggies)
- Heavy duty cooler
- Ice
- Chain-of-custody records and custody seals

When acquiring surface soil samples, use the following procedure:

1. Place padding or use knee pads when kneeling near the sample location. If necessary, place plastic sheeting to provide a clean surface for sample equipment to avoid possible cross- contamination.
2. Carefully remove vegetation, roots, twigs, litter, etc. to expose an adequate soil surface area to accommodate sample volume requirements.
3. Using a precleaned syringe or EnCore™ samplers, follow the procedure in Section 6.2.1 for collecting surface soil samples for volatile analysis. Surface soil samples for volatile organic analysis should be collected deeper than 6 inches bgs because shallower material has usually lost most of the volatiles through evaporation. Ensure that the appropriate surface soil depth is being analyzed in accordance with the planning document.
4. Using decontaminated sampling tools, thoroughly mix in place a sufficient amount of soil to fill the remaining sample containers. See Section 6.5 of this procedure for hand auger instruction, as needed.
5. Transfer the sample into those containers utilizing a stainless steel trowel.
6. Cap and securely tighten all sample containers.
7. Affix a sample label to each container. Be sure to fill out each label carefully and clearly, addressing all the categories described in SOP SA-6.3.
8. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.
9. Site restoration – Whenever removing sample materials, always restore the surface. It is our intent to leave the area better than we found it. Do NOT create trip hazards in areas when pedestrian traffic may exist.

6.4 Near-Surface Soil Sampling

Collection of samples from near the surface (depth of 6 to 18 inches) can be accomplished with tools such as shovels, hand auger, soil corers, and stainless steel or pre-cleaned disposable trowels and the equipment listed under Section 6.5 of this procedure.

To obtain near-surface soil samples, the following protocol shall be used:

Subject SOIL SAMPLING	Number SA-1.3	Page 15 of 31
	Revision 9	Effective Date 04/07/2008

1. With a clean shovel, make a series of vertical cuts in the soil to the depth required to form a square approximately 1 foot by 1 foot.
2. Lever out the formed plug and scrape the bottom of the freshly dug hole with a decontaminated stainless steel or pre-cleaned disposable trowel to remove any loose soil.
3. Follow steps 1 through 9 of Section 6.3.

6.5 Subsurface Soil Sampling With a Hand Auger

A hand augering system generally consists of a variety of stainless steel bucket bits (approximately 6.5 inches long and 2, 2.75, 3.25, and 4 inches in diameter), series of extension rods (available in 2-, 3-, 4- and 5-inch lengths), and a T-handle connected to extension rods and to the auger bucket. A larger-diameter bucket bit is commonly used to bore a hole to the desired sampling depth and then it is withdrawn. The larger-diameter bit is then replaced with a smaller-diameter bit, lowered down the hole, and slowly turned into the soil to the completion depth (approximately 6 inches). The apparatus is then withdrawn and the soil sample collected.

The hand auger can be used in a wide variety of soil conditions. It can be used to sample soil either from the surface, or to depths in excess of 12 feet. However, the presence of subsurface rocks and landfill material and collapse of the borehole normally limit sampling depth.

To accomplish soil sampling using a hand augering system, the following equipment is required:

- Complete hand auger assembly (variety of bucket bit sizes)
- Stainless steel mixing bowls
- The equipment listed in Section 6.3
- Miscellaneous hand tools as required to assemble and disassemble the hand auger units

CAUTION

Potential hazards associated with hand augering include:

- Muscle strain and sprain due to over twisting and/or over compromising yourself.
- Equipment failure due to excessive stress on the T-handle or rods through twisting. Failure of any of these components will result in a sudden release and potential injury due to that failure.

As in all situations, any intrusive activities that could damage underground utilities shall be preceded by a Dig/Excavation permit/ticket. Call the Utility Locating service in the area or your Project Health and Safety Officer for more information. When in doubt – **Get the Ticket!**

To obtain soil samples using a hand auger, use the following procedure:

1. Wearing designated PPE, attach a properly decontaminated bucket bit to a clean extension rod and attach the T-handle to the extension rod.
2. Clear the area to be sampled of any surface debris (vegetation, twigs, rocks, litter, etc.).

Subject SOIL SAMPLING	Number SA-1.3	Page 16 of 31
	Revision 9	Effective Date 04/07/2008

3. Twist the bucket into the ground while pushing vertically downward on the auger. The cutting shoes fill the bucket as it is advanced into the ground.
4. As the auger bucket fills with soil, periodically remove any unneeded soil.
5. Add rod extensions as necessary to extend the reach of the auger. Also, note (in a field notebook, boring log, and/or on a standardized data sheet) any changes in the color, texture or odor of the soil as a function of depth. The project-specific planning document (SAP, HASP, etc.) describe requirements for scanning the soil with a real-time air monitoring instrument (e.g., PID, FID, etc.) and recording the measurements.
6. After reaching the desired depth (e.g., the top of the interval to be sampled), slowly and carefully withdraw the apparatus from the borehole to prevent or minimize movement of soil from shallower intervals to the bottom of the hole.
7. Remove the soiled bucket bit from the rod extension and replace it with another properly decontaminated bucket bit. The bucket bit used for sampling is to be smaller in diameter than the bucket bit employed to initiate the borehole.
8. Carefully lower the apparatus down the borehole. Care must be taken to avoid scraping the borehole sides.
9. Slowly turn the apparatus until the bucket bit is advanced approximately 6 inches.
10. Discard the top of the core (approximately 1 inch), which represents any loose material collected by the bucket bit before penetrating the sample material.
11. Using a precleaned syringe or EnCore™ samplers, follow the procedure in Section 6.2.1 for collecting a soil sample for volatile compound analysis directly from the bucket bit.
12. Utilizing a properly decontaminated stainless steel trowel or dedicated disposable trowel, remove the remaining sample material from the bucket bit and place into a properly decontaminated stainless steel mixing bowl.
13. Homogenize the sample material as thoroughly as practicable then fill the remaining sample containers. Refer to Section 6.2.2.
14. Follow steps 4 through 7 listed in Section 6.3.

6.5.1 Sampling Using Stainless Steel Soil Corers

A soil corer is a stainless steel tube equipped with a cutting shoe and sample window in the side. The soil corer is advanced into the soil by applying downward pressure (body weight). The soil is unloaded by then forcing a ram towards the cutting shoe, which results in the discharge of the soil core through a window in the sleeve.

Use, application, and sample protocol is the same as for hand augering provided above, but without necessarily rotating the corer while advancing it.

SAFETY REMINDER

Hand augering and soil corer sampling can be physically demanding based on the type of geology and subsurface encumbrances encountered. Soil coring has some added

Subject SOIL SAMPLING	Number SA-1.3	Page 17 of 31
	Revision 9	Effective Date 04/07/2008

hazards such the corer collapsing under your weight. To reduce the potential for muscle strain and damage, the following measures will be incorporated:

- Stretch and limber your muscles before heavy exertion. This hazard becomes more predominant in the early morning hours (prior to muscles becoming limber) and later in the day (as a result of fatigue).
- Job rotation – Share the duties so that repetitive actions do not result in fatigue and injury.
- Increase break frequencies as needed, especially as ambient conditions of heat and/or cold stress may dictate.
- Do not force the hand tools or use cheater pipes or similar devices to bypass an obstruction. Move to another location near the sampling point. Exerting additional forces on the sampling devices can result in damage and/or failure that could potentially injure someone in the immediate vicinity.
- Do not over compromise yourself when applying force to the soil corer or hand auger. If there is a sudden release, it could result in a fall or muscle injury due to strain.

6.6 Subsurface Soil Sampling with a Split-Barrel Sampler

A split-barrel (split-spoon) sampler consists of a heavy carbon steel or stainless steel sampling tube that can be split into two equal halves to reveal the soil sample (see Attachment B). A drive head is attached to the upper end of the tube and serves as a point of attachment for the drill rod. A removable tapered nosepiece/drive shoe attaches to the lower end of the tube and facilitates cutting. A basket-like sample retainer can be fitted to the lower end of the split tube to hold loose, dry soil samples in the tube when the sampler is removed from the drill hole. This split-barrel sampler is made to be attached to a drill rod and forced into the ground by means of a 140-pound or larger casing driver.

Safety Reminder

It is intended through the Equipment Inspection for Drill Rigs form provided in the HASP that the hammer and hemp rope, where applicable, associated with this activity will be inspected (no physical damage is obvious), properly attached to the hammer (suitable knots or sufficient mechanical devices), and is in overall good condition.

Split-barrel samplers are used to collect soil samples from a wide variety of soil types and from depths greater than those attainable with other soil sampling equipment.

The following equipment is used for obtaining split-barrel samples:

- Drilling equipment (provided by subcontractor).
- Split-barrel samplers (2-inch OD, 1-3/8-inch ID, either 20 inches or 26 inches long); Larger OD samplers are available if a larger volume of sample is needed.
- Drive weight assembly, 140-pound weight, driving head, and guide permitting free fall of 30 inches.
- Stainless steel mixing bowls.

Subject SOIL SAMPLING	Number SA-1.3	Page 18 of 31
	Revision 9	Effective Date 04/07/2008

- Equipment listed in Section 6.3.

The following steps shall be followed to obtain split-barrel samples (Steps 1 through 4 are typically performed by the drilling subcontractor):

1. Attach the split-barrel sampler to the sampling rods.
2. Lower the sampler into the borehole inside the hollow stem auger bits.
3. Advance the split-barrel sampler by hammering the length (typically 18 or 24 inches) of the split-barrel sampler into the soil using 140-pound or larger hammer.
4. When the desired depth is achieved, extract the drill rods and sampler from the augers and/or borehole.
5. Detach the sampler from the drill rods.
6. Place the sampler securely in a vise so it can be opened using pipe wrenches.

CAUTION

Pipe wrenches are used to separate the split spoon into several components. The driller's helper should not apply excessive force through the use of cheater pipes or push or pull in the direction where, if the wrench slips, hands or fingers will be trapped against an immovable object.

7. Remove the drive head and nosepiece with the wrenches, and open the sampler to reveal the soil sample.
8. Immediately scan the sample core with a real-time air monitoring instrument (e.g., FID, PID, etc.) (as project-specific planning documents dictate). Carefully separate (or cut) the soil core, with a decontaminated stainless steel knife or trowel, at about 6-inch intervals while scanning the center of the core for elevated readings. Also scan stained soil, soil lenses, and anomalies (if present), and record readings.
9. If elevated vapor readings were observed, collect the sample scheduled for volatile analysis from the center of the core where elevated readings occurred. If no elevated readings were encountered, the sample material should be collected from the core's center (this area represents the least disturbed area with minimal atmospheric contact) (refer to Section 6.2.1).
10. Using the same trowel, remove remaining sample material from the split-barrel sampler (except for the small portion of disturbed soil usually found at the top of the core sample) and place the soil into a decontaminated stainless steel mixing bowl.
11. Homogenize the sample material as thoroughly as practicable then fill the remaining sample containers (refer to Section 6.2.2).
12. Follow steps 4 through 7 in Section 6.3.

6.7 Subsurface Soil Sampling Using Direct-Push Technology

Subsurface soil samples can be collected to depths of 40+ feet using DPT. DPT equipment,

Subject SOIL SAMPLING	Number SA-1.3	Page 19 of 31
	Revision 9	Effective Date 04/07/2008

responsibilities, and procedures are described in SOP SA-2.5.

6.8 Excavation and Sampling of Test Pits and Trenches

6.8.1 Applicability

This subsection presents routine test pit or trench excavation techniques and specialized techniques that are applicable under certain conditions.

CAUTION

During the excavation of trenches or pits at hazardous waste sites, several health and safety concerns arise from the method of excavation. No personnel shall enter any test pit or excavation over 4 feet deep except as a last resort, and then only under direct supervision of a Competent Person (as defined in 29 CFR 1929.650 of Subpart P - Excavations). Whenever possible, all required chemical and lithological samples should be collected using the excavator bucket or other remote sampling apparatus. If entrance is required, all test pits or excavations must be stabilized by bracing the pit sides using specifically designed wooden, steel, or aluminum support structures or through sloping and benching. Personnel entering the excavation may be exposed to toxic or explosive gases and oxygen-deficient environments; therefore, monitoring will be conducted by the Competent Person to determine if it is safe to enter. Any entry into a trench greater than 4 feet deep will constitute a Confined Space Entry and must be conducted in conformance with OSHA standard 29 CFR 1910.146. In all cases involving entry, substantial air monitoring, before entry, appropriate respiratory gear and protective clothing determination, and rescue provisions are mandatory. There must be at least three people present at the immediate site before entry by one of the field team members. This minimum number of people will increase based on the potential hazards or complexity of the work to be performed. The reader shall refer to OSHA regulations 29 CFR 1926.650, 29 CFR 1910.120, 29 CFR 1910.134, and 29 CFR 1910.146. High-hazard entries such as this will be supported by members of the Health Sciences Group professionally trained in these activities.

Excavations are generally not practical where a depth of more than about 15 to 20-feet is desired, and they are usually limited to a few feet below the water table. In some cases, a pumping system may be required to control water levels within the pit, providing that pumped water can be adequately stored or disposed. If soil data at depths greater than 15-feet are required, the data are usually obtained through test borings instead of test pits.

In addition, hazardous wastes may be brought to the surface by excavation equipment. This material, whether removed from the site or returned to the subsurface, must be properly handled according to any and all applicable federal, state, and local regulations.

6.8.2 Test Pit and Trench Excavation

Test pits or trench excavations are constructed with the intent that they will provide an open view of subsurface lithology and/or disposal conditions that a boring will not provide. These procedures describe the methods for excavating and logging test pits and trenches installed to determine subsurface soil and rock conditions. Test pit operations shall be logged and documented (see Attachment C).

Test pits and trenches may be excavated by hand or power equipment to permit detailed descriptions of the nature and contamination of the in-situ materials. The size of the excavation will depend primarily on the following:

Subject SOIL SAMPLING	Number SA-1.3	Page 20 of 31
	Revision 9	Effective Date 04/07/2008

- The purpose and extent of the exploration
- The space required for efficient excavation
- The chemicals of concern
- The economics and efficiency of available equipment

Test pits normally have a cross section that is 4 to 10 feet square; test trenches are usually 3 to 6 feet wide and may be extended for any length required to reveal conditions along a specific line. The following table provides guidelines for design consideration based on equipment efficiencies.

Equipment	Typical Widths, in Feet
Trenching machine	0.25 to 1.0
Backhoe/Track Hoe	2 to 6

The lateral limits of excavation of trenches and the position of test pits shall be carefully marked on area base maps. If precise positioning is required to indicate the location of highly hazardous materials, nearby utilities, or dangerous conditions, the limits of the excavation shall be surveyed. Also, if precise determination of the depth of buried materials is needed for design or environmental assessment purposes, the elevation of the ground surface at the test pit or trench location shall also be determined by survey. If the test pit/trench will not be surveyed immediately, it shall be backfilled and its position identified with stakes placed in the ground at the margin of the excavation for later surveying.

The construction of test pits and trenches shall be planned and designed in advance as much as possible. However, the following field conditions may necessitate revisions to the initial plans:

- Subsurface utilities
- Surface and subsurface encumbrances
- Vehicle and pedestrian traffic patterns
- Purpose for excavation (e.g., the excavation of potential ordnance items)

The final depth and construction method shall be collectively determined by the FOL and designated Competent Person. The actual layout of each test pit, temporary staging area, and spoils pile may further be predicated based on site conditions and wind direction at the time the test pit is excavated. Prior to excavation, the area may be surveyed by magnetometer or metal detector or other passive methods specified in SOP HS1.0, Utility Location and Excavation Clearance, to identify the presence of underground utilities or drums. Where possible, the excavator should be positioned upwind and preferably within an enclosed cab.

No personnel shall enter any test pit or excavation except as a last resort, and then only under direct supervision of a Competent Person. If entrance is required, OSHA requirements must be met (e.g., walls must be braced with wooden or steel braces, ladders must be placed for every 25 feet of lateral travel and extended 3 feet above ground surface). A temporary guard rail or vehicle stop must be placed along the surface of the hole before entry in situations where the excavation may be approached by traffic. Spoils will be stockpiled no closer than 2 feet from the sidewall of the excavation. The excavation equipment operator shall be careful not to undercut sidewalls and will, where necessary, bench back to

Subject SOIL SAMPLING	Number SA-1.3	Page 21 of 31
	Revision 9	Effective Date 04/07/2008

increase stability. The top cover, when considered clean, will be placed separately from the subsurface materials to permit clean cover. It is emphasized that the project data needs should be structured such that required samples can be collected without requiring entrance into the excavation. For example, samples of leachate, groundwater, or sidewall soil can be collected with telescoping poles or similar equipment.

Dewatering and watering may be required to ensure the stability of the side walls, to prevent the bottom of the pit from heaving, and to keep the excavation stable. This is an important consideration for excavations in cohesionless material below the groundwater table and for excavations left open greater than a day. Liquids removed as a result of dewatering operations must be handled as potentially contaminated materials. Procedures for the collection and disposal of such materials should be discussed in the site-specific project plans.

Where possible excavations and test pits shall be opened and closed within the same working day. Where this is not possible, the following engineering controls shall be put in place to control access:

- Trench covers/street plates
- Fences encompassing the entire excavation intended to control access
- Warning signs warning personnel of the hazards
- Amber flashing lights to demarcate boundaries of the excavation at night

Excavations left open will have emergency means to exit should someone accidentally enter.

6.8.3 Sampling in Test Pits and Trenches

6.8.3.1 General

Log test pits and trenches as they are excavated in accordance with the Test Pit Log presented in Attachment C. These records include plan and profile sketches of the test pit/trench showing materials encountered, their depth and distribution in the pit/trench, and sample locations. These records also include safety and sample screening information.

Entry of test pits by personnel is extremely dangerous, shall be avoided unless absolutely necessary, and can occur only after all applicable health and safety and OSHA requirements have been met as stated above. These provisions will be reiterated as appropriate in the project-specific HASP.

The final depth and type of samples obtained from each test pit will be determined at the time the test pit is excavated. Sufficient samples are usually obtained and analyzed to quantify contaminant distribution as a function of depth for each test pit. Additional samples of each waste phase and any fluids encountered in each test pit may also be collected.

In some cases, samples of soil may be extracted from the test pit for reasons other than waste sampling and chemical analysis, for instance, to obtain geotechnical information. Such information includes soil types, stratigraphy, strength, etc., and could therefore entail the collection of disturbed (grab or bulk) or relatively undisturbed (hand-carved or pushed/driven) samples that can be tested for geotechnical properties. The purposes of such explorations are very similar to those of shallow exploratory or test borings, but often test pits offer a faster, more cost-effective method of sampling than installing borings.

Subject SOIL SAMPLING	Number SA-1.3	Page 22 of 31
	Revision 9	Effective Date 04/07/2008

6.8.3.2 Sampling Equipment

The following equipment is needed for obtaining samples for chemical or geotechnical analysis from test pits and trenches:

- Backhoe or other excavating machinery.
- Shovels, picks, hand augers, and stainless steel trowels/disposable trowels.
- Sample container - bucket with locking lid for large samples; appropriate bottle ware for chemical or geotechnical analysis samples.
- Polyethylene bags for enclosing sample containers; buckets.
- Remote sampler consisting of 10-foot sections of steel conduit (1-inch-diameter), hose clamps, and right angle adapter for conduit (see Attachment D).

6.8.3.3 Sampling Methods

The methods discussed in this section refer to test pit sampling from grade level. If test pit entry is required, see Section 6.8.3.4.

- Excavate the trench or pit in several 0.5- to 1.0-foot depth increments. Where soil types support the use of a sand bar cutting plate, use of this device is recommended to avoid potentially snagging utilities with the excavator teeth. It is recommended that soil probes or similar devices be employed where buried items or utilities may be encountered. This permits the trench floor to be probed prior to the next cut.
- After each increment:
 - the operator shall wait while the sampler inspects the test pit from grade level
 - the sampler shall probe the next interval where this is considered necessary. Practical depth increments for lithological evaluations may range from 2 to 4 feet i or where lithological changes are noted.
- The backhoe operator, who will have the best view of the test pit, shall immediately cease digging if:
 - Any fluid phase, including groundwater seepage, is encountered in the test pit
 - Any drums, other potential waste containers, obstructions, or utility lines are encountered
 - Distinct changes of material being excavated are encountered

This action is necessary to permit proper sampling of the test pit and to prevent a breach of safety protocol. Depending on the conditions encountered, it may be required to excavate more slowly and carefully with the backhoe.

For obtaining test pit samples from grade level, the following procedure shall be followed:

- Use the backhoe to remove loose material from the excavation walls and floor to the greatest extent possible.

Subject SOIL SAMPLING	Number SA-1.3	Page 23 of 31
	Revision 9	Effective Date 04/07/2008

- Secure the walls of the pit, if necessary. (There is seldom any need to enter a pit or trench that would justify the expense of shoring the walls. All observations and samples should be taken from the ground surface.)
- Samples of the test pit material are to be obtained either directly from the backhoe bucket or from the material after it has been deposited on the ground, as follows:
 - a. The sampler or FOL shall direct the backhoe operator to remove material from the selected depth or location within the test pit/trench.
 - b. The backhoe operator shall bring the bucket over to a designated location on the sidewall a sufficient distance from the pit (at least 5 feet) to allow the sampler to work around the bucket.
 - c. After the bucket has been set on the ground, the backhoe operator shall either disengage the controls or shut the machine down.
 - d. When signaled by the operator that it is safe to do, the sampler will approach the bucket.
 - e. The soil shall be monitored with a photoionization or flame ionization detector (PID or FID) as directed in the project -specific planning documents.
 - f. The sampler shall collect the sample from the center of the bucket or pile in accordance with surface soil sampling procedures of Section 6.3 or 6.4, as applicable. Collecting samples from the center of a pile or bucket eliminates cross-contamination from the bucket or other depth intervals.
- If a composite sample is desired, several depths or locations within the pit/trench will be selected, and the bucket will be filled from each area. It is preferable to send individual sample bottles filled from each bucket to the laboratory for compositing under the more controlled laboratory conditions. However, if compositing in the field is required, each sample container shall be filled from materials that have been transferred into a mixing bucket and homogenized. Note that homogenization/compositing is not applicable for samples to be subjected to volatile organic analysis.

CAUTION

Care must be exercised when using the remote sampler described in the next step because of potential instability of trench walls. In situations where someone must move closer than 2 feet to the excavation edge, a board or platform should be used to displace the sampler's weight to minimize the chance of collapse of the excavation edge. Fall protection should also be employed when working near the edges or trenches greater than 6 feet deep. An immediate means to extract people who have fallen into the trench will be immediately available. These means may include ladders or rope anchor points.

- Using the remote sampler shown in Attachment D, samples can be taken at the desired depth from the sidewall or bottom of the pit as follows:
 - a. Scrape the face of the pit/trench using a long-handled shovel or hoe to remove the smeared zone that has contacted the backhoe bucket.
 - b. Collect the sample directly into the sample jar, by scraping with the jar edge, eliminating the need for sample handling equipment and minimizing the likelihood of cross-contamination.

Subject SOIL SAMPLING	Number SA-1.3	Page 24 of 31
	Revision 9	Effective Date 04/07/2008

c. Cap the sample jar, remove it from the remote sampler assembly, and package the sample for shipment in accordance with SOP SA-6.3.

- Complete documentation as described in SOP SA-6.3 and Attachment C of this SOP.

6.8.3.4 In-Pit Sampling

Under rare conditions, personnel may be required to enter the test pit/trench. This is necessary only when soil conditions preclude obtaining suitable samples from the backhoe bucket (e.g., excessive mixing of soil or wastes within the test pit/trench) or when samples from relatively small discrete zones within the test pit are required. This approach may also be necessary to sample any seepage occurring at discrete levels or zones in the test pit that are not accessible with remote samplers.

In general, personnel shall sample and log pits and trenches from the ground surface, except as provided for by the following criteria:

- There are no practical alternative means of obtaining such data.
- The SSO and Competent Person determine that such action can be accomplished without breaching site safety protocol. This determination will be based on actual monitoring of the pit/trench after it is dug (including, at a minimum, measurements of oxygen concentration, flammable gases, and toxic compounds, in that order). Action levels will be provided in project-specific planning documents.
- A company-designated Competent Person determines that the pit/trench is stable through soil classification evaluation/inspections or is made stable (by cutting/grading the sidewalls or using shoring) prior to entrance of any personnel. OSHA requirements shall be strictly observed.

If these conditions are satisfied, only one person may enter the pit/trench. On potentially hazardous waste sites, this individual shall be dressed in selected PPE as required by the conditions in the pit. He/she shall be affixed to a harness and lifeline and continuously monitored while in the pit.

A second and possible third individual shall be fully dressed in protective clothing including a self-contained breathing device and on standby during all pit entry operations to support self rescue or assisted self rescue. The individual entering the pit shall remain therein for as brief a period as practical, commensurate with performance of his/her work. After removing the smeared zone, samples shall be obtained with a decontaminated trowel or spoon.

6.8.3.5 Geotechnical Sampling

In addition to the equipment described in Section 6.8.3.2, the following equipment is needed for geotechnical sampling:

- Soil sampling equipment, similar to that used in shallow drilled boring (i.e., thin-walled tube samplers), that can be pushed or driven into the floor of the test pit.
- Suitable driving (e.g., sledge hammer) or pushing (e.g., backhoe bucket) equipment used to advance the sampler into the soil.
- Knives, spatulas, and other suitable devices for trimming hand-carved samples.
- Suitable containers (bags, jars, tubes, boxes, etc.), labels, wax, etc. for holding and safely transporting collected soil samples.

Subject SOIL SAMPLING	Number SA-1.3	Page 25 of 31
	Revision 9	Effective Date 04/07/2008

- Geotechnical equipment (pocket penetrometer, torvane, etc.) for field testing collected soil samples for classification and strength properties.

Disturbed grab or bulk geotechnical soil samples may be collected for most soil in the same manner as comparable soil samples for chemical analysis. These collected samples may be stored in jars or plastic-lined sacks (larger samples), which will preserve their moisture content. Smaller samples of this type are usually tested for their index properties to aid in soil identification and classification: larger bulk samples are usually required to perform compaction tests.

Relatively undisturbed samples are usually extracted in cohesive soil using thin-walled tube samplers, and such samples are then tested in a geotechnical laboratory for their strength, permeability, and/or compressibility. The techniques for extracting and preserving such samples are similar to those used in performing Shelby tube sampling in borings, except that the sampler is advanced by hand or backhoe, rather than by a drill rig. Also, the sampler may be extracted from the test pit by excavation around the tube when it is difficult to pull it out of the ground. If this excavation requires entry of the test pit, the requirements described in Section 6.8.3.4 shall be followed. The thin-walled tube sampler shall be pushed or driven vertically into the floor or steps excavated in the test pit at the desired sampling elevations. Extracting tube samples horizontally from the walls of the test pit is not appropriate because the sample will not have the correct orientation.

A sledge hammer or backhoe may be used to drive or push the tube into the ground. Place a piece of wood over the top of the sampler or sampling tube to prevent damage during driving/pushing of the sample. Pushing the sampler with a constant thrust is always preferable to driving it with repeated blows, thus minimizing disturbance to the sample. When using a sledge hammer, it is recommended that the sampler be stabilized using a rope/strap wrench or pipe wrench to remove the person's hands holding the sampler from the strike zone. If the sample cannot be extracted by rotating it at least two revolutions (to shear off the sample at the bottom), hook the sampler to the excavator or backhoe and extract. This means an alternative head will be used as a connection point or that multiple choke hitches will be applied to extract the sampler. If this fails and the excavator can dig deeper without potentially impacting subsurface utilities, excavate the sampler. If this fails or if the excavator cannot be used due to subsurface utilities, hand-excavate to remove the soil from around the sides of the sampler. If hand-excavation requires entry into the test pit, the requirements in Section 6.8.3.4 must be followed. Prepare the sample as described in Steps 9 through 13 in Section 6.2.3, and label, pack and transport the sample in the required manner, as described in SOPs SA-6.3 and SA-6.1.

6.8.4 Backfilling of Trenches and Test Pits

All test pits and excavations must be either backfilled, covered, or otherwise protected at the end of each day. No excavations shall remain open during non-working hours unless adequately covered or otherwise protected.

Before backfilling, the onsite crew may photograph, if required by the project-specific work plan, all significant features exposed by the test pit and trench and shall include in the photograph a scale to show dimensions. Photographs of test pits shall be marked to include site number, test pit number, depth, description of feature, and date of photograph. In addition, a geologic description of each photograph shall be entered in the site logbook. All photographs shall be indexed and maintained as part of the project file for future reference.

After inspection, backfill material shall be returned to the pit under the direction of the FOL. Backfill should be returned to the trench or test pit in 6-inch to 1-foot lifts and compacted with the bucket. Remote controlled tampers or rollers may be lowered into the trench and operated from top side. This procedure will continue to the grade surface. It is recommended that the trench be tracked or rolled in. During

Subject SOIL SAMPLING	Number SA-1.3	Page 26 of 31
	Revision 9	Effective Date 04/07/2008

excavation, clean soil from the top 2 feet may have been separated to be used to cover the last segments. Where these materials are not clean, it is recommended that clean fill be used for the top cover.

If a low-permeability layer is penetrated (resulting in groundwater flow from an upper contaminated flow zone into a lower uncontaminated flow zone), backfill material must represent original conditions or be impermeable. Backfill could consist of a soil-bentonite mix prepared in a proportion specified by the FOL (representing a permeability equal to or less than original conditions). Backfill can be covered by "clean" soil and graded to the original land contour. Revegetation of the disturbed area may also be required.

6.9 **Records**

The appropriate sample log sheet (see Attachment A of this SOP) must be completed by the site geologist/sampler for all samples collected. All soil sampling locations should be documented by tying in the location of two or more nearby permanent landmarks (building, telephone pole, fence, etc.) or obtaining GPS coordinates; and shall be noted on the appropriate sample log sheet, site map, or field notebook. Surveying may also be necessary, depending on the project requirements.

Test pit logs (see Attachment C of this SOP) shall contain a sketch of pit conditions. If the project-specific work plan requires photographs, at least one photograph with a scale for comparison shall be taken of each pit. Included in the photograph shall be a card showing the test pit number. Boreholes, test pits, and trenches shall be logged by the field geologist in accordance with SOP GH-1.5.

Other data to be recorded in the field logbook include the following:

- Name and location of job
- Date of boring and excavation
- Approximate surface elevation
- Total depth of boring and excavation
- Dimensions of pit
- Method of sample acquisition
- Type and size of samples
- Soil and rock descriptions
- Photographs if required
- Groundwater levels
- PID/FID/LEL/O₂ meter readings
- Other pertinent information, such as waste material encountered

In addition, site-specific documentation to be maintained by the SSO and/or Competent Person will be required including:

Subject SOIL SAMPLING	Number SA-1.3	Page 27 of 31
	Revision 9	Effective Date 04/07/2008

- Calibration logs
- Excavation inspection checklists
- Soil type classification

7.0 REFERENCES

American Society for Testing and Materials, 1987. ASTM Standards D1587-83 and D1586-84. ASTM Annual Book of Standards. ASTM. Philadelphia, Pennsylvania. Volume 4.08.

NUS Corporation, 1986. Hazardous Material Handling Training Manual.

NUS Corporation and CH2M Hill, August, 1987. Compendium of Field Operation Methods. Prepared for the U.S. EPA.

OSHA, Excavation, Trenching and Shoring 29 CFR 1926.650-653.

OSHA, Confined Space Entry 29 CFR 1910.146.

USEPA, November 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual.

Subject SOIL SAMPLING	Number SA-1.3	Page 28 of 31
	Revision 9	Effective Date 04/07/2008

**ATTACHMENT A
SOIL & SEDIMENT SAMPLE LOG SHEET**



Tetra Tech NUS, Inc.

SOIL & SEDIMENT SAMPLE LOG SHEET

Page ___ of ___

Project Site Name: _____	Sample ID No.: _____
Project No.: _____	Sample Location: _____
<input type="checkbox"/> Surface Soil	Sampled By: _____
<input type="checkbox"/> Subsurface Soil	C.O.C. No.: _____
<input type="checkbox"/> Sediment	Type of Sample:
<input type="checkbox"/> Other: _____	<input type="checkbox"/> Low Concentration
<input type="checkbox"/> QA Sample Type: _____	<input type="checkbox"/> High Concentration

GRAB SAMPLE DATA:			
Date:	Depth	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Time:			
Method:			
Monitor Reading (ppm):			

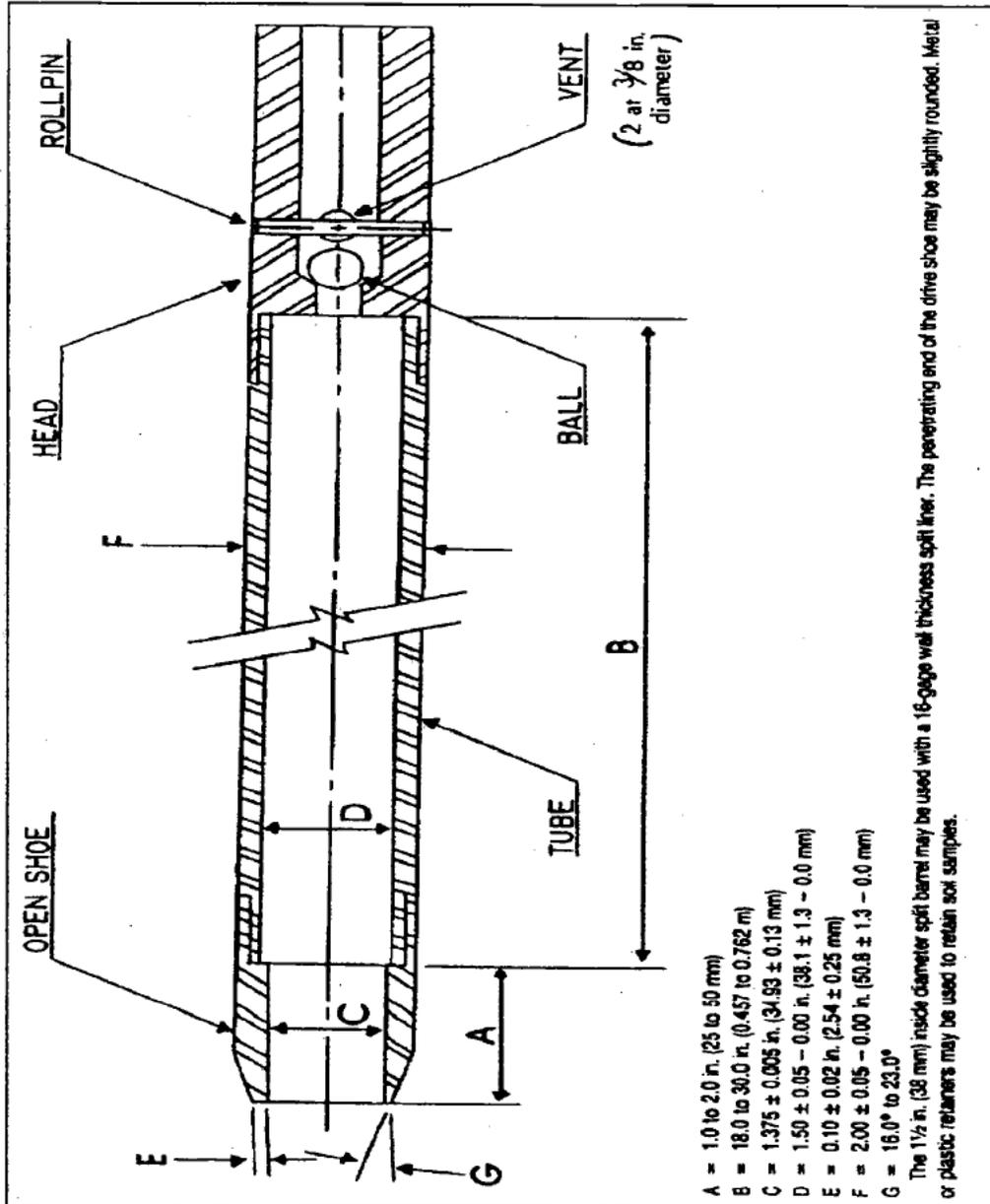
COMPOSITE SAMPLE DATA:				
Date:	Time	Depth	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Method:				
Monitor Readings (Range in ppm):				

SAMPLE COLLECTION INFORMATION:			
Analysis	Container Requirements	Collected	Other

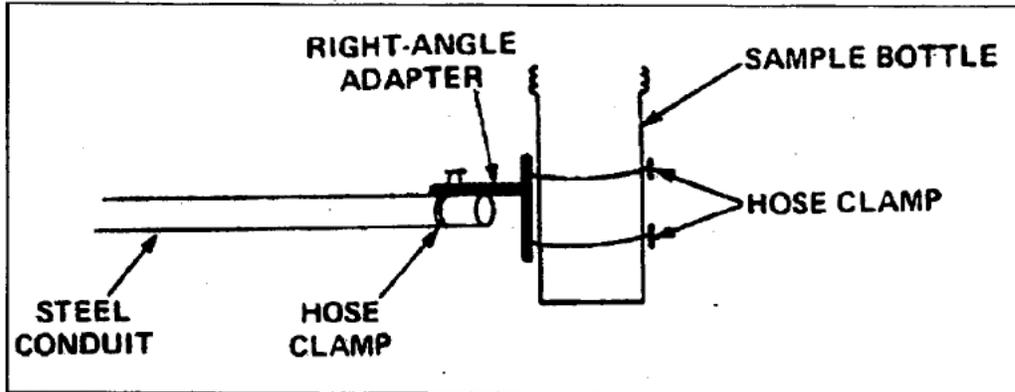
OBSERVATIONS / NOTES:	MAP:

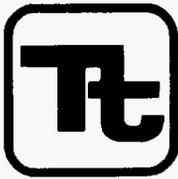
Circle if Applicable:	Signature(s):
MS/MSD Duplicate ID No.:	

ATTACHMENT B SPLIT-SPOON SAMPLER



ATTACHMENT D
REMOTE SAMPLE HOLDER FOR TEST PIT/TRENCH SAMPLING





TETRA TECH NUS, INC.

STANDARD OPERATING PROCEDURES

Number	SA-1.6	Page	1 of 21
Effective Date	09/03	Revision	1
Applicability	Tetra Tech NUS, Inc.		
Prepared	Earth Sciences Department		
Approved	D. Senovich <i>ds</i>		

Subject
NATURAL ATTENUATION PARAMETER COLLECTION

TABLE OF CONTENTS

<u>SECTION</u>	<u>PAGE</u>
1.0 PURPOSE.....	2
2.0 SCOPE.....	2
3.0 GLOSSARY	2
4.0 RESPONSIBILITIES.....	3
5.0 PROCEDURES.....	3
5.1 GENERAL.....	3
5.2 PLANNING FOR NATURAL ATTENUATION SAMPLING.....	4
5.3 SELECTION OF NATURAL ATTENUATION PARAMETERS.....	5
5.4 SELECTION OF NATURAL ATTENUATION ANALYTICAL METHODS AND PROCEDURES.....	6
5.5 PROCEDURES FOR SAMPLE COLLECTION.....	6
5.6 PROCEDURES FOR FIELD SAMPLE ANALYSIS.....	7
5.7 PROCEDURES FOR QUALITY ASSURANCE AND QUALITY CONTROL FIELD SAMPLE ANALYSIS.....	8
5.8 DOCUMENTATION PROCEDURES FOR FIELD SAMPLE ANALYSIS.....	9
5.9 WASTE HANDLING AND DISPOSAL.....	9
5.10 UNDERSTANDING FIELD SAMPLE ANALYTICAL RESULTS.....	9
6.0 REFERENCES.....	10

ATTACHMENTS

A	HYPOTHETICAL LONG-TERM MONITORING STRATEGY.....	11
B	REDOX POTENTIALS FOR VARIOUS ELECTRON ACCEPTORS.....	12
C	NATURAL ATTENUATION PARAMETERS FOR CHLORINATED VOLATILE ORGANIC COMPOUND PLUMES.....	13
D	NATURAL ATTENUATION PARAMETERS FOR PETROLEUM HYDROCARBON PLUMES.....	14
E	GEOCHEMICAL SAMPLING PARAMETERS - METHODS, EQUIPMENT, VOLUMES, CONTAINERS, PRESERVATION, HOLDING TIMES, AND DETECTION RANGES.....	15
F	FIELD ANALYTICAL LOG SHEET, GEOCHEMICAL PARAMETERS.....	19

Subject NATURAL ATTENUATION PARAMETER COLLECTION	Number SA-1.6	Page 2 of 21
	Revision 1	Effective Date 09/03

1.0 PURPOSE

The purpose of this document is to provide general reference information regarding natural attenuation parameter and methodology selection, sample collection, and a general understanding of the sample results.

2.0 SCOPE

This document provides information on selection of appropriate groundwater natural attenuation parameters, selection of sampling methods for these parameters, techniques for onsite field analysis of select parameters, and some basic understanding of the field sample results. Review of the information contained herein will facilitate planning of the field sampling effort by describing standard sampling practices and techniques. To a limited extent, it shall also facilitate the understanding and interpretation of the sampling results. It addresses field procedures for collection of data at sites with organic groundwater contaminants (e.g., chlorinated and petroleum hydrocarbons) to the extent practical. The focus of this document is on natural attenuation, not enhanced bioremediation.

The techniques described shall be followed whenever applicable, noting that site-specific conditions, project-specific objectives, local, state, and federal guidelines may be used as a basis for modification of the procedures noted herein. The intent of this document is to supplement the local, state, and federal guidance documents and manufacturer's analytical methods referenced in Section 6.0. It is not intended for this document to supersede this guidance or information. Please note that natural attenuation is a relatively dynamic science with ongoing research in the science and engineering community. It is important that data collectors and interpreters use the most recent regulatory guidance, which may be updated on a periodic basis from that noted in Section 6.

3.0 GLOSSARY

Aerobe: Bacteria that use oxygen as an electron acceptor.

Anaerobe: Organisms that can use electron acceptors other than molecular oxygen to support their metabolism.

Anoxic groundwater: Groundwater that contains oxygen in concentrations less than about 0.5 mg/L. This term is synonymous with the term anaerobic.

Anthropogenic: Man-made.

Cometabolism: The process in which a compound is fortuitously degraded by an enzyme or cofactor produced during microbial metabolism of another compound.

Daughter product: A compound that results directly from the biotic or abiotic degradation of another. For example, *cis*-1,2-dichloroethene (*cis*-1,2-DCE) is a common daughter product of trichloroethene (TCE).

Diffusion: The process whereby molecules move from a region of higher concentration to a region of lower concentration as a result of Brownian motion.

Dispersion: The tendency for a solute to spread from the path that it would be expected to follow under advective transport.

Electron acceptor: A compound capable of accepting electrons during oxidation-reduction reactions. Microorganisms obtain energy by transferring electrons from an electron donor such as an organic compound (or sometimes a reduced inorganic compound such as sulfide) to an electron acceptor. Electron acceptors are compounds that are relatively oxidized and include oxygen, nitrate, iron(III), manganese(IV), sulfate, carbon dioxide, or in some cases chlorinated aliphatic hydrocarbons such as tetrachloroethene (PCE), TCE, DCE and vinyl chloride (VC).

Electron donor: A compound capable of supplying (giving up) electrons during oxidation-reduction reactions. Microorganisms obtain energy by transferring electrons from an electron donor such as an organic compound (or sometimes a reduced inorganic compound such as sulfide) to an

Subject NATURAL ATTENUATION PARAMETER COLLECTION	Number SA-1.6	Page 3 of 21
	Revision 1	Effective Date 09/03

electron acceptor. Electron donors are compounds that are relatively reduced and include fuel hydrocarbons and native organic carbon.

Metabolic byproduct: A product of the reaction between an electron donor and an electron acceptor. Metabolic byproducts include volatile fatty acids, daughter products of chlorinated aliphatic hydrocarbons, methane, and chloride.

Oxic groundwater: Groundwater that contains oxygen in concentrations greater than about 0.5 mg/L.

Oxidation/reduction reaction: A chemical or biological reaction wherein an electron is transferred from an electron donor (donor is oxidized) to an electron acceptor (acceptor is reduced).

Predominant terminal electron-accepting process: The electron-accepting process (oxygen reduction, nitrate reduction, iron(III) reduction, etc.) that sequesters the majority of the electron flow in a given system.

Reductive dechlorination: Reduction of a chlorine-containing organic compound via the replacement of chlorine with hydrogen.

Respiration: The process of coupling the oxidation of organic compounds with the reduction of inorganic compounds such as oxygen, nitrate, iron(III), manganese(IV), and sulfate.

Seepage velocity: The average velocity of groundwater in a porous medium.

Substrate: A compound used by microorganisms to obtain energy for growth. The term can refer to either an electron acceptor or an electron donor.

4.0 RESPONSIBILITIES

Project Manager (PM) / Task Order Manager (TOM) - Responsible for ensuring that all field activities are conducted in accordance with this standard operating procedure (SOP).

Project Hydrogeologist or Geochemist - Responsible for selecting and detailing the specific groundwater sampling techniques, onsite water quality testing (type, frequency, and location), and equipment to be used, and providing detailed input in this regard to the project plan documents. The project hydrogeologist or geochemist is also responsible for properly briefing and overseeing the performance of the site sampling personnel.

Site Manager (SM) / Field Operations Leader (FOL) - Responsible for the onsite verification that all field activities are performed in compliance with approved SOPs or as otherwise directed by the approved project plan(s).

Project Geologist - is primarily responsible for the proper acquisition of the groundwater samples. He/she is also responsible for the actual analyses of onsite water quality samples, as well as instrument calibration, care, and maintenance. When appropriate, such responsibilities may be performed by other qualified personnel (e.g., field sampling technicians or site personnel).

5.0 PROCEDURES

5.1 General

Natural attenuation includes physical, chemical, and biochemical processes affecting the concentrations of dissolved contaminants in groundwater. These processes may include advection, dispersion, volatilization, dilution, sorption to aquifer solids, and/or precipitation or mineralization of compounds. Of greatest importance are those processes that lead to a reduction in contaminant mass (by degrading or destroying contaminants) such as biodegradation. These biochemical processes remove organic contaminants from the aquifer by destruction. Depending on the type of contaminant, particularly the organic contaminant (e.g., petroleum hydrocarbons or chlorinated organic solvents), the biochemical environment in the aquifer will vary. The biochemical environment within the aquifer influences and is influenced by the activities of aquifer microbiota. Specific types of microbiota, working singly or in complex consortia, may use organic contaminants as part of their normal cell functions. Natural

Subject NATURAL ATTENUATION PARAMETER COLLECTION	Number SA-1.6	Page 4 of 21
	Revision 1	Effective Date 09/03

attenuation monitoring is designed to measure indicators of the biochemical environment within the aquifer and, with direct and indirect lines of evidence and associated chemical concentration data, evaluate the likely fate (i.e., transformation, destruction, dilution, attenuation, etc.) of organic contaminants.

5.2 Planning for Natural Attenuation Sampling

The first step in preparing a natural attenuation investigation is to develop a site-specific conceptual model. The first step in development of this model is the analysis and review of available site-specific characterization data. The development and refinement of this model should be supplemented with additional data as needed. The data should include but is not limited to:

- Geologic and hydrogeologic information in three dimensions
- Nature, extent, and magnitude of contamination
- Location and presence of potential receptors to contamination

Lines of Evidence

Several lines of evidence are used to determine whether natural attenuation is working. The most compelling, primary evidence is decreasing groundwater contaminant concentrations over time. Decreasing concentration trends can be demonstrated in several ways including:

- Isoconcentration maps of the dissolved plume over time wherein the extent of the plume is either stable or decreasing.
- Time series plots of contaminant concentrations within a well illustrating a clear downward trend.
- Contaminant concentration profiles in a series of monitoring wells along a groundwater flow path illustrating decreasing concentrations beyond that attributable to dilution and dispersion.

Secondary, or supporting, lines of evidence include:

- Analytical data showing production and subsequent destruction of primary contaminant breakdown products.
- Geochemical data indicating that the biochemical environment is favorable for the appropriate microbiota.
- Geochemical data that indicate the aquifer microbiota are active.

Monitoring Well Location and Sampling Frequency

The number and locations of wells required to monitor natural attenuation will depend on the physical setting at each location. One possible array of monitoring wells is illustrated in Attachment A. In this scenario, one well is used to monitor conditions upgradient of the source, one well is located in the source area, and several wells are used to define and monitor the downgradient and lateral extent of the dissolved plume. At a minimum, there should be at least one upgradient well (ideally with no contamination present), one well in the source area, one well downgradient from the source area in the dissolved plume, and one downgradient well where contaminant concentrations are below regulatory criteria. Note that the number and locations of monitoring wells will vary depending on the site complexity and site objectives.

Subject NATURAL ATTENUATION PARAMETER COLLECTION	Number SA-1.6	Page 5 of 21
	Revision 1	Effective Date 09/03

Sampling frequency will be dictated by the ultimate use of the data and site-specific characteristics. Contaminant concentrations may be used to define statistically meaningful trends in contaminant concentrations. The sampling frequency may be defined by the hydrogeologic and/or geochemical conditions as well as the proposed statistical method for data analysis. For example, groundwater flow and contaminant characteristics (e.g., seepage velocity and contaminant loading) may dictate the sample frequency. Regardless of the factors, sampling frequency and duration will need to establish the range of natural chemical variability within the aquifer. After a sufficient amount of data has been collected and the geochemical conditions are understood, the frequency of sampling may be reduced. See Section 5.4 for additional information on sample collection and frequency.

5.3 Selection of Natural Attenuation Parameters

Natural attenuation via biodegradation depends on the nature of the organic contaminants and the oxidation-reduction (redox) environment within the aquifer. Simply stated, if the contaminants are fuels, biodegradation will be most effective if the redox conditions are aerobic or oxidizing. If the contaminants are chlorinated solvents, the biodegradation will be most effective (in the source and near source areas) if redox conditions in the aquifer are anaerobic or reducing.

Several parameters are needed to evaluate whether natural attenuation is taking place and, if so, the rate at which it may be occurring. The primary parameter providing direct evidence of natural attenuation is the aqueous concentrations of parent and daughter volatile organic compounds. More specifically, a decrease in parent products, an increase in daughter products, evidence that the plume is stable or shrinking in size, and overall decline in contaminant concentrations is direct evidence of natural attenuation. Natural attenuation or geochemical parameters that provide information about the redox conditions in the aquifer include:

- Dissolved oxygen
- Nitrate/nitrite
- Dissolved manganese
- Iron
- Sulfate/sulfide
- Methane
- Oxidation-reduction potential (ORP)

Secondary parameters that indicate biological activity in the aquifer and thereby support the natural attenuation evaluation include:

- Dissolved hydrogen
- Alkalinity
- Dissolved carbon dioxide

The concentrations of natural attenuation parameters are used to define the aquifer redox conditions. It is important to record and document the presence or absence (i.e., measurable or not measurable concentration) of certain natural attenuation parameters. The presence or absence of a certain substance may be sufficient to indicate the redox condition within the aquifer. By reference to Attachment B, which illustrates the typical sequence of biologically mediated redox reactions in natural systems, it is apparent that, for example, sulfate reduction (producing dissolved sulfide in groundwater) does not operate in an aerobic environment. Therefore, measurable sulfide should not be present if there is also dissolved oxygen at concentrations indicating an aerobic environment. Attachment B also illustrates the redox potential (measured in millivolts) associated with the redox reactions. ORP readings, also in millivolts, measured during well purging, may be compared with the range of values in Attachment B but with caution. Redox potentials measured with a platinum electrode in natural water samples may be misleading, especially when biologically mediated reactions are important, because many of the critical

Subject NATURAL ATTENUATION PARAMETER COLLECTION	Number SA-1.6	Page 6 of 21
	Revision 1	Effective Date 09/03

reactions in Attachment B do not generate a response in the electrode. Dissolved hydrogen concentration ranges associated with important redox reactions are also indicated in Attachment B. Because dissolved hydrogen is actually used by microbiota during redox reactions, its concentration may provide an additional indicator of the overall redox condition in the aquifer.

Attachments C and D tabulate the natural attenuation parameters for chlorinated volatile organic compound and petroleum hydrocarbon plumes, respectively. The parameters listed in these tables are organized in order of importance. Parameters selected for analysis shall be determined based on site conditions, project-specific plans, and/or other criteria established for the project. Based on these criteria, it is possible that all of the parameters may be selected.

5.4 Selection of Natural Attenuation Analytical Methods and Procedures

There are many analytical methods available to measure concentrations of the natural attenuation parameters discussed in the previous sections. Attachment E summarizes the sample methodologies, sampling equipment needed, sample volume, container, preservation, and holding time requirements. This table also summarizes the detection limits and the detection ranges for each method. A number of factors should be considered when selecting the appropriate sample analytical methodology including the required parameters, appropriate detection ranges for each compound, cost, and ease of use in the field. For example, when determining the correct methodology for measuring concentrations of total sulfide, the metabolic byproduct of sulfate reducing conditions, it is important to analyze for each of the forms of sulfide (H_2S , S^{2-} , and HS^-). Also, when the detection limit of the selected method is exceeded, another method may be considered, or the sampler may be able to dilute the sample (per manufacturer's instructions) to quantify it within the detected range. In terms of cost, some parameters are very time consuming when performed in the field. Without sacrificing sample integrity it may be more appropriate to select a methodology performed in a fixed-base laboratory. Finally, in terms of ease of use, certain field methods are generally easier compared to other methods. Using simpler methods may result in better quality sample results and increased sample repeatability without sacrificing sample integrity. For example, in some cases CHEMetrics Titret® Titration Ampule kits may be a good alternative to other hand digital titration methods.

The sample technicians should be aware that based on geochemical conditions recorded in the field, certain geochemical parameters may not have positive detections. For example, if dissolved oxygen concentrations indicate aerobic conditions then it is unlikely that dissolved hydrogen is present (see Section 5.10 for additional information). Another example is alkalinity. If the pH of the groundwater sample is less than 4.5, then it is unlikely that alkalinity will be measurable. Despite the potential for non-detect results, in cases such as those described above, all parameters should be collected in the field based upon project plans. The value in collecting the parameters in the future shall be determined by the project hydrogeologist and/or geochemist in accordance with the projects planning documents data quality objectives (DQO) and the items discussed in Section 5.2.

5.5 Procedures for Sample Collection

Groundwater sample collection for natural attenuation sampling should be performed using low flow purging and sampling techniques. These techniques are described in detail in SOP SA-1.1. Low flow purging and sampling procedures should be used to ensure the collection of a sample that is "representative" of the water present in the aquifer formation. Minimizing stress on the aquifer formation during low flow purging and sample collection ensures that there are minimal alternations to the water chemistry of the sample. The criteria used in the purging process should include minimization of drawdown in the well, stabilization of applicable indicator parameters, and evacuation of a sufficient amount of purge volume in accordance with SOP SA-1.1, project plans, and/or applicable regulatory guidance.

Subject NATURAL ATTENUATION PARAMETER COLLECTION	Number SA-1.6	Page 7 of 21
	Revision 1	Effective Date 09/03

Groundwater purging and sampling for natural attenuation should be performed using submersible pumps (e.g., bladder pumps) in accordance with SOP SA-1.1. However, in accordance with project plans and applicable regulatory guidance, peristaltic pumps may also be used for this purpose. Limitations of and factors associated with using these devices should be considered (see SOP SA-1.1 for more information). As a result of difficulties in collecting "representative" groundwater samples, bailers should not be used for the collection of natural attenuation samples.

It is critical that disturbance and aeration of samples monitored and collected at the well head are minimized. As a result, a flow-through sampling cell and a direct reading meter shall be used for the measurement of well stabilization indicator parameters (e.g., pH, conductivity, temperature, dissolved oxygen, turbidity, and ORP) at the well head. The pump effluent tubing should be placed at the bottom of the flow-through cell allowing effluent water from the cell to discharge at the top of the meter (above the detector probes) to minimize the agitation of water in the cell.

Documentation of the purging process shall be recorded during and at the completion of purging as discussed in Section 5.8. Immediately following the purging process and before sampling, all applicable indicator parameters must be measured and recorded on the appropriate sample log sheets as discussed in Section 5.8.

After all of the purging requirements have been met, groundwater sampling and natural attenuation data collection can begin. Monitoring wells will be sampled using the same pump and tubing used during well purging.

5.6 Procedures for Field Sample Analysis

Each of the field and fixed-base laboratory sample parameters requires different sampling procedures and holding times. Attachment E presents parameter-specific requirements for sampling, analysis, and storage of all of the parameters and methods sampled as part of natural attenuation analysis.

Due to parameter procedure and holding times, it is important to consider the sequence of sample collection and analysis. Generally speaking, with the exception of volatile organic compounds, field parameters shall be analyzed first followed by fixed-base laboratory sample collection. All samples will be collected in a sequence and manner that minimizes volatilization, oxidation, and/or chemical transformation of compounds. As a result, the following sample and analysis order should be followed:

- | | |
|---|------------------------------------|
| 1. Volatile organic compounds | 8. Nitrate / Nitrite |
| 2. Dissolved oxygen | 9. Dissolved manganese |
| 3. Alkalinity | 10. Semivolatile organic compounds |
| 4. Dissolved carbon dioxide | 11. Other dissolved metals |
| 5. Dissolved ferrous iron | 12. Total metals |
| 6. Dissolved sulfide (hydrogen sulfide and sulfide) | 13. All other constituents |
| 7. Dissolved hydrogen, methane, ethene, and ethane | |

Field-analyzed parameters should be collected and immediately analyzed directly from the pump effluent per the requirements on Attachment E and manufacturer's recommendations. Care should be taken to minimize any unnecessary disturbance, aeration, or agitation of the sample prior to analysis. It is not acceptable to collect and store samples that are to be analyzed immediately at the well head in a temporary holding container (e.g., open topped pitcher) to be analyzed at a later time.

The manufacturer's procedure manual for each of the field-based analyses shall be maintained in the field during the entire sampling program. The procedures give a detailed explanation of how to perform each particular method and include information on sampling, storage, accuracy checks, interferences, reagents, and apparatus needed to perform each analysis.

Subject NATURAL ATTENUATION PARAMETER COLLECTION	Number SA-1.6	Page 8 of 21
	Revision 1	Effective Date 09/03

5.7 Procedures for Quality Assurance and Quality Control Field Sample Analysis

Accuracy and precision checks shall be performed to check the performance of the reagents, apparatus, and field analytical procedures per the manufacturer's recommendations. The accuracy checks should include the use of standard solutions (i.e., standard addition), as appropriate. The manufacturer's field test kit manual provides details on how to perform each of the accuracy checks for each parameter where applicable. Refer to Section 6.0 for manufacturer contact information.

Precision checks must include the performance of duplicate analysis. When using a colorimeter, precision checks may also include reagent blank corrections and standard curve adjustments as recommended by the manufacturer. Field duplicate results shall be performed and evaluated for relative percent difference (RPD) at a rate of 1 per 10 samples or as determined by the project plans. The RPD can be calculated as follows:

$$RPD = \left| \frac{\text{First result} - \text{Second result}}{\text{Mean arithmetic (average) of first and second result}} \right| \times 100$$

If the RPD exceeds 50 percent, it is required that the test be performed again to verify the result. The duplicate results shall be documented in the 'Notes' section for that specific parameter on the appropriate sample logsheet (see Section 5.8).

If a colorimeter (e.g., HACH DR-890 or equivalent) is used for parameter analysis, an instrument performance verification test using absorbance standards may also be performed to ensure the meter is providing accurate measurements.

The following table lists examples of the types and frequencies of accuracy checks required for each parameter. Refer to the manufacturer's instructions for information regarding other analyses.

Parameter	Method	Standard Solution	Field Duplicate	Reagent Blank Correction
Alkalinity	CHEMetrics K-9810, -15, -20	None	1 per 10	None
Carbon dioxide	CHEMetrics K-1910, -20, -25	None	1 per 10	None
Dissolved oxygen	CHEMetrics K-7501, -12	None	1 per 10	None
Ferrous iron	HACH DR-890	None	1 per 10	None
Nitrite	HACH DR-890	1 per round	1 per 10	1 per lot
Nitrate	HACH DR-890	1 per round	1 per 10	1 per lot
Sulfide	HACH DR-890	None	1 per 10	None
Hydrogen sulfide	HACH HS-C	None	1 per 10	None

Prior to analysis, the expiration dates of reagents shall be checked. If the reagents have exceeded their expiration date or shelf life, the reagents shall be replaced. If deviations from the applicable analytical procedure are identified, the deviations shall be corrected and the associated samples re-analyzed. If problems are identified with the reagents, apparatus, or procedures, data interferences may be present. Interferences may also be due to other factors (e.g., pH, presence or concentration of other ions, turbidity, temperature, etc.) that may interfere with the sample result. The manufacturer's procedures (e.g., Hach, 1999) should be reviewed prior to analysis to avoid or minimize such interferences. Associated problems

Subject NATURAL ATTENUATION PARAMETER COLLECTION	Number SA-1.6	Page 9 of 21
	Revision 1	Effective Date 09/03

or suspected interferences shall be documented in the 'Notes' section of the sample logsheet. Often, interferences cannot be avoided. In these cases, the sampler should be aware of these potential interferences and document them properly.

5.8 Documentation Procedures for Field Sample Analysis

Field results shall be properly documented in the field as noted in SOP SA-6.3. The sample log sheet titled "Field Analytical Log Sheet, Geochemical Parameters" shall be prepared for each sample collected and analyzed in the field. A copy of this form can be found as Attachment F of this SOP. Other field log sheets (e.g., low flow purge log sheet, groundwater sample logsheet, etc.) shall also be completed in accordance with SOP SA-6.3.

Specific information shall also be recorded in the project logbook. This information shall include, but is not limited to, the test kit name and model number, lot number and expiration date of the test kit and reagents used, serial number of the instrument (e.g., colorimeter) used for the analysis, and results of the quality assurance and quality control field sample analysis. Because environmental conditions and changes in those conditions may affect the field analytical results, it is important to document the site conditions (weather, temperature, etc.) at the time of sampling in the logbook in accordance with SOP SA-6.3.

5.9 Waste Handling and Disposal

Several of the test kits listed in Attachment E require the use of chemicals and materials that must be properly handled and disposed of in a proper and responsible manner. Refer to specific manufacturer's guidance for handling and disposal practices. See also Section 6.0 for more detailed and complete information. Handling and disposal of these items should be conducted in accordance with all local, state, and federal guidelines.

5.10 Understanding Field Sample Analytical Results

Natural attenuation data interpretation is complicated by the complex inter-relationships of various parameters. The complexity reflects the myriad of biochemical processes. Real-time evaluation of field analytical data can be misleading because a full interpretation often requires combining the field analytical results with fixed-base laboratory results. Regardless, some simple observations and data interpretations in the field may provide insights about the monitoring system or early warnings about sample collection and handling problems.

Data collected from the designated upgradient monitoring well is the baseline from which other interpretations are made. Field analytical data will indicate that the upgradient environment is either oxidizing or reducing. The redox condition within the upgradient area of the aquifer may be natural or impacted by other contaminant source areas (see Section 5.2 for upgradient well selection). Regardless, the redox condition of the upgradient groundwater will influence the source area. Changes in field analytical results from the upgradient well to the source area well will be reflected in samples from monitoring wells further downgradient.

The general characteristics of the two redox environments are summarized in the following table.

Subject NATURAL ATTENUATION PARAMETER COLLECTION	Number SA-1.6	Page 10 of 21
	Revision 1	Effective Date 09/03

Aerobic/Oxidizing	Anaerobic/Reducing
<ul style="list-style-type: none"> • Measurable dissolved oxygen (>1 to 2 ppm) • Measurable nitrate • No measurable dissolved manganese • No measurable dissolved ferrous iron • Measurable dissolved sulfate • No measurable dissolved sulfide • No measurable dissolved methane • No measurable dissolved hydrogen 	<ul style="list-style-type: none"> • No measurable dissolved oxygen (<1 ppm) • No measurable nitrate • Measurable dissolved manganese • Measurable dissolved ferrous iron • No measurable dissolved sulfate • Measurable dissolved sulfide • Measurable dissolved methane • Measurable dissolved hydrogen

Transitional environments between these two extremes may have intermediate characteristics and are actually quite common. Because reactions are mediated by biological systems, equilibrium (the basis for the figure in Attachment B) conditions within the aquifer should not be expected. For example, sulfate reduction environments may occur in close proximity to methanogenic environments, and this natural attenuation data may be difficult to interpret. Carefully collected and analyzed field measurements and sample collections for fixed-base laboratory analyses are designed to characterize the aquifer environment along the continuum between strongly aerobic and strongly anaerobic. Because the land surface environment is generally more oxidizing than any groundwater environment, sample handling at the point of collection and analysis is extremely important in preserving the chemical integrity of the groundwater sample.

6.0 REFERENCES

American Society for Testing and Materials (ASTM), 1998. Standard Guide for Remediation of Ground Water by Natural Attenuation at Petroleum Release Sites, Designation: E1943-98, West Conshohocken, Pennsylvania.

Chemetrics, 2002, <http://www.chemetrics.com>.

Department of the Navy, 1998. Technical Guidelines for Evaluating Monitored Natural Attenuation of Petroleum Hydrocarbons and Chlorinated Solvents in Ground Water at Naval and Marine Corps Facilities, Department of the Navy, September. Prepared by T. H. Weidemeier and F. H. Chappelle.

USEPA (United States Environmental Protection Agency), 1998. Technical Protocol for Evaluating Natural Attenuation of Chlorinated Solvents in Ground Water, EPA/600/R-98/128, Office of Research and Development, Washington, D.C.

Hach Company, 1999. DR-890 Colorimeter Procedures Manual, Product Number 48470-22, Loveland Colorado.

Hach Company, 1999. Digital Titrator (manual), Model Number 16900, Catalog Number 16900-08. Loveland, Colorado.

Hach Company, 2002, <http://www.hach.com/>.

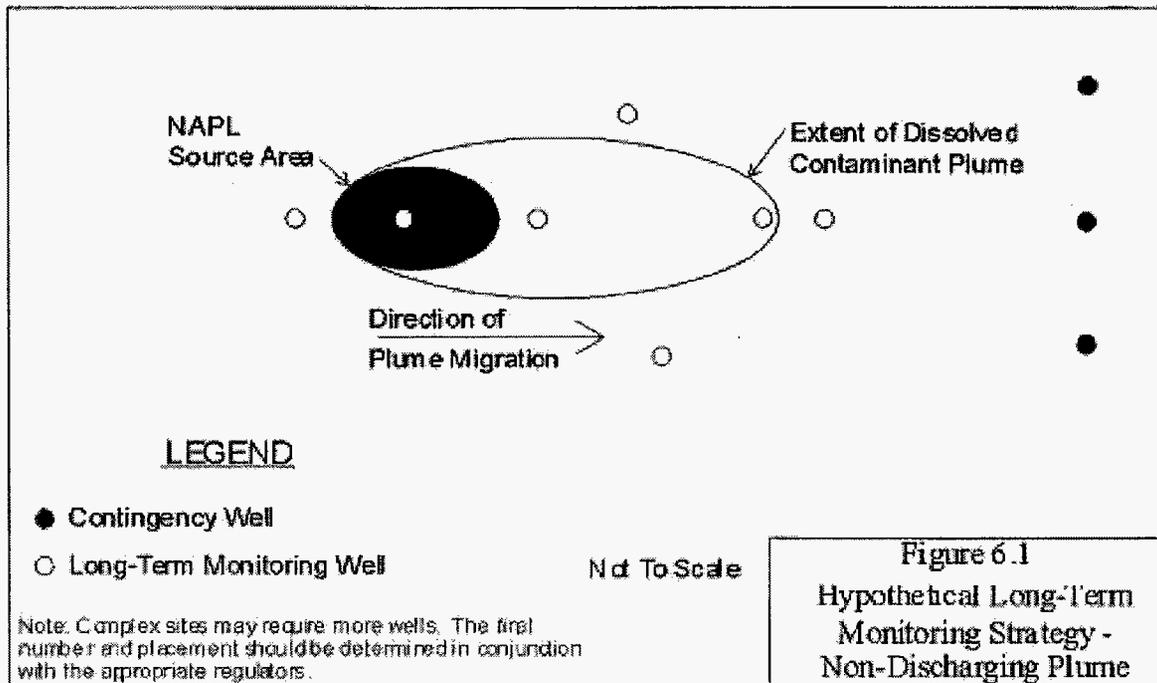
USEPA, 1997. Draft EPA Region 4 Suggested Practices for Evaluation of a Site for Natural Attenuation (Biological Degradation) of Chlorinated Solvents; Version 3.0. November.

USEPA, 1999. Use of Monitored Natural Attenuation at Superfund, RCRA Corrective Action, and Underground Storage Tank Sites, USEPA OSWER Directive 9200.4-17P, April 21, 1999

Subject NATURAL ATTENUATION PARAMETER COLLECTION	Number SA-1.6	Page 11 of 21
	Revision 1	Effective Date 09/03

ATTACHMENT A

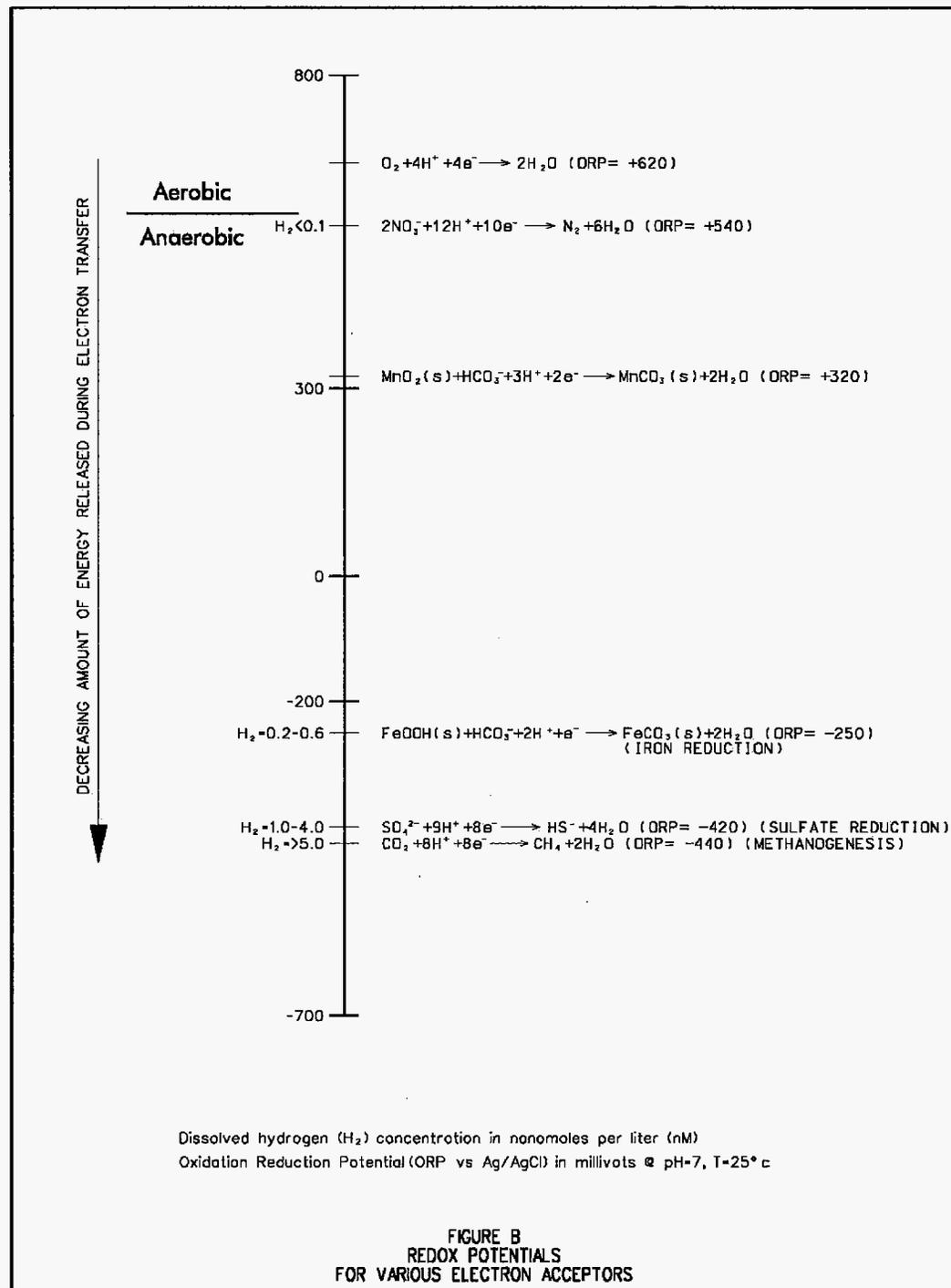
HYPOTHETICAL LONG-TERM MONITORING STRATEGY



Taken from: Department of the Navy, 1998, Technical Guidelines for Evaluating Monitored Natural Attenuation of Petroleum Hydrocarbons and Chlorinated Solvents in Ground Water at Naval and Marine Corps Facilities, Prepared by Todd Weidemeier and Francis Chappelle.

ATTACHMENT B

REDOX POTENTIALS FOR VARIOUS ELECTRON ACCEPTORS



k:\dgr\mavy\orlando\files\sa2\sa2-033.dgn 9-19-02

Subject NATURAL ATTENUATION PARAMETER COLLECTION	Number SA-1.6	Page 13 of 21
	Revision 1	Effective Date 09/03

ATTACHMENT C

NATURAL ATTENUATION PARAMETERS FOR CHLORINATED VOLATILE ORGANIC COMPOUND PLUMES SCREENING PROCESS SUMMARY FOR REDUCTIVE (ANAEROBIC) DECHLORINATION

Potential Electron Donors	Electron Acceptors:	Reduced Species:	Related Dechlorination Pathway:
Native total organic carbon (TOC) Anthropogenic carbon (e.g., leachate) Fuel hydrocarbons (e.g., BTEX) Lightly chlorinated solvents (DCE/VC)	Dissolved Oxygen	⇒ Carbon Dioxide (CO ₂)	~ DCE → VC → CO ₂
	Manganese (Mn ⁴⁺)	⇒ Manganese (Mn ²⁺)	~ DCE → VC
	Nitrate (NO ₃)	⇒ Nitrite (NO ₂)	~ DCE → VC
	Ferric Iron (Fe ³⁺)	⇒ Ferrous Iron (Fe ²⁺)	~ DCE → VC → CO ₂
	Sulfate (SO ₄)	⇒ Sulfide (S ²⁻ , HS ⁻ , H ₂ S)	~ TCE → DCE → VC → Ethene
	Carbon Dioxide (CO ₂)	⇒ Methane (CH ₄)	~ PCE → TCE → DCE → VC → Ethene

Geochemical Parameter List:

Parameter	Field or Lab	Rationale	Importance
Volatile organic compounds	L	Source products; daughter products; electron donors (e.g., benzene, toluene, ethylbenzene, and xylene; BTEX)	1
Dissolved oxygen	F	Primary electron acceptor (respiration); an/aerobic indicator	1
Nitrate (and nitrite), dissolved	F or L	Anaerobic electron acceptor (product of nitrate reduction)	1
Manganese, dissolved	F or L	Anaerobic electron acceptor	1
Ferrous Iron (Fe ²⁺)	F	Product of iron reduction	1
Sulfate [and sulfide (S ²⁻)]	F or L	Common anaerobic electron acceptor (product of sulfate reduction)	1
Sulfide (H ₂ S)	F	Common product of sulfate reduction	1
Methane, ethane, ethene	L	Product of methanogenesis; daughter products of reductive dechlorination	1
Chloride	L	Ultimate daughter product of reductive dechlorination	1
TOC - upgradient groundwater	L	Electron donor	1
ORP, pH, specific conductance, temperature, turbidity	F	General water quality determination	1
Carbon dioxide (CO ₂)	F	Anaerobic electron acceptor (methanogenesis); biotic respiration indicator	2
Alkalinity/DIC	F	Buffering capacity; biotic respiration indicator	2
Hydrogen, dissolved	L	Fingerprint for characterizing electron acceptor pathway - indicator of what redox is occurring	2
TOC - upgradient soil	L	Input to analytical NA models; quantifies soil-water distribution coefficient and retardation factor	2
Volatile fatty acids	L	Determination of anthropogenic carbon used as an electron donor	3

Importance: 1=Most important; 3=Least important (depending on DQOs, all may be recommended). See Attachment E for details regarding analytical methods.

Subject NATURAL ATTENUATION PARAMETER COLLECTION	Number SA-1.6	Page 14 of 21
	Revision 1	Effective Date 09/03

ATTACHMENT D

**NATURAL ATTENUATION PARAMETERS FOR
PETROLEUM HYDROCARBON PLUMES
SCREENING PROCESS SUMMARY FOR OXIDATIVE (AEROBIC) DEGRADATION**

Parameter	Field or Lab	Rationale	Importance
Volatile organic compounds	L	Source products; daughter products; electron donors (BTEX)	1
Dissolved oxygen	F	Primary electron acceptor (respiration); an/aerobic indicator	1
Nitrate (and nitrite), dissolved	F or L	Anaerobic electron acceptor (and product of nitrate reduction)	1
Manganese, dissolved	F or L	Anaerobic electron acceptor	1
Ferrous Iron (Fe ²⁺)	F	Product of iron reduction	1
Sulfate [and Sulfide (S ⁻²)]	F or L	Common anaerobic electron acceptor (product of sulfate reduction)	1
Sulfide (H ₂ S)	F	Common product of sulfate reduction	1
TOC - upgradient groundwater	L	Electron donor	1
ORP, pH, specific conductance temperature, turbidity	F	General water quality determination	1
Dissolved methane (CH ₄)	L	Product of methanogenesis	1
Anions: chloride (Cl), nitrate (NO ₃), nitrite (NO ₂), phosphate (PO ₄), sulfate (SO ₄)	L		1
TOC - Upgradient soil	L	Input to analytical NA models; quantifies soil-water distribution coefficient and retardation factor	2
Biological oxygen demand (BOD)	L	Understanding of aquifer oxygen demand	3
Chemical oxygen demand (COD)	L	Understanding of aquifer oxygen demand	3

Importance: 1=Most important; 3=Least important (depending on DQOs, all may be recommended).

See Attachment E for details regarding analytical methods.

Subject

NATURAL ATTENUATION
PARAMETER COLLECTION

Number

SA-1.6

Page

15 of 21

Revision

1

Effective Date

09/03

ATTACHMENT E
GEOCHEMICAL SAMPLING PARAMETERS - METHODS, EQUIPMENT, VOLUMES, CONTAINERS,
PRESERVATION, HOLDING TIMES, AND DETECTION RANGES
PAGE 1 OF 4

Parameter	Method / Reference	Equipment / Method Chemistry	Sample Volume, Container, Preservation, & Holding Time	Range (mg/L)	Precision (mg/L)	Estimated Detection Limit (mg/L)
Alkalinity	CHEMetrics K-9810, K-9815, K-9820 -ASTM D 1067-92 -EPA 310.1	Titret® Titration Ampules / Hydrochloric Acid, Phenolphthalein	Field. Follow test kit instructions. Avoid agitation and analyze at well head to determine total alkalinity. Filter if turbid (>10 NTU).	10-100 (K-9810) 50-500 (K-9815) 100-1000 (K-9820)	N/A	10 50 100-
Alkalinity	Fixed-base lab -EPA 310.1	N/A	100 to 250 mL in glass or plastic container. Cool to 4°C. Analyze within 14 days. Filter if turbid.	N/A	N/A	N/A
Alkalinity / Dissolved Inorganic Carbon	HACH AL-DT -HACH 8203 -SM 2320 / SM 403	Digital Titration / Hydrochloric Acid, Phenolphthalein (P) and Total (M)	Field. Follow test kit instructions. Avoid agitation and analyze at well head to determine carbonate, bicarbonate, and hydroxide ions. Filter if turbid as recommended by manufacture. May use a pH meter for colored samples.	10-4000	N/A	10
Arsenic	Fixed-base lab -SW-6010 B	N/A	1 liter glass or polyethylene container, HNO ₃ to pH ≤ 2. 6 months.	N/A	N/A	N/A
Biochemical Oxygen Demand	Fixed-base lab -EPA 410.1	N/A	2 liter HDPE. Cool to 4°C. Analyze within 48 hours.	N/A	N/A	N/A
Carbon Dioxide, dissolved	CHEMetrics K-1910, K-1920, K-1925 -ASTM D 513.82 -SM 4500-CO ₂ -C	Titret® Titration Ampules / Sodium Hydroxide, Phenolphthalein	Field. Follow test kit instructions. Avoid agitation and analyze at well head.	10-100 (K-1910) 100-1000 (K-1920) 250-2500 (K-1925)	N/A	10 100 250
Carbon Dioxide, dissolved	Fixed-base lab -VOA water sample (Vaportech)	GC-ECD/RGD/FID Detector	40 mL in VOA vial. 2 to 3 vials by (Vaportech).	N/A	N/A	N/A
Carbon Dioxide, dissolved	Fixed-base lab -Microseeps gas stripping cell	GC-ECD/RGD/FID Detector	Field bubble-strip sampling required. Ship in glass septum vial (Microseeps only).	N/A	N/A	N/A
Carbon Dioxide, dissolved	HACH CA-DT -HACH 8205 -Mod. SM 406	Digital Titration / Sodium Hydroxide, Phenolphthalein	Field. Follow test kit instructions. Do not aerate or agitate. Analyze at well head.	10-1000	N/A	10
Chemical Oxygen Demand	Fixed-base lab -EPA 410.1	N/A	125 mL HDPE. H ₂ SO ₄ to pH <2.0. Cool to 4°C. Analyze within 28 days.	N/A	N/A	N/A
Chloride (Cl)	Fixed-base lab -EPA 300	N/A	100 to 250 mL in glass or plastic container. Cool to 4°C. Analyze within 28 days.	N/A	N/A	N/A
Chlorine - Total (Cl ₂)	HACH DR-850 -HACH 8167 -SM 4500-Cl	Colorimeter / DPD Method	Field. Follow test kit instructions.	0.02-2.00	± 0.01 mg/L with a 1.00 mg/L chlorine solution.	1
Conductance, Specific	Field Meter -SW-9050 A	Direct Reading Meter	100 to 250 mL in glass or plastic container. Analyze immediately.	N/A	N/A	N/A
Ethane, dissolved	Fixed-base lab -VOA water sample, Vaportech -RSK SOP-147 & 175	GC-ECD/RGD/FID Detector	40 mL in VOA vial. 2 to 3 vials by (Vaportech).	N/A	N/A	N/A
Ethane, dissolved	Fixed-base lab -Microseeps gas stripping cell -RSK SOP-147 & 175	GC-ECD/RGD/FID Detector	Field bubble-strip sampling required. Ship in glass septum vial (Microseeps only).	N/A	N/A	N/A

Subject

NATURAL ATTENUATION
PARAMETER COLLECTION

Number

SA-1.6

Page

16 of 21

Revision

1

Effective Date

09/03

ATTACHMENT E

GEOCHEMICAL SAMPLING PARAMETERS - METHODS, EQUIPMENT, VOLUME, CONTAINER,
PRESERVATION, HOLDING TIME, AND DETECTION RANGES
PAGE 2 OF 4

Parameter	Method / Reference	Equipment / Method Chemistry	Sample Volume, Container, Preservation, & Holding Time	Range (mg/L)	Precision (mg/L)	Estimated Detection Limit (mg/L)
Ethene, dissolved	Fixed-base lab -VOA water sample, Vaportech -RSK SOP-147 & 175	GC-ECD/RGD/FID Detector	40 mL in VOA vial. 2 to 3 vials by (Vaportech).	N/A	N/A	N/A
Ethene, dissolved	Fixed-base lab -Microseeps gas stripping cell -RSK SOP-147 & 175	GC-ECD/RGD/FID Detector	Field bubble-strip sampling required. Ship in glass septum vial (Microseeps only).	N/A	N/A	N/A
Fraction Organic Carbon (foc)-Soil Upgradient Saturated Soil	Fixed-base lab -Walk-Black -SW-846 9060	N/A	200 gram glass jar. Cool to 4°C. Analyze within 14 days.	N/A	N/A	N/A
Hydrogen, dissolved	Fixed-base lab -Microseeps or Vapor Tech gas stripping cell -RSK SOP-147 & 175	GC-ECD/RGD/FID Detector	Field bubble-strip sampling required. Ship in glass septum vial.	N/A	N/A	N/A
Iron, ferrous (Fe ²⁺)	HACH DR-850 -HACH 8146 -Mod. SM 315 B	Colorimeter 1, 10 Phenanthroline	Field. Follow test kit instructions. Analyze immediately at well head. Filter if turbid (>10 NTU) as recommended by the manufacture.	0-3.00	±0.017 mg/L with a 2.00 mg/L Fe ²⁺ solution.	0.03
Iron, ferrous (Fe ²⁺)	HACH IR-18C -Mod. SM 315 B	Color Disc 1, 10 Phenanthroline	Field. Follow test kit instructions. Analyze immediately at well head. Filter if turbid (>10 NTU) as recommended by the manufacture.	0-10	N/A	0.2
Iron, total dissolved (Filtered)	Fixed-base lab -SW-846 6010B	N/A	250 mL in plastic container. Field filter to 0.45 µ. HCl to pH <2. Cool to 4°C. Analyze within 6 months.	N/A	N/A	N/A
Manganese (Mn ²⁺)	HACH DR-850 -HACH 8034 -CFR 44(116) 34193	Colorimeter / Cold Periodate Oxidation	Field. Follow test kit instructions. Avoid agitation and analyze at well head. Filter if turbid as recommended by the manufacture.	0-20.0	+ 0.19 mg/L with a 10.00 mg/L Mn solution.	0.12
Manganese (Mn ²⁺)	HACH MN-5 -Mod. SM 319 B -CFR 44(116) 34193	Color Disc / Cold Periodate Oxidation	Field. Follow test kit instructions. Avoid agitation and analyze at well head. Filter if turbid as recommended by the manufacture.	0-3	N/A	0.1
Manganese, total dissolved (Filtered)	Fixed-base lab -SW-846 6010B	N/A	250 mL in plastic container. Field filter to 0.45 µ. HCl to pH <2. Cool to 4°C. Analyze within 6 months.	N/A	N/A	N/A
Methane, dissolved	Fixed-base lab -VOA water sample, Vaportech -RSK SOP-147 & 175	GC-ECD/RGD/FID Detector	40 mL in VOA vial. 2 to 3 vials by (Vaportech).	N/A	N/A	N/A
Methane, dissolved	Fixed-base lab -Microseeps gas stripping cell -RSK SOP-147 & 175	GC-ECD/RGD/FID Detector	Field bubble-strip sampling required. Ship in glass septum vial (Microseeps only).	N/A	N/A	N/A
Nitrate (NO ₃)	Fixed-base lab -EPA 300	N/A	250 mL plastic container. Cool to 4°C. Analyze within 48 hours.	N/A	N/A	N/A
Nitrate (NO ₃)	HACH DR-850 -HACH 8192 -Mod. EPA 353.2	Colorimeter / Cadmium Reduction	Field. Follow test kit instructions. Avoid agitation and analyze at well head. Pretreatment required if nitrite is present.	0-0.50	± 0.03 mg/L with a 0.25 mg/L of nitrate nitrogen (NO ₃ ⁻ N) solution.	0.01
Nitrite (NO ₂)	Fixed-base lab -EPA 300	N/A	250 mL plastic container. Cool to 4°C. Analyze within 48 hours. Filter if turbid as recommended by the manufacture.	N/A	N/A	N/A

Subject
**NATURAL ATTENUATION
 PARAMETER COLLECTION**

Number
SA-1.6
 Revision
1

Page
17 of 21
 Effective Date
09/03

ATTACHMENT E

**GEOCHEMICAL SAMPLING PARAMETERS - METHODS, EQUIPMENT, VOLUME, CONTAINER,
 PRESERVATION, HOLDING TIME, AND DETECTION RANGES
 PAGE 3 OF 4**

Parameter	Method / Reference	Equipment / Method Chemistry	Sample Volume, Container, Preservation, & Holding Time	Range (mg/L)	Precision (mg/L)	Estimated Detection Limit (mg/L)
Nitrite (NO ₂ ⁻)	HACH DR-850 -HACH 8507 -Mod. EPA 354.1 -Mod. SM 419 -CFR 44(85) 25595	Colorimeter / Diazotization	Field. Follow test kit instructions. Avoid agitation and analyze at well head. Filter if turbid as recommended by the manufacture.	0-0.350	± 0.001 mg/L with a 0.250 mg/L nitrite nitrogen solution.	0.005
Nitrogen, dissolved	Fixed-base lab -Microseeps gas stripping cell -Vaportech VOA water sample	GC-ECD/RGD/FID Detector	Field bubble-strip sampling required for Microseeps. Ship in glass septum vial (Microseeps) or VOA vial (Vaportech).	N/A	N/A	N/A
Nitrogen, Total Kjeldahl	Fixed-base lab -EPA 351.2	N/A	500 mL plastic/glass container. Cool to 4°C. H ₂ SO ₄ to pH ≤ 2. Analyze within 28 days.	N/A	N/A	N/A
Oxidation Reduction Potential	Field Meter - ASTM D-1498	Direct Reading Meter	Field. Do not aerate. Gently agitate probe using flow over or flow-through method. Analyze immediately at well head.	N/A	N/A	N/A
Oxygen, dissolved	CHEMetrics K-7501, K-7512 -ASTM D 5543-94 -ASTM D 887-92	CHEMets® Vacuum Vials / Rhodazine D and Indigo Carmine	Field. Follow test kit instructions. Avoid agitation and analyze immediately at well head.	0-1 (K-7501) 1-12 (K-7512)	N/A	0.025 1
Oxygen, dissolved	Fixed-base lab -VOA water sample, Vaportech -RSK SOP-147 & 175	GC-ECD/RGD/FID Detector	40 mL in VOA vial. 2 to 3 vials by (Vaportech).	N/A	N/A	N/A
Oxygen, dissolved	Fixed-base lab -Microseeps gas stripping cell -RSK SOP-147 & 175	GC-ECD/RGD/FID Detector	Field bubble-strip sampling required. Ship in glass septum vial (Microseeps only).	N/A	N/A	N/A
Oxygen, dissolved	HACH OX-DT -HACH 8215 -SM 4500-O-G	Digital Titration / Azide Modification of Winkler Digital Titration Method	Field. Follow test kit instructions. Avoid agitation and analyze immediately at well head.	1-10	N/A	1
Oxygen, dissolved	HACH DR-850 (AccuVac Ampules) LR HRDO Method	-Indigo Carmine Method -Rhodazine D Method	Field. Follow test kit instructions. Avoid agitation and analyze immediately at well head.	0-0.8 ppm 0-10 ppm	0.01 ppm 0.1 ppm	N/A
Oxygen, dissolved	Field Meter	Direct Reading Meter	Analyze immediately at well head. Avoid agitation and analyze immediately at well head. Used for well stabilization measurement parameter only.	N/A	N/A	N/A
pH	Field Meter -SW 9040B	Direct Reading Meter	Analyze immediately at well head.	N/A	N/A	N/A
Phosphate (ortho)	Fixed-base lab -EPA 300	Ion Chromatography	250 mL plastic container. Cool to 4°C. Analyze within 48 hours. Filter if turbid as recommended by the manufacture.	N/A	N/A	N/A
Phosphate, potassium	Fixed-base lab -SV-846 6010B	Inductively Coupled Plasma	250 mL plastic container. Cool to 4°C. Analyze within 48 hours. Filter if turbid as recommended by the manufacture.	N/A	N/A	N/A
Salinity	Field Meter	Direct Reading Meter	Analyze immediately.	N/A	N/A	N/A
Sulfate (SO ₄ ²⁻)	Fixed-base lab	N/A	250 mL plastic container. Cool to 4°C. Analyze within 48 hours. Filter if turbid as recommended by the manufacture.	N/A	N/A	N/A
Sulfate (SO ₄ ²⁻)	HACH DR-850 -HACH 8051 -EPA 375.4	Colorimeter / Turbimetric SulfaVer 4	Field. Follow test kit instructions. Filter if turbid as recommended by the manufacture.	0-70	± 0.5 mg/L with a 50 mg/L sulfate solution.	4.9
Sulfide (Hydrogen Sulfide, H ₂ S)	HACH HS-C -HACH Proprietary -Mod. SM 426 C	Color Chart / Effervescence of H ₂ S through sulfide reactive paper.	Field. Follow test kit instructions. Avoid agitation and analyze immediately at well head.	0-5	N/A	0.1
Sulfide (S ²⁻)	CHEMetrics K-9510 -SM 4500-S ²	CHEMets® Vacuum Vials / Methylene Blue	Field. Follow test kit instructions. Avoid agitation and analyze immediately at well head.	0-1 1-10	N/A	0.1 1

Subject
**NATURAL ATTENUATION
 PARAMETER COLLECTION**

Number
 SA-1.6
 Revision
 1

Page
 18 of 21
 Effective Date
 09/03

ATTACHMENT E

**GEOCHEMICAL SAMPLING PARAMETERS - METHODS, EQUIPMENT, VOLUME, CONTAINER,
 PRESERVATION, HOLDING TIME, AND DETECTION RANGES
 PAGE 4 OF 4**

Parameter	Method / Reference	Equipment / Method Chemistry	Sample Volume, Container, Preservation, & Holding Time	Range (mg/L)	Precision (mg/L)	Estimated Detection Limit (mg/L)
Sulfide (S ²⁻)	Fixed-base lab -EPA 376.1/376.2	N/A	1 liter in plastic container, no headspace. NaOH to pH >9. Cool to 4°C. Avoid agitation and analyze within 7 days.	N/A	N/A	N/A
Sulfide (S ²⁻)	HACH DR-850 -HACH 8131 -SM 4500-S ²	Colorimeter / Methylene Blue	Field. Follow test kit instructions. Avoid agitation and analyze immediately at well head. Pretreatment required for turbid samples as recommended by the manufacture.	0-0.70	± 0.02 mg/L with a 0.73 mg/L sulfide solution.	0.01
Sulfide (S ²⁻)	HACH HS-WR -SM 4500-S ²	Color Disc / Methylene Blue	Field. Follow test kit instructions. Avoid agitation and analyze immediately at well head. Pretreatment required for turbid samples as recommended by the manufacture.	0-11.25	N/A	0.1-2.5
Temperature	Field Meter / Thermometer - E 170.1	Direct Reading Meter / Thermometer	Analyze immediately.	N/A	N/A	N/A
Total Organic Carbon (TOC)-Groundwater	Fixed-base lab -E 415.1	N/A	125 mL HDPE. H ₂ SO ₄ to pH < 2.0. Cool to 4°C. Analyze within 28 days.	N/A	N/A	N/A
Turbidity	Field Meter - E 180.1	Direct Reading Meter	Analyze immediately.	N/A	N/A	N/A

N/A = Not applicable.

ATTACHMENT F

**FIELD ANALYTICAL LOG SHEET, GEOCHEMICAL PARAMETERS
PAGE 1 OF 3**

Note: Analyte, method, and/or equipment may be deleted from form if not being performed.



**FIELD ANALYTICAL LOG SHEET
GEOCHEMICAL PARAMETERS**

Tetra Tech NUS, Inc.

Page of

Project Site Name: _____				Sample ID No.: _____																																											
Project No.: _____				Sample Location: _____																																											
Sampled By: _____				Duplicate: <input type="checkbox"/>																																											
Field Analyst: _____				Blank: <input type="checkbox"/>																																											
Field Form Checked as per QA/QC Checklist (initials): _____																																															
SAMPLING DATA:																																															
Date: _____	Color	pH	S.C.	Temp.	Turbidity	DO	Salinity	ORP (Eh)																																							
Time: _____	(Visual)	(S.U.)	(mS/cm)	(°C)	(NTU)	(mg/l)	(%)	(+/- mv)																																							
Method: _____																																															
SAMPLE COLLECTION/ANALYSIS INFORMATION:																																															
ORP (Eh) (+/- mv)				Electrode Make & Model: _____																																											
Reference Electrode (circle one): Silver-Silver Chloride / Calomel / Hydrogen																																															
Dissolved Oxygen:																																															
Equipment: Chemetrics Test Kit				Concentration: _____ ppm																																											
<table border="1" style="width:100%; border-collapse: collapse;"> <tr> <th>Range Used:</th> <th>Range</th> <th>Method</th> <th>Concentration ppm</th> </tr> <tr> <td><input type="checkbox"/></td> <td>0 to 1 ppm</td> <td>K-7510</td> <td></td> </tr> <tr> <td><input type="checkbox"/></td> <td>1 to 12 ppm</td> <td>K-7512</td> <td></td> </tr> </table>				Range Used:	Range	Method	Concentration ppm	<input type="checkbox"/>	0 to 1 ppm	K-7510		<input type="checkbox"/>	1 to 12 ppm	K-7512		Analysis Time: _____																															
Range Used:	Range	Method	Concentration ppm																																												
<input type="checkbox"/>	0 to 1 ppm	K-7510																																													
<input type="checkbox"/>	1 to 12 ppm	K-7512																																													
Equipment: HACH Digital Titrator OX-DT				Analysis Time: _____																																											
<table border="1" style="width:100%; border-collapse: collapse;"> <tr> <th>Range Used:</th> <th>Range</th> <th>Sample Vol.</th> <th>Cartridge</th> <th>Multiplier</th> </tr> <tr> <td><input type="checkbox"/></td> <td>1-5 mg/L</td> <td>200 ml</td> <td>0.200 N</td> <td>0.01</td> </tr> <tr> <td><input type="checkbox"/></td> <td>2-10 mg/L</td> <td>100 ml</td> <td>0.200 N</td> <td>0.02</td> </tr> </table>				Range Used:	Range	Sample Vol.	Cartridge	Multiplier	<input type="checkbox"/>	1-5 mg/L	200 ml	0.200 N	0.01	<input type="checkbox"/>	2-10 mg/L	100 ml	0.200 N	0.02	<table border="1" style="width:100%; border-collapse: collapse;"> <tr> <th>Titration Count</th> <th>Multiplier</th> <th>Concentration</th> </tr> <tr> <td>_____</td> <td>x 0.01</td> <td>= _____ mg/L</td> </tr> <tr> <td>_____</td> <td>x 0.02</td> <td>= _____ mg/L</td> </tr> </table>				Titration Count	Multiplier	Concentration	_____	x 0.01	= _____ mg/L	_____	x 0.02	= _____ mg/L																
Range Used:	Range	Sample Vol.	Cartridge	Multiplier																																											
<input type="checkbox"/>	1-5 mg/L	200 ml	0.200 N	0.01																																											
<input type="checkbox"/>	2-10 mg/L	100 ml	0.200 N	0.02																																											
Titration Count	Multiplier	Concentration																																													
_____	x 0.01	= _____ mg/L																																													
_____	x 0.02	= _____ mg/L																																													
Notes: _____																																															
Carbon Dioxide:																																															
Equipment: Chemetrics Test Kit				Concentration: _____ ppm																																											
<table border="1" style="width:100%; border-collapse: collapse;"> <tr> <th>Range Used:</th> <th>Range</th> <th>Method</th> <th>Concentration ppm</th> </tr> <tr> <td><input type="checkbox"/></td> <td>10 to 100 ppm</td> <td>K-1910</td> <td></td> </tr> <tr> <td><input type="checkbox"/></td> <td>100 to 1000 ppm</td> <td>K-1920</td> <td></td> </tr> <tr> <td><input type="checkbox"/></td> <td>250 to 2500 ppm</td> <td>K-1925</td> <td></td> </tr> </table>				Range Used:	Range	Method	Concentration ppm	<input type="checkbox"/>	10 to 100 ppm	K-1910		<input type="checkbox"/>	100 to 1000 ppm	K-1920		<input type="checkbox"/>	250 to 2500 ppm	K-1925		Analysis Time: _____																											
Range Used:	Range	Method	Concentration ppm																																												
<input type="checkbox"/>	10 to 100 ppm	K-1910																																													
<input type="checkbox"/>	100 to 1000 ppm	K-1920																																													
<input type="checkbox"/>	250 to 2500 ppm	K-1925																																													
Equipment: HACH Digital Titrator CA-DT				Analysis Time: _____																																											
<table border="1" style="width:100%; border-collapse: collapse;"> <tr> <th>Range Used:</th> <th>Range</th> <th>Sample Vol.</th> <th>Cartridge</th> <th>Multiplier</th> </tr> <tr> <td><input type="checkbox"/></td> <td>10-50 mg/L</td> <td>200 ml</td> <td>0.3636 N</td> <td>0.1</td> </tr> <tr> <td><input type="checkbox"/></td> <td>20-100 mg/L</td> <td>100 ml</td> <td>0.3636 N</td> <td>0.2</td> </tr> <tr> <td><input type="checkbox"/></td> <td>100-400 mg/L</td> <td>200 ml</td> <td>3.636 N</td> <td>1.0</td> </tr> <tr> <td><input type="checkbox"/></td> <td>200-1000 mg/L</td> <td>100 ml</td> <td>3.636 N</td> <td>2.0</td> </tr> </table>				Range Used:	Range	Sample Vol.	Cartridge	Multiplier	<input type="checkbox"/>	10-50 mg/L	200 ml	0.3636 N	0.1	<input type="checkbox"/>	20-100 mg/L	100 ml	0.3636 N	0.2	<input type="checkbox"/>	100-400 mg/L	200 ml	3.636 N	1.0	<input type="checkbox"/>	200-1000 mg/L	100 ml	3.636 N	2.0	<table border="1" style="width:100%; border-collapse: collapse;"> <tr> <th>Titration Count</th> <th>Multiplier</th> <th>Concentration</th> </tr> <tr> <td>_____</td> <td>x 0.1</td> <td>= _____ mg/L</td> </tr> <tr> <td>_____</td> <td>x 0.2</td> <td>= _____ mg/L</td> </tr> <tr> <td>_____</td> <td>x 1.0</td> <td>= _____ mg/L</td> </tr> <tr> <td>_____</td> <td>x 2.0</td> <td>= _____ mg/L</td> </tr> </table>				Titration Count	Multiplier	Concentration	_____	x 0.1	= _____ mg/L	_____	x 0.2	= _____ mg/L	_____	x 1.0	= _____ mg/L	_____	x 2.0	= _____ mg/L
Range Used:	Range	Sample Vol.	Cartridge	Multiplier																																											
<input type="checkbox"/>	10-50 mg/L	200 ml	0.3636 N	0.1																																											
<input type="checkbox"/>	20-100 mg/L	100 ml	0.3636 N	0.2																																											
<input type="checkbox"/>	100-400 mg/L	200 ml	3.636 N	1.0																																											
<input type="checkbox"/>	200-1000 mg/L	100 ml	3.636 N	2.0																																											
Titration Count	Multiplier	Concentration																																													
_____	x 0.1	= _____ mg/L																																													
_____	x 0.2	= _____ mg/L																																													
_____	x 1.0	= _____ mg/L																																													
_____	x 2.0	= _____ mg/L																																													
Standard Additions: <input type="checkbox"/> Titrant Molarity: _____ Digits Required: 1st: _____ 2nd: _____ 3rd: _____																																															
Notes: _____																																															
Hydrogen, dissolved																																															
Equipment: Bubble strip sampling field method																																															
Start stripper at _____ (time)																																															
End stripper at _____ (time)																																															
Total stripper time _____																																															
Pump rate _____ milliliters/minute																																															

Subject NATURAL ATTENUATION PARAMETER COLLECTION	Number SA-1.6	Page 21 of 21
	Revision 1	Effective Date 09/03

ATTACHMENT F

**FIELD ANALYTICAL LOG SHEET, GEOCHEMICAL PARAMETERS
PAGE 3 OF 3**

Note: Analyte, method, and/or equipment may be deleted from form if not being performed.



**FIELD ANALYTICAL LOG SHEET
GEOCHEMICAL PARAMETERS**

Tetra Tech NUS, Inc.

Page of

Project Site Name: _____		Sample ID No.: _____	
Project No.: _____		Sample Location: _____	
Sampled By: _____		Duplicate: <input type="checkbox"/>	
Field Analyst: _____		Blank: <input type="checkbox"/>	
Sulfate (SO₄²⁻):			
Equipment	DR-850	DR-8 __	Range: 0 - 70 mg/L
Concentration:	_____ ppm		
Program/Module:	_____ 91		
Analysis Time:	_____		
Standard Solution:	<input type="checkbox"/>	Results: _____	Filtered: <input type="checkbox"/>
Standard Additions:	<input type="checkbox"/>	Digits Required: 0.1ml: _____ 0.2ml: _____ 0.3ml: _____	
Notes: _____			
Nitrate (NO₃⁻-N):			
Equipment	DR-850	DR-8 __	Range: 0 - 0.50 mg/L ⁽¹⁾
Concentration:	_____ ppm		
Program/Module:	_____ 55		
Analysis Time:	_____		
Filtered:	<input type="checkbox"/>		
Standard Solution:	<input type="checkbox"/>	Results: _____	Nitrite Interference Treatment: <input type="checkbox"/>
Standard Additions:	<input type="checkbox"/>	Digits Required: 0.1ml: _____ 0.2ml: _____ 0.3ml: _____	Reagent Blank Correction: <input type="checkbox"/>
Alternate forms: NO ₂ _____ NaNO ₂ _____ mg/L			
Notes (1): If results are over limit use dilution method at step 3, 5ml sample 10ml DI result X3, range upto 1.5mg/L			
Notes: _____			
Nitrite (NO₂⁻-N):			
Equipment	DR-850	DR-8 __	Range: 0 - 0.350 mg/L
Concentration:	_____ ppm		
Program/Module:	_____ 62		
Analysis Time:	_____		
Filtered:	<input type="checkbox"/>		
Standard Solution:	<input type="checkbox"/>	Results: _____	Reagent Blank Correction: <input type="checkbox"/>
Notes: _____			
Manganese (Mn²⁺):			
Equipment	DR-850	DR-8 __	Range: 0 - 20.0 mg/L
Concentration:	_____ ppm		
Program/Module:	_____ 525nm 41		
Analysis Time:	_____		
Filtered:	<input type="checkbox"/>		
Standard Solution:	<input type="checkbox"/>	Results: _____	Digestion: <input type="checkbox"/>
Standard Additions:	<input type="checkbox"/>	Digits Required: 0.1ml: _____ 0.2ml: _____ 0.3ml: _____	Reagent Blank Correction: <input type="checkbox"/>
Notes: _____			
QA/QC Checklist:			
All data fields have been completed as necessary: <input type="checkbox"/>			
Correct measurement units are cited in the SAMPLING DATA block: <input type="checkbox"/>			
Values cited in the SAMPLING DATA block are consistent with the Groundwater Sample Log Sheet: <input type="checkbox"/>			
Multiplication is correct for each Multiplier table: <input type="checkbox"/>			
Final calculated concentration is within the appropriate Range Used block: <input type="checkbox"/>			
Alkalinity Relationship is determined appropriately as per manufacturer (HACH) instructions: <input type="checkbox"/>			
QA/QC sample (e.g., Std. Additions, etc.) frequency is appropriate as per the project planning documents: <input type="checkbox"/>			
Nitrite Interference treatment was used for Nitrate test if Nitrite was detected: <input type="checkbox"/>			
Title block on each page of form is initialized by person who performed this QA/QC Checklist: <input type="checkbox"/>			



TETRA TECH NUS, INC.

STANDARD OPERATING PROCEDURES

Number	SA-2.5	Page	1 of 6
Effective Date	09/03	Revision	3
Applicability	Tetra Tech NUS, Inc.		
Prepared	Earth Sciences Department		
Approved	D. Senovich <i>DS</i>		

Subject DIRECT PUSH TECHNOLOGY (GEOPROBE®/HYDROPUNCH™)

TABLE OF CONTENTS

<u>SECTION</u>	<u>PAGE</u>
1.0 PURPOSE	2
2.0 SCOPE	2
3.0 GLOSSARY	2
4.0 RESPONSIBILITIES	2
5.0 SOIL SAMPLING PROCEDURES.....	3
5.1 GENERAL	3
5.2 SAMPLING EQUIPMENT	3
5.3 DPT SAMPLING METHODOLOGY	3
6.0 GROUNDWATER SAMPLING PROCEDURES.....	4
6.1 GENERAL	4
6.2 SAMPLING EQUIPMENT	4
6.3 DPT TEMPORARY WELL POINT INSTALLATION AND SAMPLING METHODOLOGY	5
7.0 RECORDS.....	5
 <u>ATTACHMENTS</u>	
1 SAFE WORK PERMIT	6

Subject DIRECT PUSH TECHNOLOGY (GEOPROBE®/HYDROPUNCH™)	Number SA-2.5	Page 2 of 6
	Revision 3	Effective Date 09/03

1.0 PURPOSE

The purpose of this procedure is to provide general reference information on Direct Push Technology (DPT). DPT is designed to collect soil, groundwater, and soil gas samples without using conventional drilling techniques. The advantage of using DPT over conventional drilling includes the generation of little or no drill cuttings, sampling in locations with difficult accessibility, reduced overhead clearance requirements, no fluid introduction during probing, and typical lower costs per sample than with conventional techniques. Disadvantages include a maximum penetration depth of approximately 15 to 40 feet in dense soils (although it may be as much as 60 to 80 feet in certain types of geological environments), reduced capability of obtaining accurate water-level measurements, and the inability to install permanent groundwater monitoring wells. The methods and equipment described herein are for collection of surface and subsurface soil samples and groundwater samples. Soil gas sampling is discussed in SOP SA-2.4.

2.0 SCOPE

This procedure provides information on proper sampling equipment and techniques for DPT. Review of the information contained herein will facilitate planning of the field sampling effort by describing standard sampling techniques. The techniques described shall be followed whenever applicable, noting that site-specific conditions or project-specific plans may require adjustments in methodology.

3.0 GLOSSARY

Direct Push Technology (DPT) - DPT refers to sampling tools and sensors that are driven directly into the ground without the use of conventional drilling equipment. DPT typically utilizes hydraulic pressure and/or percussion hammers to advance the sampling tools. A primary advantage of DPT over conventional drilling techniques is that DPT results in the generation of little or no investigation derived waste.

Geoprobe® - Geoprobe® is a manufacturer of a hydraulically-powered, percussion/probing machines utilizing DPT to collect subsurface environmental samples. Geoprobe® relies on a relatively small amount of static weight (vehicle) combined with percussion as the energy for advancement of a tool string. The Geoprobe® equipment can be mounted in a multitude of vehicles for access to all types of environmental sites.

HydroPunch™ - HydroPunch™ is a manufacturer of stainless steel and Teflon® sampling tools that are capable of collecting representative groundwater and/or soil samples without requiring the installation of a groundwater monitoring well or conventional soil boring. HydroPunch™ is an example of DPT sampling equipment.

Flame Ionization Detector (FID) - A portable instrument for the measurement of many combustible organic compounds and a few inorganic compounds in air at parts-per million levels. The basis for the detection is the ionization of gaseous species utilizing a flame as the energizing source.

Photo Ionization Detector (PID) - A portable instrument for the measurement of many combustible organic compounds and a few inorganic compounds in air at parts-per million levels. The basis for the detection is the ionization of gaseous species utilizing ultraviolet radiation as the energizing source.

4.0 RESPONSIBILITIES

Project Manager - The Project Manager is responsible for selecting and/or reviewing the appropriate DPT drilling procedure required to support the project objectives.

Subject DIRECT PUSH TECHNOLOGY (GEOPROBE®/HYDROPUNCH™)	Number SA-2.5	Page 3 of 6
	Revision 3	Effective Date 09/03

Field Operations Leader (FOL)- The FOL is primarily responsible for performing the DPT in accordance with the project-specific plan.

5.0 SOIL SAMPLING PROCEDURES

5.1 General

The common methodology for the investigation of the vadose zone is soil boring drilling and soil sampling. However, drilling soil borings can be very expensive. Generally the advantage of DPT for subsurface soil sampling is the reduced cost of disposal of drilling cuttings and shorter sampling times.

5.2 Sampling Equipment

Equipment needed for conducting DPT drilling for subsurface soil sampling includes, but is not limited to, the following:

- Geoprobe® Sampling Kit
- Cut-resistant gloves
- 4-foot x 1.5-inch diameter macrocore sampler
- Probe sampling adapters
- Roto-hammer with 1.5-inch bit
- Disposable acetate liners for soil macrocore sampler
- Cast aluminum or steel drive points
- Geoprobe® AT-660 Series Large Bore Soil Sampler, or equivalent
- Standard decontamination equipment and solutions

For health and safety equipment and procedures, follow the direction provided in the Safe Work Permit in Attachment 1, or the more detailed directions provided in the project's Health and Safety Plan.

5.3 DPT Sampling Methodology

There are several methods for the collection of soil samples using DPT drilling. The most common method is discussed in the following section. Variations of the following method may be conducted upon approval of the Project Manager in accordance with the project-specific plan.

- Macrocore samplers fitted with detachable aluminum or steel drive points are driven into the ground using hydraulic pressure. If there is concrete or pavement over a sampling location, a Roto-hammer is used to drill a minimum 1.5-inch diameter hole through the surface material. A Roto-hammer may also be used if very dense soils are encountered.
- The sampler is advanced continuously in 4-foot intervals or less if desired. No soil cuttings are generated because the soil which is not collected in the sampler is displaced within the formation.
- The sampler is retracted from the hole, and the 4-foot continuous sample is removed from the outer coring tube. The sample is contained within an inner acetate liner.
- Attach the metal trough from the Geoprobe® Sampling Kit firmly to the tail gate of a vehicle. If a vehicle with a tail gate is not available, secure the trough on another suitable surface.
- Place the acetate liner containing the soils in the trough.

Subject DIRECT PUSH TECHNOLOGY (GEOPROBE®/HYDROPUNCH™)	Number SA-2.5	Page 4 of 6
	Revision 3	Effective Date 09/03

- While wearing cut-resistant gloves (constructed of leather or other suitable material), cut the acetate liner through its entire length using the double-bladed knife that accompanies the Geoprobe® Sampling Kit. Then remove the strip of acetate from the trough to gain access to the collected soils. Do not attempt to cut the acetate liner while holding it in your hand.
- Field screen the sample with an FID or PID, and observe/examine the sample (according to SOP GH-1.3). If appropriate, transfer the sample to sample bottles for laboratory analysis. If additional volume is required, push an additional boring adjacent to the first and composite/mix the same interval. Field compositing is usually not acceptable for sample requiring volatile organics analysis.
- Once sampling has been completed, the hole is backfilled with bentonite chips or bentonite cement grout, depending upon project requirements. Asphalt or concrete patch is used to cap holes through paved or concrete areas. All holes should be finished smooth to existing grade.
- In the event the direct push van/truck cannot be driven to a remote location or a sampling location with difficult accessibility, sampling probes may be advanced and sampled manually or with air/electric operated equipment (e.g., jack hammer).
- Sampling equipment is decontaminated prior to collecting the next sample.

6.0 GROUNDWATER SAMPLING PROCEDURES

6.1 General

The most common methodology for the investigation of groundwater is the installation and sampling of permanent monitoring wells. If only groundwater screening is required, the installation and sampling of temporary well points may be performed. The advantage of temporary well point installation using DPT is reduced cost due to no or minimal disposal of drilling cuttings and well construction materials, and shorter installation/times sampling.

Two disadvantages of DPT drilling for well point installation are:

- In aquifers with low yields, well points may have to be sampled without purging or development.
- If volume requirements are high, this method can be time consuming for low yield aquifers.

6.2 Sampling Equipment

Equipment needed for temporary well installation and sampling using DPT includes, but is not limited, to the following:

- 2-foot x 1-inch diameter mill-slotted (0.005 to 0.02-inch) well point
- Connecting rods
- Roto-hammer with 1.5-inch bit
- Mechanical jack
- 1/4-inch OD polyethylene tubing
- 3/8-inch OD polyethylene tubing
- Peristaltic pump
- Standard decontamination equipment and solutions

Subject DIRECT PUSH TECHNOLOGY (GEOPROBE®/HYDROPUNCH™)	Number SA-2.5	Page 5 of 6
	Revision 3	Effective Date 09/03

6.3 DPT Temporary Well Point Installation and Sampling Methodology

There are several methods for the installation and sampling of temporary well points using DPT. The most common methodology is discussed below. Variations of the following method may be conducted upon approval of the Project Manager in accordance with the project specific plan.

- A 2-foot x 1-inch diameter mill-slotted (0.005 to 0.02-inch) well point attached to connecting rods is driven into the ground to the desired depth using a rotary electric hammer or other direct push drill rig. If there is concrete or pavement over a sampling location, a Roto-hammer or electric coring machine is used to drill a hole through the surface material.
- The well point will be allowed to equilibrate for at least 15 minutes, after which a measurement of the static water level will be taken. The initial measurement of the water level will be used to assess the amount of water which is present in the well point and to determine the amount of silt and sand infiltration that may have occurred.
- The well point will be developed using a peristaltic pump and polyethylene tubing to remove silt and sand which may have entered the well point. The well point is developed by inserting polyethylene tubing to the bottom of the well point and lifting and lowering the tubing slightly while the pump is operating. The pump will be operated at a maximum rate of approximately 2 liters per minute. After removal of sediment from the bottom of the well point, the well point will be vigorously pumped at maximum capacity until discharge water is visibly clear and no further sediments are being generated. Measurements of pH, specific conductance, temperature, and turbidity shall be recorded every 5 to 10 minutes during the purging process. After two consistent readings of pH, specific conductance, temperature and turbidity (± 10 percent), the well may be sampled.
- A sample will be collected using the peristaltic pump set at the same or reduced speed as during well development. Samples (with the exception of the samples to be analyzed for volatile organic compounds, VOCs) will be collected directly from the pump discharge. Sample containers for VOCs will be filled by (first shutting off the pump) crimping the discharge end of the sample tubing when filled, removing the inlet end of the sample tubing from the well, suspending the inlet tubing above the vial, and allowing water to fill each vial by gravity flow.
- Once the groundwater sample has been collected, the connecting rods and well point will be removed from the hole with the direct push rig hydraulics. The hole will be backfilled with bentonite chips or bentonite cement grout, depending upon project requirements. Asphalt or concrete patch will be used to cap holes through paved or concrete areas. All holes will be finished smooth to existing grade.
- In the event the direct push van/truck cannot be driven to a remote location or sampling location with difficult accessibility, sampling probes may be advanced and sampled manually or with air/electric-operated equipment (e.g., jack hammer).
- Decontaminate the equipment before moving to the next location.

7.0 RECORDS

A record of all field procedures, tests, and observations must be recorded in the field logbook, boring logs, and sample log sheets, as needed. Entries should include all pertinent data regarding the investigation. The use of sketches and field landmarks will help to supplement the investigation and evaluation.

Subject DIRECT PUSH TECHNOLOGY (GEOPROBE®/HYDROPUNCH™)	Number SA-2.5	Page 6 of 6
	Revision 3	Effective Date 09/03

**ATTACHMENT 1
SAFE WORK PERMIT FOR DPT OPERATIONS**

Permit No. _____ Date: _____ Time: From _____ to _____

SECTION I: General Job Scope

- I. Work limited to the following (description, area, equipment used): **Monitoring well drilling and installation through direct push technology**
- II. Required Monitoring Instruments: _____
- III. Field Crew: _____
- IV. On-site Inspection conducted Yes No Initials of Inspector TtNUS

SECTION II: General Safety Requirements (To be filled in by permit issuer)

- V. Protective equipment required
 - Level D Level B
 - Level C Level A
 - Detailed on Reverse
- Respiratory equipment required
 - Full face APR
 - Half face APR
 - SKA-PAC SAR
 - Skid Rig
- Escape Pack
- SCBA
- Bottle Trailer
- None

Level D Minimum Requirements: Sleeved shirt and long pants, safety footwear, and work gloves. Safety glasses, hard hats, and hearing protection will be worn when working near or sampling in the vicinity of the DPT rig.

Modifications/Exceptions.

VI. Chemicals of Concern	Action Level(s)	Response Measures
_____	_____	_____

VII. Additional Safety Equipment/Procedures

- | | |
|---|--|
| Hard-hat <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No | Hearing Protection (Plugs/Muffs) <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No |
| Safety Glasses <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No | Safety belt/harness <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No |
| Chemical/splash goggles <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No | Radio <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No |
| Splash Shield <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No | Barricades <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No |
| Splash suits/coveralls <input type="checkbox"/> Yes <input type="checkbox"/> No | Gloves (Type - _____) <input type="checkbox"/> Yes <input type="checkbox"/> No |
| Steel toe Work shoes or boots <input checked="" type="checkbox"/> Yes <input type="checkbox"/> No | Work/warming regimen <input type="checkbox"/> Yes <input type="checkbox"/> No |

Modifications/Exceptions: Reflective vests for high traffic areas.

VIII. Procedure review with permit acceptors	Yes	NA	Yes	NA
Safety shower/eyewash (Location & Use).....	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Daily tail gate meetings.....	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Contractor tools/equipment/PPE inspected	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Emergency alarms	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Evacuation routes	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Assembly points	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>

IX. Site Preparation

- Utility Clearances obtained for areas of subsurface investigation Yes No
- Physical hazards removed or blockaded Yes No
- Site control boundaries demarcated/signage Yes No

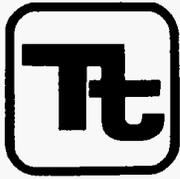
X. Equipment Preparation

- | | | |
|---|------------------------------|--|
| Equipment drained/depressurized | <input type="checkbox"/> Yes | <input checked="" type="checkbox"/> NA |
| Equipment purged/cleaned | <input type="checkbox"/> Yes | <input checked="" type="checkbox"/> NA |
| Isolation checklist completed | <input type="checkbox"/> Yes | <input checked="" type="checkbox"/> NA |
| Electrical lockout required/field switch tested | <input type="checkbox"/> Yes | <input checked="" type="checkbox"/> NA |
| Blinds/misalignments/blocks & bleeds in place | <input type="checkbox"/> Yes | <input checked="" type="checkbox"/> NA |
| Hazardous materials on walls/behind liners considered | <input type="checkbox"/> Yes | <input checked="" type="checkbox"/> NA |

- XI. Additional Permits required (Hot work, confined space entry). Yes No
If yes, complete permit required or contact Health Sciences, Pittsburgh Office

XII. Special instructions, precautions:

Permit Issued by: _____ Permit Accepted by: _____



TETRA TECH NUS, INC.

STANDARD OPERATING PROCEDURES

Number	SA-6.1	Page	1 of 11
Effective Date	02/04	Revision	3
Applicability	Tetra Tech NUS, Inc.		
Prepared	Earth Sciences Department		
Approved	D. Senovich <i>[Signature]</i>		

Subject
NON-RADIOLOGICAL SAMPLE HANDLING

TABLE OF CONTENTS

<u>SECTION</u>	<u>PAGE</u>
1.0 PURPOSE.....	2
2.0 SCOPE.....	2
3.0 GLOSSARY	2
4.0 RESPONSIBILITIES.....	3
5.0 PROCEDURES.....	3
5.1 SAMPLE CONTAINERS.....	3
5.2 SAMPLE PRESERVATION.....	3
5.2.1 Overview	4
5.2.2 Preparation and Addition of Reagents	4
5.3 FIELD FILTRATION.....	5
5.4 SAMPLE PACKAGING AND SHIPPING.....	6
5.4.1 Environmental Samples	6
6.0 REFERENCES.....	7
 <u>ATTACHMENTS</u>	
A GENERAL SAMPLE CONTAINER AND PRESERVATION REQUIREMENTS.....	8
B ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES.....	9

Subject NON-RADIOLOGICAL SAMPLE HANDLING	Number SA-6.1	Page 2 of 11
	Revision 3	Effective Date 02/04

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide information on sample preservation, packaging, and shipping procedures to be used in handling environmental samples submitted for chemical constituent, biological, or geotechnical analysis. Sample chain-of-custody procedures and other aspects of field documentation are addressed in SOP SA-6.3. Sample identification is addressed in SOP CT-04.

2.0 SCOPE

This procedure describes the appropriate containers to be used for samples depending on the analyses to be performed, and the steps necessary to preserve the samples when shipped off site for chemical analysis.

3.0 GLOSSARY

Hazardous Material - A substance or material which has been determined by the Secretary of Transportation to be capable of posing an unreasonable risk to health, safety, and property when transported in commerce, and which has been so designated. Under 49 CFR, the term includes hazardous substances, hazardous wastes, marine pollutants, and elevated temperature materials, as well as materials designated as hazardous under the provisions of §172.101 and §172.102 and materials that meet the defining criteria for hazard classes and divisions in Part 173. With slight modifications, IATA has adopted DOT "hazardous materials" as IATA "Dangerous Goods."

Hazardous Waste - Any substance listed in 40 CFR, Subpart D (y261.30 et seq.), or otherwise characterized as ignitable, corrosive, reactive, or toxic (as defined by Toxicity Characteristic Leaching Procedure, TCLP, analysis) as specified under 40 CFR, Subpart C (y261.20 et seq.), that would be subject to manifest requirements specified in 40 CFR 262. Such substances are defined and regulated by EPA.

Marking - A descriptive name, identification number, instructions, cautions, weight, specification or UN marks, or combination thereof required on outer packaging of hazardous materials.

n.o.i - Not otherwise indicated (may be used interchangeably with n.o.s.).

n.o.s. - Not otherwise specified.

Packaging - A receptacle and any other components or materials necessary for compliance with the minimum packaging requirements of 49 CFR 174, including containers (other than freight containers or overpacks), portable tanks, cargo tanks, tank cars, and multi-unit tank-car tanks to perform a containment function in conformance with the minimum packaging requirements of 49 CFR 173.24(a) & (b).

Placard - Color-coded, pictorial sign which depicts the hazard class symbol and name and which is placed on the side of a vehicle transporting certain hazardous materials.

Common Preservatives:

- Hydrochloric Acid - HCl
- Sulfuric Acid - H₂SO₄
- Nitric Acid - HNO₃
- Sodium Hydroxide - NaOH

Subject NON-RADIOLOGICAL SAMPLE HANDLING	Number SA-6.1	Page 3 of 11
	Revision 3	Effective Date 02/04

Other Preservatives

- Zinc Acetate
- Sodium Thiosulfate - Na₂S₂O₃

Normality (N) - Concentration of a solution expressed as equivalent per liter, an equivalent being the amount of a substance containing 1 gram-atom of replaceable hydrogen or its equivalent.

Reportable Quantity (RQ) - For the purposes of this SOP, means the quantity specified in column 3 of the Appendix to DOT 49 CFR §172.101 for any material identified in column 1 of the appendix. A spill greater than the amount specified must be reported to the National Response Center.

Sample - A sample is physical evidence collected from a facility or the environment, which is representative of conditions at the location and time of collection.

4.0 RESPONSIBILITIES

Field Operations Leader - Directly responsible for the bottling, preservation, labeling, packaging, shipping, and custody of samples up to and including release to the shipper.

Field Samplers - Responsible for initiating the Chain-of-Custody Record (per SOP SA-6.3), implementing the packaging and shipping requirements, and maintaining custody of samples until they are relinquished to another custodian or to the shipper.

5.0 PROCEDURES

Sample identification, labeling, documentation, and chain-of-custody are addressed by SOP SA-6.3.

5.1 Sample Containers

Different types of chemicals react differently with sample containers made of various materials. For example, trace metals adsorb more strongly to glass than to plastic, whereas many organic chemicals may dissolve various types of plastic containers. Attachments A and B show proper containers (as well as other information) per 40 CFR 136. In general, the sample container shall allow approximately 5-10 percent air space ("ullage") to allow for expansion/vaporization if the sample warms during transport. However, for collection of volatile organic compounds, head space shall be omitted. The analytical laboratory will generally provide certified-clean containers for samples to be analyzed for chemical constituents. Shelby tubes or other sample containers are generally provided by the driller for samples requiring geotechnical analysis. Sufficient lead time shall be allowed for a delivery of sample container orders. Therefore, it is critical to use the correct container to maintain the integrity of the sample prior to analysis.

Once opened, the container must be used at once for storage of a particular sample. Unused but opened containers are to be considered contaminated and must be discarded. Because of the potential for introduction of contamination, they cannot be reclosed and saved for later use. Likewise, any unused containers which appear contaminated upon receipt, or which are found to have loose caps or a missing Teflon liner (if required for the container), shall be discarded.

5.2 Sample Preservation

Many water and soil samples are unstable and therefore require preservation to prevent changes in either the concentration or the physical condition of the constituent(s) requiring analysis. Although complete and irreversible preservation of samples is not possible, preservation does retard the chemical and biological

Subject NON-RADIOLOGICAL SAMPLE HANDLING	Number SA-6.1	Page 4 of 11
	Revision 3	Effective Date 02/04

changes that inevitably take place after the sample is collected. Preservation techniques are usually limited to pH control, chemical addition(s), and refrigeration/ freezing (certain biological samples only).

5.2.1 Overview

The preservation techniques to be used for various analytes are listed in Attachments A and B. Reagents required for sample preservation will either be added to the sample containers by the laboratory prior to their shipment to the field or be added in the field (in a clean environment). Only high purity reagents shall be used for preservation. In general, aqueous samples of low-concentration organics (or soil samples of low- or medium-concentration organics) are cooled to 4°C. Medium-concentration aqueous samples, high-hazard organic samples, and some gas samples are typically not preserved. Low-concentration aqueous samples for metals are acidified with HNO₃, whereas medium-concentration and high-hazard aqueous metal samples are not preserved. Low- or medium-concentration soil samples for metals are cooled to 4°C, whereas high-hazard samples are not cooled.

The following subsections describe the procedures for preparing and adding chemical preservatives. Attachments A and B indicate the specific analytes which require these preservatives.

The FOL is responsible for ensuring that an accurate Chemical Inventory is created and maintained for all hazardous chemicals brought to the work site (see Section 5 of the TtNUS Health and Safety Guidance Manual). Furthermore, the FOL must ensure that a corresponding Material Safety Data Sheet (MSDS) is collected for every substance entered on the site Chemical Inventory, and that all persons using/handling/ disposing of these substances review the appropriate MSDS for substances they will work with. The Chemical Inventory and the MSDSs must be maintained at each work site in a location and manner where they are readily-accessible to all personnel.

5.2.2 Preparation and Addition of Reagents

Addition of the following acids or bases may be specified for sample preservation; these reagents shall be analytical reagent (AR) grade or purer and shall be diluted to the required concentration with deionized water before field sampling commences. To avoid uncontrolled reactions, be sure to Add Acid to water (not vice versa). A dilutions guide is provided below.

Acid/Base	Dilution	Concentration	Estimated Amount Required for Preservation
Hydrochloric Acid (HCl)	1 part concentrated HCl: 1 part double-distilled, deionized water	6N	5-10 mL
Sulfuric Acid (H ₂ SO ₄)	1 part concentrated H ₂ SO ₄ : 1 part double-distilled, deionized water	18N	2 - 5 mL
Nitric Acid (HNO ₃)	Undiluted concentrated HNO ₃	16N	2 - 5 mL
Sodium Hydroxide (NaOH)	400 grams solid NaOH dissolved in 870 mL double-distilled, deionized water; yields 1 liter of solution	10N	2 mL

The amounts required for preservation shown in the above table assumes proper preparation of the preservative and addition of the preservative to one liter of aqueous sample. This assumes that the sample is initially at pH 7, is poorly buffered, and does not contain particulate matter; as these conditions vary, more preservative may be required. Consequently, the final sample pH must be checked using narrow-range pH paper, as described in the generalized procedure detailed below:

Subject NON-RADIOLOGICAL SAMPLE HANDLING	Number SA-6.1	Page 5 of 11
	Revision 3	Effective Date 02/04

- Pour off 5-10 mL of sample into a dedicated, clean container. Use some of this sample to check the initial sample pH using wide range (0-14) pH paper. Never dip the pH paper into the sample; always apply a drop of sample to the pH paper using a clean stirring rod or pipette.
- Add about one-half of the estimated preservative required to the original sample bottle. Cap and invert gently several times to mix. Check pH (as described above) using medium range pH paper (pH 0-6 or pH 7.5-14, as applicable).
- Cap sample bottle and seal securely.

Additional considerations are discussed below:

- To test if ascorbic acid must be used to remove oxidizing agents present in the sample before it can be properly preserved, place a drop of sample on KI-starch paper. A blue color indicates the need for ascorbic acid addition.

If required, add a few crystals of ascorbic acid to the sample and retest with the KI-starch paper. Repeat until a drop of sample produces no color on the KI-starch paper. Then add an additional 0.6 grams of ascorbic acid per each liter of sample volume.

Continue with proper base preservation of the sample as described above.

- Samples for sulfide analysis must be treated by the addition of 4 drops (0.2 mL) of 2N zinc acetate solution per 100 ml of sample.

The 2N zinc acetate solution is made by dissolving 220 grams of zinc acetate in 870 mL of double-distilled, deionized water to make 1 liter of solution.

The sample pH is then raised to 9 using the NaOH preservative.

- Sodium thiosulfate must be added to remove residual chlorine from a sample. To test the sample for residual chlorine use a field test kit specially made for this purpose.

If residual chlorine is present, add 0.08 grams of sodium thiosulfate per liter of sample to remove the residual chlorine.

Continue with proper acidification of the sample as described above.

For biological samples, 10% buffered formalin or isopropanol may also be required for preservation. Questions regarding preservation requirements should be resolved through communication with the laboratory before sampling begins.

5.3 Field Filtration

At times, field-filtration may be required to provide for the analysis of dissolved chemical constituents. Field-filtration must be performed prior to the preservation of samples as described above. General procedures for field filtration are described below:

- The sample shall be filtered through a non-metallic, 0.45-micron membrane filter, immediately after collection. The filtration system shall consist of dedicated filter canister, dedicated tubing, and a peristaltic pump with pressure or vacuum pumping squeeze action (since the sample is filtered by mechanical peristalsis, the sample travels only through the tubing).

Subject NON-RADIOLOGICAL SAMPLE HANDLING	Number SA-6.1	Page 6 of 11
	Revision 3	Effective Date 02/04

- To perform filtration, thread the tubing through the peristaltic pump head. Attach the filter canister to the discharge end of the silicon tubing (note flow direction arrow); attach the aqueous sample container to the intake end of the silicon tubing. Turn the peristaltic pump on and perform filtration. Run approximately 100 ml of sample through the filter and discard prior to sample collection.
- Continue by preserving the filtrate (contained in the filter canister), as applicable and generally described above.

5.4 **Sample Packaging and Shipping**

Only employees who have successfully completed the TtNUS "Shipping Hazardous Materials" training course are authorized to package and ship hazardous substances. These trained individuals are responsible for performing shipping duties in accordance with this training.

Samples collected for shipment from a site shall be classified as either environmental or hazardous material samples. Samples from drums containing materials other than Investigative Derived Waste (IDW) and samples obtained from waste piles or bulk storage tanks are generally shipped as hazardous materials. A distinction must be made between the two types of samples in order to:

- Determine appropriate procedures for transportation of samples (if there is any doubt, a sample shall be considered hazardous and shipped accordingly.)
- Protect the health and safety of transport and laboratory personnel receiving the samples (special precautions are used by the shipper and at laboratories when hazardous materials are received.)

Detailed procedures for packaging environmental samples are outlined in the remainder of this section.

5.4.1 **Environmental Samples**

Environmental samples are packaged as follows:

- Place properly identified sample container, with lid securely fastened, in a plastic bag (e.g. Ziploc baggie), and seal the bag.
- Place sample in a cooler constructed of sturdy material which has been lined with a large, plastic bag (e.g. "garbage" bag). Drain plugs on coolers must be taped shut.
- Pack with enough cushioning materials such as bubble wrap (shoulders of bottles must be iced if required) to minimize the possibility of the container breaking.
- If cooling is required (see Attachments A and B), place ice around sample container shoulders, and on top of packing material (minimum of 8 pounds of ice for a medium-size cooler).
- Seal (i.e., tape or tie top in knot) large liner bag.
- The original (top, signed copy) of the COC form shall be placed inside a large Ziploc-type bag and taped inside the lid of the shipping cooler. If multiple coolers are sent but are included on one COC form, the COC form should be sent with the cooler containing the vials for VOC analysis. The COC form should then state how many coolers are included with that shipment.
- Close and seal outside of cooler as described in SOP SA-6.3. Signed custody seals must be used.

Subject NON-RADIOLOGICAL SAMPLE HANDLING	Number SA-6.1	Page 7 of 11
	Revision 3	Effective Date 02/04

Coolers must be marked as containing "Environmental Samples." The appropriate side of the container must be marked "This End Up" and arrows placed appropriately. No DOT marking or labeling is required; there are no DOT restrictions on mode of transportation.

6.0 REFERENCES

American Public Health Association, 1981. Standard Methods for the Examination of Water and Wastewater, 15th Edition. APHA, Washington, D.C.

International Air Transport Association (latest issue). Dangerous Goods Regulations, Montreal, Quebec, Canada.

U.S. Department of Transportation (latest issue). Hazardous Materials Regulations, 49 CFR 171-177.

U.S. EPA, 1984. "Guidelines Establishing Test Procedures for the Analysis of Pollutants under Clean Water Act." Federal Register, Volume 49 (209), October 26, 1984, p. 43234.

U.S. EPA, 1979. Methods for Chemical Analysis of Water and Wastes. EPA-600/4-79-020, U.S. EPA-EMSL, Cincinnati, Ohio.

Subject NON-RADIOLOGICAL SAMPLE HANDLING	Number SA-6.1	Page 8 of 11
	Revision 3	Effective Date 02/04

ATTACHMENT A

GENERAL SAMPLE CONTAINER AND PRESERVATION REQUIREMENTS

Sample Type and Concentration	Container ⁽¹⁾	Sample Size	Preservation ⁽²⁾	Holding Time ⁽²⁾
-------------------------------	--------------------------	-------------	-----------------------------	-----------------------------

WATER

Organics (GC&GC/MS)	VOC	Low	Borosilicate glass	2 x 40 mL	Cool to 4°C HCl to ≤ 2	14 days ⁽⁹⁾
	Extractables SVOCs and pesticide/PCBs)	(Low	Amber glass	2x2 L or 4x1 L	Cool to 4°C	7 days to extraction; 40 days after extraction
	Extractables SVOCs and pesticide/PCBs)	(Medium	Amber glass	2x2 L or 4x1 L	None	7 days to extraction; 40 days after extraction
Inorganics	Metals	Low	High-density polyethylene	1 L	HNO ₃ to pH ≤ 2	6 months (Hg-28 days)
		Medium	Wide-mouth glass	16 oz.	None	6 months
	Cyanide	Low	High-density polyethylene	1 L	NaOH to pH>12	14 days
	Cyanide	Medium	Wide-mouth glass	16 oz.	None	14 days
Organic/ Inorganic	High Hazard		Wide-mouth glass	8 oz.	None	14 days

SOIL

Organics (GC&GC/MS)	VOC		EnCore Sampler	(3) 5 g Samplers	Cool to 4°C	48 hours to lab preservation
	Extractables SVOCs and pesticides/PCBs)	(Low	Wide-mouth glass	8 oz.	Cool to 4°C	14 days to extraction; 40 days after extraction
	Extractables SVOCs and pesticides/PCBs)	(Medium	Wide-mouth glass	8 oz.	Cool to 4°C	14 days to extraction; 40 days after extraction
Inorganics	Low/Medium		Wide-mouth glass	8 oz.	Cool to 4°C	6 months (Hg - 28 days) Cyanide (14 days)
Organic/Inorga nic	High Hazard		Wide-mouth glass	8 oz.	None	NA
Dioxin/Furan	All		Wide-mouth glass	4 oz.	None	35 days until extraction; 40 days after extraction
TCLP	All		Wide-mouth glass	8 oz.	None	7 days until preparation; analysis as per fraction

AIR

Volatile Organics	Low/Medium		Charcoal tube -- 7 cm long, 6 mm OD, 4 mm ID	100 L air	Cool to 4°C	5 days recommended
----------------------	------------	--	---	-----------	-------------	--------------------

1 All glass containers should have Teflon cap liners or septa.

2 See Attachment E. Preservation and maximum holding time allowances per 40 CFR 136.

Subject NON-RADIOLOGICAL SAMPLE HANDLING	Number SA-6.1	Page 9 of 11
	Revision 3	Effective Date 02/04

ATTACHMENT B

**ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES,
AND HOLDING TIMES**

Parameter Number/Name	Container ⁽¹⁾	Preservation ⁽²⁾⁽³⁾	Maximum Holding Time ⁽⁴⁾
-----------------------	--------------------------	--------------------------------	-------------------------------------

INORGANIC TESTS:

Acidity	P, G	Cool, 4°C	14 days
Alkalinity	P, G	Cool, 4°C	14 days
Ammonia - Nitrogen	P, G	Cool, 4°C; H ₂ SO ₄ to pH 2	28 days
Biochemical Oxygen Demand (BOD)	P, G	Cool, 4°C	48 hours
Bromide	P, G	None required	28 days
Chemical Oxygen Demand (COD)	P, G	Cool, 4°C; H ₂ SO ₄ to pH 2	28 days
Chloride	P, G	None required	28 days
Chlorine, Total Residual	P, G	None required	Analyze immediately
Color	P, G	Cool, 4°C	48 hours
Cyanide, Total and Amenable to Chlorination	P, G	Cool, 4°C; NaOH to pH 12; 0.6 g ascorbic acid ⁽⁵⁾	14 days ⁽⁶⁾
Fluoride	P	None required	28 days
Hardness	P, G	HNO ₃ to pH 2; H ₂ SO ₄ to pH 2	6 months
Total Kjeldahl and Organic Nitrogen	P, G	Cool, 4°C; H ₂ SO ₄ to pH 2	28 days
Nitrate - Nitrogen	P, G	None required	48 hours
Nitrate-Nitrite - Nitrogen	P, G	Cool, 4°C; H ₂ SO ₄ to pH 2	28 days
Nitrite - Nitrogen	P, G	Cool, 4°C	48 hours
Oil & Grease	G	Cool, 4°C; H ₂ SO ₄ to pH 2	28 days
Total Organic Carbon (TOC)	P, G	Cool, 4°C; HCl or H ₂ SO ₄ to pH 2	28 days
Orthophosphate	P, G	Filter immediately; Cool, 4°C	48 hours
Oxygen, Dissolved-Probe	G Bottle & top	None required	Analyze immediately
Oxygen, Dissolved-Winkler	G Bottle & top	Fix on site and store in dark	8 hours
Phenols	G	Cool, 4°C; H ₂ SO ₄ to pH 2	28 days
Phosphorus, Total	P, G	Cool, 4°C; H ₂ SO ₄ to pH 2	28 days
Residue, Total	P, G	Cool, 4°C	7 days
Residue, Filterable (TDS)	P, G	Cool, 4°C	7 days
Residue, Nonfilterable (TSS)	P, G	Cool, 4°C	7 days
Residue, Settleable	P, G	Cool, 4°C	48 hours
Residue, Volatile (Ash Content)	P, G	Cool, 4°C	7 days
Silica	P	Cool, 4°C	28 days
Specific Conductance	P, G	Cool, 4°C	28 days
Sulfate	P, G	Cool, 4°C	28 days

Subject NON-RADIOLOGICAL SAMPLE HANDLING	Number SA-6.1	Page 10 of 11
	Revision 3	Effective Date 02/04

**ATTACHMENT B
ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES,
AND HOLDING TIMES
PAGE TWO**

Parameter Number/Name	Container ⁽¹⁾	Preservation ⁽²⁾⁽³⁾	Maximum Holding Time ⁽⁴⁾
-----------------------	--------------------------	--------------------------------	-------------------------------------

INORGANIC TESTS (Cont'd):

Sulfide	P, G	Cool, 4°C; add zinc acetate plus sodium hydroxide to pH 9	7 days
Sulfite	P, G	None required	Analyze immediately
Turbidity	P, G	Cool, 4°C	48 hours

METALS:⁽⁷⁾

Chromium VI (Hexachrome)	P, G	Cool, 4°C	24 hours
Mercury (Hg)	P, G	HNO ₃ to pH 2	28 days
Metals, except Chromium VI and Mercury	P, G	HNO ₃ to pH 2	6 months

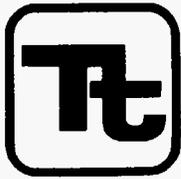
ORGANIC TESTS:⁽⁸⁾

Purgeable Halocarbons	G, Teflon-lined septum	Cool, 4°C; 0.008% Na ₂ S ₂ O ₃ ⁽⁵⁾	14 days
Purgeable Aromatic Hydrocarbons	G, Teflon-lined septum	Cool, 4°C; 0.008% Na ₂ S ₂ O ₃ ⁽⁵⁾ HCl to pH 2 ⁽⁹⁾	14 days
Acrolein and Acrylonitrile	G, Teflon-lined septum	Cool, 4°C; 0.008% Na ₂ S ₂ O ₃ ⁽⁵⁾ adjust pH to 4-5 ⁽¹⁰⁾	14 days
Phenols ⁽¹¹⁾	G, Teflon-lined cap	Cool, 4°C; 0.008% Na ₂ S ₂ O ₃ ⁽⁵⁾	7 days until extraction; 40 days after extraction
Benzidines ^{(11), (12)}	G, Teflon-lined cap	Cool, 4°C; 0.008% Na ₂ S ₂ O ₃ ⁽⁵⁾	7 days until extraction ⁽¹³⁾
Phthalate esters ⁽¹¹⁾	G, Teflon-lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction
Nitrosamines ^{(11), (14)}	G, Teflon-lined cap	Cool, 4°C; store in dark; 0.008% Na ₂ S ₂ O ₃ ⁽⁵⁾	7 days until extraction; 40 days after extraction
PCBs ⁽¹¹⁾	G, Teflon-lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction
Nitroaromatics & Isophorone ⁽¹¹⁾	G, Teflon-lined cap	Cool, 4°C; 0.008% Na ₂ S ₂ O ₃ ⁽⁵⁾ ; store in dark	7 days until extraction; 40 days after extraction
Polynuclear Aromatic Hydrocarbons (PAHs) ^{(11), (14)}	G, Teflon-lined cap	Cool, 4°C; 0.008% Na ₂ S ₂ O ₃ ⁽⁵⁾ ; store in dark	7 days until extraction; 40 days after extraction
Haloethers ⁽¹¹⁾	G, Teflon-lined cap	Cool, 4°C; 0.008% Na ₂ S ₂ O ₃ ⁽⁵⁾	7 days until extraction; 40 days after extraction
Dioxin/Furan (TCDD/TCDF) ⁽¹¹⁾	G, Teflon-lined cap	Cool, 4°C; 0.008% Na ₂ S ₂ O ₃ ⁽⁵⁾	7 days until extraction; 40 days after extraction

Subject NON-RADIOLOGICAL SAMPLE HANDLING	Number SA-6.1	Page 11 of 11
	Revision 3	Effective Date 02/04

**ATTACHMENT B
ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES,
AND HOLDING TIMES
PAGE THREE**

- (1) Polyethylene (P): generally 500 ml or Glass (G): generally 1L.
- (2) Sample preservation should be performed immediately upon sample collection. For composite chemical samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.
- (3) When any sample is to be shipped by common carrier or sent through the United States Mail, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172).
- (4) Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer periods, and has received a variance from the Regional Administrator.
- (5) Should only be used in the presence of residual chlorine.
- (6) Maximum holding time is 24 hours when sulfide is present. Optionally, all samples may be tested with lead acetate paper before pH adjustments are made to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.
- (7) Samples should be filtered immediately on site before adding preservative for dissolved metals.
- (8) Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.
- (9) Sample receiving no pH adjustment must be analyzed within 7 days of sampling.
- (10) The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.
- (11) When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9; samples preserved in this manner may be held for 7 days before extraction and for 40 days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re: the requirement for thiosulfate reduction of residual chlorine) and footnotes 12, 13 (re: the analysis of benzidine).
- (12) If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0±0.2 to prevent rearrangement to benzidine.
- (13) Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.
- (14) For the analysis of diphenylnitrosamine, add 0.008% Na₂S₂O₃ and adjust pH to 7-10 with NaOH within 24 hours of sampling.
- (15) The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% Na₂S₂O₃.



TETRA TECH NUS, INC.

STANDARD OPERATING PROCEDURES

Number SA-6.3	Page 1 of 12
Effective Date 09/03	Revision 2
Applicability Tetra Tech NUS, Inc.	
Prepared Earth Sciences Department	
Approved D. Senovich <i>ds</i>	

Subject
FIELD DOCUMENTATION

TABLE OF CONTENTS

<u>SECTION</u>	<u>PAGE</u>
1.0 PURPOSE.....	2
2.0 SCOPE.....	2
3.0 GLOSSARY	2
4.0 RESPONSIBILITIES.....	2
5.0 PROCEDURES.....	2
5.1 SITE LOGBOOK	2
5.1.1 General.....	2
5.1.2 Photographs.....	3
5.2 FIELD NOTEBOOKS	3
5.3 FIELD FORMS	4
5.3.1 Sample Collection, Labeling, Shipment, Request for Analysis, and Field Test Results..	4
5.3.2 Hydrogeological and Geotechnical Forms	5
5.3.3 Equipment Calibration and Maintenance Form.....	6
5.4 FIELD REPORTS.....	6
5.4.1 Daily Activities Report.....	6
5.4.2 Weekly Status Reports.....	7
6.0 LISTING OF TETRA TECH NUS FIELD FORMS FOUND ON THE TTNUS INTRANET SITE. <u>HTTP://INTRANET.TTNUS.COM</u> CLICK ON FIELD LOG SHEETS.....	7

ATTACHMENTS

A	TYPICAL SITE LOGBOOK ENTRY	9
B	SAMPLE LABEL.....	10
C	CHAIN-OF-CUSTODY RECORD FORM.....	11
D	CHAIN-OF-CUSTODY SEAL	12

Subject FIELD DOCUMENTATION	Number SA-6.3	Page 2 of 12
	Revision 2	Effective Date 09/03

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to identify and designate the field data record forms, logs and reports generally initiated and maintained for documenting Tetra Tech NUS field activities.

2.0 SCOPE

Documents presented within this procedure (or equivalents) shall be used for all Tetra Tech NUS field activities, as applicable. Other or additional documents may be required by specific client contracts or project planning documents.

3.0 GLOSSARY

None

4.0 RESPONSIBILITIES

Project Manager (PM) - The Project Manager is responsible for obtaining hardbound, controlled-distribution logbooks (from the appropriate source), as needed. In addition, the Project Manager is responsible for placing all field documentation used in site activities (i.e., records, field reports, sample data sheets, field notebooks, and the site logbook) in the project's central file upon the completion of field work.

Field Operations Leader (FOL) - The Field Operations Leader is responsible for ensuring that the site logbook, notebooks, and all appropriate and current forms and field reports illustrated in this guideline (and any additional forms required by the contract) are correctly used, accurately filled out, and completed in the required time-frame.

5.0 PROCEDURES

5.1 Site Logbook

5.1.1 General

The site logbook is a hard-bound, paginated, controlled-distribution record book in which all major onsite activities are documented. At a minimum, the following activities/events shall be recorded or referenced (daily) in the site logbook:

- All field personnel present
- Arrival/departure of site visitors
- Time and date of H&S training
- Arrival/departure of equipment
- Time and date of equipment calibration
- Start and/or completion of borehole, trench, monitoring well installation, etc.
- Daily onsite activities performed each day
- Sample pickup information
- Health and Safety issues (level of protection observed, etc.)
- Weather conditions

A site logbook shall be maintained for each project. The site logbook shall be initiated at the start of the first onsite activity (e.g., site visit or initial reconnaissance survey). Entries are to be made for every day

Subject FIELD DOCUMENTATION	Number SA-6.3	Page 3 of 12
	Revision 2	Effective Date 09/03

that onsite activities take place which involve Tetra Tech NUS or subcontractor personnel. Upon completion of the fieldwork, the site logbook must become part of the project's central file.

The following information must be recorded on the cover of each site logbook:

- Project name
- Tetra Tech NUS project number
- Sequential book number
- Start date
- End date

Information recorded daily in the site logbook need not be duplicated in other field notebooks (see Section 5.2), but must summarize the contents of these other notebooks and refer to specific page locations in these notebooks for detailed information (where applicable). An example of a typical site logbook entry is shown in Attachment A.

If measurements are made at any location, the measurements and equipment used must either be recorded in the site logbook or reference must be made to the field notebook in which the measurements are recorded (see Attachment A).

All logbook, notebook, and log sheet entries shall be made in indelible ink (black pen is preferred). No erasures are permitted. If an incorrect entry is made, the entry shall be crossed out with a single strike mark, and initialed and dated. At the completion of entries by any individual, the logbook pages used must be signed and dated. The site logbook must also be signed by the Field Operations Leader at the end of each day.

5.1.2 Photographs

When movies, slides, or photographs are taken of a site or any monitoring location, they must be numbered sequentially to correspond to logbook/notebook entries. The name of the photographer, date, time, site location, site description, and weather conditions must be entered in the logbook/notebook as the photographs are taken. A series entry may be used for rapid-sequence photographs. The photographer is not required to record the aperture settings and shutter speeds for photographs taken within the normal automatic exposure range. However, special lenses, films, filters, and other image-enhancement techniques must be noted in the logbook/notebook. If possible, such techniques shall be avoided, since they can adversely affect the accuracy of photographs. Chain-of-custody procedures depend upon the subject matter, type of camera (digital or film), and the processing it requires. Film used for aerial photography, confidential information, or criminal investigation require chain-of-custody procedures. Once processed, the slides of photographic prints shall be consecutively numbered and labeled according to the logbook/notebook descriptions. The site photographs and associated negatives and/or digitally saved images to compact disks must be docketed into the project's central file.

5.2 Field Notebooks

Key field team personnel may maintain a separate dedicated field notebook to document the pertinent field activities conducted directly under their supervision. For example, on large projects with multiple investigative sites and varying operating conditions, the Health and Safety Officer may elect to maintain a separate field notebook. Where several drill rigs are in operation simultaneously, each site geologist assigned to oversee a rig must maintain a field notebook.

Subject FIELD DOCUMENTATION	Number SA-6.3	Page 4 of 12
	Revision 2	Effective Date 09/03

5.3 **Field Forms**

All Tetra Tech NUS field forms (see list in Section 6.0 of this SOP) can be found on the company's intranet site (<http://intranet.ttnus.com>) under Field Log Sheets. Forms may be altered or revised for project-specific needs contingent upon client approval. Care must be taken to ensure that all essential information can be documented. Guidelines for completing these forms can be found in the related sampling SOP.

5.3.1 **Sample Collection, Labeling, Shipment, Request for Analysis, and Field Test Results**

5.3.1.1 Sample Log Sheet

Sample Log Sheets are used to record specified types of data while sampling. The data recorded on these sheets are useful in describing the sample as well as pointing out any problems, difficulties, or irregularities encountered during sampling. A log sheet must be completed for each sample obtained, including field quality control (QC) samples.

5.3.1.2 Sample Label

A typical sample label is illustrated in Attachment B. Adhesive labels must be completed and applied to every sample container. Sample labels can usually be obtained from the appropriate Program source electronically generated in-house, or are supplied from the laboratory subcontractor.

5.3.1.3 Chain-of-Custody Record Form

The Chain-of-Custody (COC) Record is a multi-part form that is initiated as samples are acquired and accompanies a sample (or group of samples) as they are transferred from person to person. This form must be used for any samples collected for chemical or geotechnical analysis whether the analyses are performed on site or off site. One carbonless copy of the completed COC form is retained by the field crew, one copy is sent to the Project Manager (or designee), while the original is sent to the laboratory. The original (top, signed copy) of the COC form shall be placed inside a large Ziploc-type bag and taped inside the lid of the shipping cooler. If multiple coolers are sent but are included on one COC form, the COC form should be sent with the cooler containing vials for VOC analysis or the cooler with the air bill attached. The air bill should then state how many coolers are included with that shipment. An example of a Chain-of-Custody Record form is provided as Attachment C. Once the samples are received at the laboratory, the sample cooler and contents are checked and any problems are noted on the enclosed COC form (any discrepancies between the sample labels and COC form and any other problems that are noted are resolved through communication between the laboratory point-of-contact and the Tetra Tech NUS Project Manager). The COC form is signed and copied. The laboratory will retain the copy while the original becomes part of the samples' corresponding analytical data package.

5.3.1.4 Chain-of-Custody Seal

Attachment D is an example of a custody seal. The Custody seal is an adhesive-backed label. It is part of a chain-of-custody process and is used to prevent tampering with samples after they have been collected in the field and sealed in coolers for transport to the laboratory. The COC seals are signed and dated by the sampler(s) and affixed across the lid and body of each cooler (front and back) containing environmental samples (see SOP SA-6.1). COC seals may be available from the laboratory; these seals may also be purchased from a supplier.

Subject FIELD DOCUMENTATION	Number SA-6.3	Page 5 of 12
	Revision 2	Effective Date 09/03

5.3.1.5 Geochemical Parameters Log Sheets

Field Analytical Log Sheets are used to record geochemical and/or natural attenuation field test results.

5.3.2 **Hydrogeological and Geotechnical Forms**

5.3.2.1 Groundwater Level Measurement Sheet

A Groundwater Level Measurement Sheet must be filled out for each round of water level measurements made at a site.

5.3.2.2 Data Sheet for Pumping Test

During the performance of a pumping test (or an in-situ hydraulic conductivity test), a large amount of data must be recorded, often within a short time period. The Pumping Test Data Sheet facilitates this task by standardizing the data collection format for the pumping well and observation wells, and allowing the time interval for collection to be laid out in advance.

5.3.2.3 Packer Test Report Form

A Packer Test Report Form must be completed for each well upon which a packer test is conducted.

5.3.2.4 Boring Log

During the progress of each boring, a log of the materials encountered, operation and driving of casing, and location of samples must be kept. The Summary Log of Boring, or Boring Log is used for this purpose and must be completed for each soil boring performed. In addition, if volatile organics are monitored on cores, samples, cuttings from the borehole, or breathing zone, (using a PID or FID), these readings must be entered on the boring log at the appropriate depth. The "Remarks" column can be used to subsequently enter the laboratory sample number, the concentration of key analytical results, or other pertinent information. This feature allows direct comparison of contaminant concentrations with soil characteristics.

5.3.2.5 Monitoring Well Construction Details Form

A Monitoring Well Construction Details Form must be completed for every monitoring well, piezometer, or temporary well point installed. This form contains specific information on length and type of well riser pipe and screen, backfill, filter pack, annular seal and grout characteristics, and surface seal characteristics. This information is important in evaluating the performance of the monitoring well, particularly in areas where water levels show temporal variation, or where there are multiple (immiscible) phases of contaminants. Depending on the type of monitoring well (in overburden or bedrock, stick-up or flush mount), different forms are used.

5.3.2.6 Test Pit Log

When a test pit or trench is constructed for investigative or sampling purposes, a Test Pit Log must be filled out by the responsible field geologist or sampling technician.

Subject FIELD DOCUMENTATION	Number SA-6.3	Page 6 of 12
	Revision 2	Effective Date 09/03

5.3.2.7 Miscellaneous Monitoring Well Forms

Monitoring Well Materials Certificate of Conformance should be used as the project directs to document all materials utilized during each monitoring well installation.

The Monitoring Well Development Record should be used as the project directs to document all well development activities.

5.3.2.8 Miscellaneous Field Forms - QA and Checklists

Container Sample and Inspection Sheet should be used as the project directs each time a container (drum, tank, etc.) is sampled and/or inspected.

QA Sample Log Sheet should be used at the project directs each time a QA sample is collected, such as Rinsate Blank, Source Blank, etc.

Field Task Modification Request (FTMR) will be prepared for all deviations from the project planning documents. The FOL is responsible for initiating the FTMRs. Copies of all FTMRs will be maintained with the onsite planning documents and originals will be placed in the final evidence file.

The Field Project Daily Activities Check List and Field Project Pre-Mobilization Checklist should be used during both the planning and field effort to assure that all necessary tasks are planned for and completed. These two forms are not a requirement but a useful tool for most field work.

5.3.3 **Equipment Calibration and Maintenance Form**

The calibration or standardization of monitoring, measuring or test equipment is necessary to assure the proper operation and response of the equipment, to document the accuracy, precision or sensitivity of the measurement, and determine if correction should be applied to the readings. Some items of equipment require frequent calibration, others infrequent. Some are calibrated by the manufacturer, others by the user.

Each instrument requiring calibration has its own Equipment Calibration Log which documents that the manufacturer's instructions were followed for calibration of the equipment, including frequency and type of standard or calibration device. An Equipment Calibration Log must be maintained for each electronic measuring device used in the field; entries must be made for each day the equipment is used or in accordance with the manufacturer's recommendations.

5.4 Field Reports

The primary means of recording onsite activities is the site logbook. Other field notebooks may also be maintained. These logbooks and notebooks (and supporting forms) contain detailed information required for data interpretation or documentation, but are not easily useful for tracking and reporting of progress. Furthermore, the field logbook/notebooks remain onsite for extended periods of time and are thus not accessible for timely review by project management.

5.4.1 **Daily Activities Report**

To provide timely oversight of onsite contractors, Daily Activities Reports are completed and submitted as described below.

Subject FIELD DOCUMENTATION	Number SA-6.3	Page 7 of 12
	Revision 2	Effective Date 09/03

5.4.1.1 Description

The Daily Activities Report (DAR) documents the activities and progress for each day's field work. This report must be filled out on a daily basis whenever there are drilling, test pitting, well construction, or other related activities occurring which involve subcontractor personnel. These sheets summarize the work performed and form the basis of payment to subcontractors. The DAR form can be found on the TtNUS intranet site.

5.4.1.2 Responsibilities

It is the responsibility of the rig geologist to complete the DAR and obtain the driller's signature acknowledging that the times and quantities of material entered are correct.

5.4.1.3 Submittal and Approval

At the end of the shift, the rig geologist must submit the Daily Activities Report to the Field Operations Leader (FOL) for review and filing. The Daily Activities Report is not a formal report and thus requires no further approval. The DAR reports are retained by the FOL for use in preparing the site logbook and in preparing weekly status reports for submission to the Project Manager.

5.4.2 **Weekly Status Reports**

To facilitate timely review by project management, photocopies of logbook/notebook entries may be made for internal use.

It should be noted that in addition to summaries described herein, other summary reports may also be contractually required.

All Tetra Tech NUS field forms can be found on the company's intranet site at <http://intranet.ttnus.com> under Field Log Sheets.

6.0 **LISTING OF TETRA TECH NUS FIELD FORMS FOUND ON THE TTNUS INTRANET SITE. HTTP://INTRANET.TTNUS.COM CLICK ON FIELD LOG SHEETS**

Groundwater Sample Log Sheet
Surface Water Sample Log Sheet
Soil/Sediment Sample Log Sheet
Container Sample and Inspection Sheet
Geochemical Parameters (Natural Attenuation)
Groundwater Level Measurement Sheet
Pumping Test Data Sheet
Packer Test Report Form
Boring Log
Monitoring Well Construction Bedrock Flush Mount
Monitoring Well Construction Bedrock Open Hole
Monitoring Well Construction Bedrock Stick Up
Monitoring Well Construction Confining Layer
Monitoring Well Construction Overburden Flush Mount
Monitoring Well Construction Overburden Stick Up
Test Pit Log
Monitoring Well Materials Certificate of Conformance
Monitoring Well Development Record

Subject FIELD DOCUMENTATION	Number SA-6.3	Page 8 of 12
	Revision 2	Effective Date 09/03

Daily Activities Record
Field Task Modification Request
Hydraulic Conductivity Test Data Sheet
Low Flow Purge Data Sheet
QA Sample Log Sheet
Equipment Calibration Log
Field Project Daily Activities Checklist
Field Project Pre-Mobilization Checklist

Subject FIELD DOCUMENTATION	Number SA-6.3	Page 9 of 12
	Revision 2	Effective Date 09/03

**ATTACHMENT A
TYPICAL SITE LOGBOOK ENTRY**

START TIME: _____ DATE: _____

SITE LEADER: _____

PERSONNEL: _____

TtNUS	DRILLER	SITE VISITORS
_____	_____	_____
_____	_____	_____
_____	_____	_____

WEATHER: Clear, 68°F, 2-5 mph wind from SE

ACTIVITIES:

1. Steam jenny and fire hoses were set up.
2. Drilling activities at well ____ resumes. Rig geologist was _____. See Geologist's Notebook, No. 1, page 29-30, for details of drilling activity. Sample No. 123-21-S4 collected; see sample logbook, page 42. Drilling activities completed at 11:50 and a 4-inch stainless steel well installed. See Geologist's Notebook, No. 1, page 31, and well construction details for well _____.
3. Drilling rig No. 2 steam-cleaned at decontamination pit. Then set up at location of well _____.
4. Well _____ drilled. Rig geologist was _____. See Geologist's Notebook, No. 2, page ____ for details of drilling activities. Sample numbers 123-22-S1, 123-22-S2, and 123-22-S3 collected; see sample logbook, pages 43, 44, and 45.
5. Well _____ was developed. Seven 55-gallon drums were filled in the flushing stage. The well was then pumped using the pitcher pump for 1 hour. At the end of the hour, water pumped from well was "sand free."
6. EPA remedial project manger arrives on site at 14:25 hours.
7. Large dump truck arrives at 14:45 and is steam-cleaned. Backhoe and dump truck set up over test pit _____.
8. Test pit _____ dug with cuttings placed in dump truck. Rig geologist was _____. See Geologist's Notebook, No. 1, page 32, for details of test pit activities. Test pit subsequently filled. No samples taken for chemical analysis. Due to shallow groundwater table, filling in of test pit ____ resulted in a very soft and wet area. A mound was developed and the area roped off.
9. Express carrier picked up samples (see Sample Logbook, pages 42 through 45) at 17:50 hours. Site activities terminated at 18:22 hours. All personnel off site, gate locked.

Field Operations Leader

Subject FIELD DOCUMENTATION	Number SA-6.3	Page 10 of 12
	Revision 2	Effective Date 09/03

ATTACHMENT B

	Tetra Tech NUS, Inc. 661 Andersen Drive Pittsburgh, 15220 (412)921-7090		Project:
			Site:
		Location:	
Sample No:		Matrix:	
Date:	Time:	Preserve:	
Analysis:			
Sampled by:		Laboratory:	

Subject FIELD DOCUMENTATION	Number SA-6.3	Page 12 of 12
	Revision 2	Effective Date 09/03

ATTACHMENT D

CHAIN-OF-CUSTODY SEAL

<u>Signature</u> <hr/> <u>Date</u> <hr/> CUSTODY SEAL	CUSTODY SEAL <hr/> Date <hr/> Signature
--	--



STANDARD OPERATING PROCEDURES

Number SA-7.1	Page 1 of 16
Effective Date 04/07/2008	Revision 5
Applicability Tetra Tech NUS, Inc.	
Prepared Earth Sciences Department	
Approved Tom Johnston <i>T.E. Johnston</i>	

Subject DECONTAMINATION OF FIELD EQUIPMENT

TABLE OF CONTENTS

<u>SECTION</u>	<u>PAGE</u>
1.0 PURPOSE.....	3
2.0 SCOPE AND APPLICABILITY	3
3.0 GLOSSARY	3
4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS	4
5.0 HEALTH AND SAFETY	4
6.0 EQUIPMENT LIST	4
7.0 PROCEDURES.....	5
7.1 Decontamination Pad Design/Construction Considerations	6
7.1.1 Temporary Decontamination Pads.....	6
7.1.2 Decontamination Activities at Drill Rigs/DPT Units	8
7.1.3 Decontamination Activities at Remote Sample Locations	8
7.2 Equipment Decontamination Procedures	8
7.2.1 Monitoring Well Sampling Equipment.....	8
7.2.2 Downhole Drilling Equipment	10
7.2.3 Soil/Sediment Sampling Equipment	12
7.3 Contact Waste/Materials	12
7.3.1 Investigation-Derived Wastes - Decontamination Wash Waters and Sediments	13
7.4 Decontamination Evaluation	14

ATTACHMENTS

A INVESTIGATION-DERIVED WASTE LABEL.....	15
--	----

Subject DECONTAMINATION OF FIELD EQUIPMENT	Number SA-7.1	Page 2 of 16
	Revision 5	Effective Date 04/07/2008

1.0 PURPOSE

Decontamination is the process of removing and/or neutralizing site contaminants that have contacted and/or accumulated on equipment. The purpose of this Standard Operating Procedure (SOP) is to protect site personnel, the general public, and the environment while preserving or maintaining sample integrity. It is further intended through this procedure to describe the steps necessary for proper decontamination of drilling equipment, earth-moving equipment, chemical sampling equipment and field operation and analytical equipment.

2.0 SCOPE AND APPLICABILITY

This procedure applies to all equipment used to provide access to/acquire environmental samples that may have become contaminated through direct contact with contaminated media including air, water, and soil. This equipment includes drilling and heavy equipment and chemical sampling and field analytical equipment. Where technologically and economically feasible, single-use sealed disposable equipment will be employed to minimize the potential for cross-contamination. This SOP also provides general reference information on the control of contaminated materials.

Decontamination methods and equipment requirements may differ from one project to another. General equipment items are specified in Section 6.0, but project-specific equipment must be obtained to address the project-specific decontamination procedures presented in Section 7.0 and applicable subsections.

3.0 GLOSSARY

Alconox/Liquinox - A brand of phosphate-free laboratory-grade detergent.

Decontamination Solution - A solution selected/identified in the Health and Safety Plan or Project-Specific Quality Assurance Plan. The solution is selected and employed as directed by the project chemist/health and safety professional.

Deionized Water (DI) - Tap water that has been treated by passing through a standard deionizing resin column. This water may also pass through additional filtering media to attain various levels of analyte-free status. The DI water should meet College of American Pathologists (CAP) and National Committee for Clinical Laboratory Standards (NCCLS) specifications for reagent-grade Type I water.

Potable Water - Tap water from any municipal water treatment system. Use of an untreated potable water supply is not an acceptable substitute for tap water.

Pressure Washing - Process employing a high-pressure pump and nozzle configuration to create a high-pressure spray of potable water. High-pressure spray is employed to remove solids from equipment.

Solvent - A liquid in which solid chemicals or other liquids are dissolved. The solvent of choice is pesticide-grade isopropanol. Use of other solvents (methanol, acetone, or hexane) may be required for particular projects or for a particular purpose (e.g., removal of concentrated waste) and must be justified in the project planning documents. For example, it may be necessary to use hexane when analyzing for trace levels of pesticides, PCBs, or fuels. In addition, because many of these solvents are not miscible in water, the equipment should be air dried prior to use. Solvents should not be used on PVC equipment or well construction materials.

Steam Pressure Washing - A cleaning method employing a high-pressure spray of heated potable water to remove various organic/inorganic chemicals from equipment.

Subject DECONTAMINATION OF FIELD EQUIPMENT	Number SA-7.1	Page 3 of 16
	Revision 5	Effective Date 04/07/2008

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager - Responsible for ensuring that all field activities are conducted in accordance with approved project plan(s) requirements.

Decontamination Personnel - Individuals assigned the task of decontamination. It is the responsibility of these individuals to understand the use and application of the decontamination process and solutions as well as the monitoring of that process to ensure that it is working properly. This is accomplished through visual evaluation, monitoring instrument scanning of decontaminated items, and/or through the collection of rinsate blanks to verify contaminant removal.

Field Operations Leader (FOL) - Responsible for the implementation of project-specific planning documents. This includes on-site verification that all field activities are performed in compliance with approved SOPs or as otherwise dictated by the approved project plan(s). The FOL is also responsible for the completion and accuracy of all field documentation.

Site Safety Officer (SSO) - Exercises shared responsibility with the FOL concerning decontamination effectiveness. All equipment arriving on site (as part of the equipment inspection), leaving the site, and moving between locations is required to go through a decontamination evaluation. This is accomplished through visual examination and/or instrument screening to determine the effectiveness of the decontamination process. Improper or incomplete decontamination is sufficient to restrict equipment from entering the site, exiting the site, or moving to a new location on the site until the objectives are successfully completed.

General personnel qualifications for decontamination activities include the following:

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.
- Capability of performing field work under the expected physical and environmental (i.e., weather) conditions.
- Familiarity with appropriate decontamination procedures.

5.0 HEALTH AND SAFETY

In addition to the health and safety issues and reminders specified in subsections of this SOP, the following considerations and requirements must be observed as SOPs for field equipment decontamination activities:

- If any solvents or hazardous chemicals (e.g., isopropyl alcohol) are to be used in equipment decontamination activities, the FOL must first obtain the manufacturer's/supplier's Material Safety Data Sheet (MSDS) and assure that it is reviewed by all users (prior to its use), added to the site Hazardous Chemical Inventory, and maintained on site as part of the project Hazard Communication Program.
- Review and observe specific health and safety requirements (e.g., personal protective equipment [PPE]) specified in the project-specific health and safety plan for this activity.

6.0 EQUIPMENT LIST

- Wood for decontamination pad construction, when applicable (see Section 7.1).

Subject	DECONTAMINATION OF FIELD EQUIPMENT	Number	SA-7.1	Page	4 of 16
		Revision	5	Effective Date	04/07/2008

- Tools for constructing decontamination pad frame, when applicable (see Section 7.1).
- Visqueen sheeting or comparable material to cover decontamination pad frame, when applicable (see Section 7.1).
- Wash/drying racks for auger flights and drill/drive rods, when applicable (see Section 7.2).
- PPE as specified in the project health and safety plan.
- Soap and water for washing and rinsing.
- Deionized water for final rinsing.
- Solvents (e.g., pesticide-grade isopropanol) for rinsing (see applicable portions of Section 7.2).
- Tubs, buckets, etc. for containerizing rinse water (see applicable portions of Section 7.2).
- Sample bottles for collecting rinsate blanks (see Section 7.2).
- Calibrated photoionization detector (PID) or flame ionization detector (FID) to monitor decontaminated equipment for organic vapors generated through the existence of residual contamination or the presence of decontamination solvent remaining after the piece was rinsed.
- Aluminum foil or clear clean plastic bag for covering cleaned equipment (see applicable portions of Section 7.2).
- Paper towels or cloths for wiping.
- Brushes, scrapers, or other hand tools useful for removing solid materials from equipment.
- Clear plastic wrap for covering or wrapping large decontaminated equipment items (see Section 7.2.2).
- Drum-moving equipment for moving filled waste drums (optional) (see Section 7.3).
- Drum labels for waste drums (see Attachment A).

7.0 PROCEDURES

The process of decontamination is accomplished through the removal of contaminants, neutralization of contaminants, or isolation of contaminants. To accomplish this activity, preparation is required including site preparation, equipment selection, and evaluation of the decontamination requirements and processes. Site contaminant types, concentrations, and media types are primary drivers in the selection of the types of decontamination and where it will be conducted. For purposes of this SOP, discussion is limited to decontamination procedures for general environmental investigations.

Decontamination processes will be performed at the location(s) specified in project-specific planning documents. Typical decontamination locations include the following:

- Temporary decontamination pads/facilities
- Sample locations
- Centralized decontamination pad/facilities

Subject DECONTAMINATION OF FIELD EQUIPMENT	Number SA-7.1	Page 5 of 16
	Revision 5	Effective Date 04/07/2008

- Combination of some or all of the above

The following discussion includes general considerations for the decontamination process. Specific construction and implementation procedures will be as specified in the project-specific planning documents and/or may be as dictated by site-specific conditions as long as the intent of the requirements in the planning documents is met. This intent is to contain any residual fluids and solids generated through the decontamination process.

7.1 Decontamination Pad Design/Construction Considerations

7.1.1 Temporary Decontamination Pads

Temporary decontamination pads may be constructed at satellite locations within the site area in support of temporary work areas. These structures are generally constructed to support the decontamination of heavy equipment such as drill rigs and earth-moving equipment but can be employed for smaller articles.

The purpose of the decontamination pad is to contain wash waters and potentially contaminated soil generated during decontamination procedures. Therefore, construction of these pads should take into account the following considerations:

- Site location – The decontamination site selected should be far enough from the work site to maximize decontamination effectiveness while minimizing travel distance. The location of the decontamination site shall be selected to provide, in the judgment of the FOL or FOL designee, compliance with as many of the following characteristics as practicable:
 - Well removed from pedestrian/vehicle thoroughfares.
 - Avoidance of areas where control/custody cannot be maintained.
 - Avoidance of areas where potential releases of contaminated media or decontamination fluids may be compounded through access to storm water transport systems, streams, or other potentially sensitive areas.
 - Avoidance of potentially contaminated areas.
 - Avoidance of areas too close to the ongoing operation, where cross-contamination may occur.

The selected decontamination site should include the following, where possible:

- Areas where potable water and electricity are provided.

Safety Reminder

When utilizing electrical power sources, either hard-wired or portable-generated sources, ensure that:

- All power is routed through a Ground Fault Circuit Interrupter (GFCI).
- All power cords are in good condition (no physical damage), rated for the intended energy load, and designated for outdoor use.

In situations where accomplishing these elements is not possible, it will be necessary to implement a site electrical grounding program.

Subject	DECONTAMINATION OF FIELD EQUIPMENT	Number	SA-7.1	Page	6 of 16
		Revision	5	Effective Date	04/07/2008

- Areas where support activities such as removing decontamination waters soil and sediment are possible without entering an active exclusion zone.
- Areas that offer sufficient size to carry out the specific decontamination sequence.
- Decontamination pad (decon pad) – The decon pad shall be constructed to meet the following characteristics:
 - Size – The size of the pad should be sufficient to accept the equipment to be decontaminated as well as permitting free movement around the equipment by the personnel conducting the decontamination. The size should permit these movements utilizing pressure/steam washer wands and hoses and minimizing splash due to work in close quarters.
 - Slope – An adequate slope will be constructed to permit the collection of water and potentially contaminated soil within a trough or sump constructed at one end. The collection point for wash waters should be of adequate distance that the decontamination workers do not have to walk through the wash waters while completing their tasks. Because the pad will be sloped, place a light coating of sand over the plastic to minimize potential slips and falls. See the text about liners below.
 - Sidewalls – The sidewalls shall be at least 6 inches in height (or as high as possible if 6 inches is not achievable) to provide adequate containment for wash waters and soil. If splash represents a potential problem, splash guards should be constructed to control overspray. Sidewalls may be constructed of wood, inflatables, sand bags, etc. to permit containment. Splash guards are typically wood frames with Visqueen coverings to control overspray.
 - Liner – Depending on the types of equipment and decontamination method to be used, the liner should be of sufficient thickness to provide a puncture-resistant barrier between the decontamination operation and the unprotected environment. Care should be taken to examine the surface area prior to placing the liner to remove sharp articles (sticks, stones, debris) that could puncture the liner. Liners are intended to form an impermeable barrier. The thickness may vary from a minimum recommended thickness of 10 mil to 30 mil. The desired thickness may be achieved through layering materials of lighter construction. It should be noted that various materials (rubber, polyethylene sheeting) become slippery when wet. To minimize this potential hazard associated with a sloped liner, a light coating of sand shall be applied to provide traction as necessary.
 - Wash/drying racks – Auger flights, drill/drive rods, and similar equipment require racks positioned off of the ground to permit these articles to be washed, drained, and dried while secured from falling during this process.

For decontamination of direct-push technology (DPT) equipment, the pad may be as simple as a mortar tub containing buckets of soapy water for washing and an empty bucket to capture rinse waters. Decontamination may be conducted at the rear of the rig to permit rapid tool exchange.

- Maintenance – Maintain the decontamination area by:
 - Periodically clearing the work area of standing water, soil, and debris, and coiling hoses to aid in eliminating slip, trip, and fall hazards. In addition, these articles will reduce potential backsplash and cross-contamination.

Subject	DECONTAMINATION OF FIELD EQUIPMENT	Number	SA-7.1	Page	7 of 16
		Revision	5	Effective Date	04/07/2008

- Regularly changing the decontamination fluids to ensure proper cleaning and prevent cross-contamination.
- PPE – Periodically evaluate the condition of, and maintain the decontamination equipment, including regular cleaning of face shields and safety glasses. This is critical to ensuring the safety of decontamination personnel and the integrity of the decontamination process, and it will ensure that equipment is functioning properly.

7.1.2 Decontamination Activities at Drill Rigs/DPT Units

During subsurface sampling activities including drilling and DPT activities, decontamination of drive rods, Macro Core Samplers, split spoons, etc. is typically conducted at an area adjacent to the operation. Decontamination is generally accomplished using a soap/water wash and rinse utilizing buckets and brushes. This area requires sufficient preparation to accomplish the decontamination objectives.

Buckets shall be placed within mortar tubs or similar secondary containment tubs to prevent splash and spills from reaching unprotected environmental media. Drying racks shall be employed as directed for temporary pads to permit parts to dry and be evaluated prior to use/reuse. Methodology regarding this activity is provided in Section 7.2.

7.1.3 Decontamination Activities at Remote Sample Locations

When sampling at remote locations, sampling equipment such as trowels and pumps/tubing should be evacuated of potentially contaminated media to the extent possible. This equipment should be wrapped in plastic for transport to the temporary/centralized decontamination location for final cleaning and disposition. Flushing and cleaning of single-use equipment such as disposable trowels, tubing, and surgeon's gloves may allow disposal of this equipment after visible soil and water remnants have been removed.

7.2 Equipment Decontamination Procedures

The following represents procedures to be employed for the decontamination of equipment that may have contacted and/or accumulated contamination through site investigation activities.

7.2.1 Monitoring Well Sampling Equipment

7.2.1.1 Groundwater sampling equipment – This includes pumps inserted into monitoring wells such as bladder pumps, Whale pumps, and Redi-Flo pumps and reusable bailers, etc.

1. Evacuate to the extent possible, any purge water within the pump/bailer.
2. Scrub using soap and water and/or steam clean the outside of the pump/bailer and, if applicable, the pump tubing.
3. Insert the pump and tubing/bailer into a clean container of soapy water. Pump/run a sufficient amount of soapy water through the pump/bailer to flush out any residual well water. After the pump is flushed, circulate soapy water through the pump to ensure that the internal components are thoroughly flushed.
4. Remove the pump and tubing/bailer from the container
5. Rinse external pump components using tap water.

Subject DECONTAMINATION OF FIELD EQUIPMENT	Number SA-7.1	Page 8 of 16
	Revision 5	Effective Date 04/07/2008

6. Insert the pump and tubing/bailer into a clean container of tap water. Pump/run a sufficient amount of tap water through the pump/bailer to evacuate all of the soapy water (until clear).

CAUTION

Do not rinse PE, PVC, and associated tubing with solvents –
Use the procedures defined in the project-specific planning documents. If they are not defined, contact the FOL for guidance. The solvent rinse described in Step 7 may be omitted if groundwater does not contain oil, grease, PAHs, PCBs, or other hard to remove organic materials.

7. If groundwater contains or is suspected to contain oil, grease, PAHs, PCBs, or other hard to remove organic materials, rinse the equipment to be cleaned with pesticide-grade isopropanol.
8. Pass deionized water through the hose to flush out the tap water and solvent residue as applicable.
9. Drain residual deionized water to the extent possible.
10. Allow components of the equipment to air dry.
11. For bladder pumps, disassemble the pump and wash the internal components with soap and water, then rinse with tap water, isopropanol, and deionized water and allow to dry. After the parts are dry, conduct a visual inspection and a monitoring instrument scan to ensure that potential contaminants and all decontamination solvent have been removed. Collect a rinsate blank in accordance with the project-specific planning documents to ensure that the decontamination process is functioning as intended. The typical frequency of collection for rinsate blanks is 1 per 20 field samples. In addition, wipe samples or field tests such as UV light may be used.
12. Wrap pump/bailer in aluminum foil or a clear clean plastic bag for storage.

SAFETY REMINDER

Remember when handling powered equipment to disconnect the power source and render the equipment to a zero energy state (both potential and kinetic) before opening valves, disconnecting lines, etc.

7.2.1.2 Electronic Water Level Indicators/Sounders/Tapes

During water level measurements, rinsing the extracted tape and probe with deionized water and wiping the surface of the extracted tape between locations is acceptable. However, periodic full decontamination should be conducted as follows:

1. Wash with soap and water
2. Rinse with tap water
3. Rinse with deionized water

NOTE

In situations where oil, grease, free product, other hard to remove materials are encountered, probes and exposed tapes should be washed in hot soapy water. If probes or tapes cannot be satisfactorily decontaminated (they are still stained, discolored, etc.), they should be removed from service.

Subject DECONTAMINATION OF FIELD EQUIPMENT	Number SA-7.1	Page 9 of 16
	Revision 5	Effective Date 04/07/2008

7.2.1.3 Miscellaneous Equipment

Miscellaneous equipment including analytical equipment (water quality testing equipment) shall be cleaned per manufacturers' instructions. This generally includes wiping the sensor housing and rinsing with tap and deionized water.

Coolers/shipping containers employed to ship samples are received from the laboratory in a variety of conditions including marginal to extremely poor. Coolers shall be evaluated prior to use for the following:

- Structural integrity – Coolers missing handles or having breaks in the outer housing should be removed and not used. Notify the laboratory that the risk of shipping samples in the cooler(s) provided is too great and request a replacement unit.
- Cleanliness – As per protocol, only volatile organic samples are accompanied by a trip blank. If a cooler's cleanliness is in question (visibly dirty/stained) or if there are noticeable odors, the cooler should be decontaminated prior to use as follows:
 1. Wash with soap and water
 2. Rinse with tap water
 3. Dry

If these measures fail to clean the cooler to an acceptable level, remove the unit from use as a shipping container and ask the cooler provider (e.g., the analytical laboratory) to provide a replacement unit.

7.2.2 **Downhole Drilling Equipment**

This includes any portion of the drill rig that is over the borehole, including auger flights, drill stems, rods, and associated tooling that would extend over the borehole. The following procedure is to be employed prior to initiating the drilling/sampling activity, then between locations:

CAUTION
 Exercise care when using scrapers to remove soil and debris from downhole drilling equipment. Inadvertent slips of scrapers have resulted in cuts, scrapes, and injured knuckles, so use scrapers carefully when removing soil from these items.

1. Remove loose soil using shovels, scrapers, etc.
2. Through a combination of scrubbing using soap and water and/or steam cleaning or pressure washing, remove visible dirt/soil from the equipment being decontaminated.

CAUTION
 In Step 3, do not rinse PE, PVC, and associated tubing with solvents. The appropriate procedures should be defined within the project-specific planning documents. If they are not defined, contact the FOL for guidance. The solvent rinse described in Step 4 may be omitted if groundwater does not contain oil, grease, PAHs, PCBs, or other hard to remove organic materials.

3. Rinse the equipment with tap water, where applicable (steam cleaning and pressure washing incorporate rinsing as part of the process).

Subject	DECONTAMINATION OF FIELD EQUIPMENT	Number	SA-7.1	Page	10 of 16
		Revision	5	Effective Date	04/07/2008

4. If the equipment has directly or indirectly contacted contaminated sample media and is known or suspected of being contaminated with oil, grease, PAHs, PCBs, or other hard to remove organic materials, rinse equipment with pesticide-grade isopropanol
5. To the extent possible, allow components to air dry.
6. If the decontaminated equipment is to be used immediately after decontamination, screen it with a calibrated photoionization detector (PID)/flame ionization detector (FID) to ensure that all contaminants and possible decontamination solvents (if they were used) have been adequately removed.
7. Wrap or cover equipment in clear plastic until it is time to be used.

SAFETY REMINDER

Even when equipment is disconnected from power sources, dangers such as the following may persist:

Falls - An auger flight standing on its end may fall and injure someone. Secure all loose articles to prevent heavy articles from falling onto people or equipment.

Burns - Steam cleaner water is heated to more than 212 °F and exhibits thermal energy that can cause burns. Prevent contact of skin with hot water or surfaces.

High water pressure - Pressure washer discharge can have 2,000 to 4,000 psi of water pressure. Water under this amount of pressure can rupture skin and other human tissues. Water at 4,000 psi exiting a 0° tip can be dangerous because of its relatively high cutting power. The exit velocity and cutting power of the water are reduced when exiting a 40° fan tip, but damage to soft tissues is still possible.

In general, follow the rules below to avoid injury, equipment damage, or incomplete decontamination:

1. Read the operating manual and follow the manufacturers' recommended safety practices before operating pressure washers and steam cleaners.
2. Never point the pressure washer or steam cleaner at another person or use to clean your boots or other parts of your body. Water lacerations and burns may appear to be minor at first but can be life threatening. Do not attempt to hold small parts in your hand while washing them with high-temperature or high-pressure water.
3. Always wear PPE as specified in the HASP such as:
 - Hard hat, safety glasses, splash shield, impermeable apron or splash suit, and hearing protection. Remember that excessive noise is a hazard when operating gas-powered engines and electrically driven pressure washers. PPE will be identified in your project specific planning documents.
4. Inspect each device before use. An inspection checklist will be provided in the project-specific planning documents. If it is a rented device, safety measures are typically provided by the vendor. In all cases, if you are not familiar with the operation of a pressure washer/steam cleaner, do not operate it until you obtain and thoroughly review operating instructions and recommended safety practices.
5. Do not modify equipment unless the manufacturer has approved the modifications.

Subject DECONTAMINATION OF FIELD EQUIPMENT	Number SA-7.1	Page 11 of 16
	Revision 5	Effective Date 04/07/2008

7.2.3 Soil/Sediment Sampling Equipment

This section applies to soil sampling equipment including but not limited to hand augers, stainless steel trowels/spoons, bowls, dredges, scoops, split spoons, Macro Core samplers, etc.

1. Remove all loose soil from the equipment through manual means.
2. Through a combination of scrubbing using soap and water and/or steam cleaning or pressure washing, remove visible dirt/soil from the equipment.
3. Rinse the equipment with tap water.

CAUTION

Do not rinse PE, PVC, and associated tubing with solvents. The appropriate procedures should be defined within the project-specific planning documents. If they are not defined, contact the FOL for guidance. The solvent rinse described in Step 4 may be omitted if groundwater does not contain oil, grease, PAHs, PCBs, or other hard to remove organic materials.

4. If the equipment is contaminated or suspected to be contaminated with oil, grease, PAHs, PCBs, or other hard to remove organic materials, rinse the equipment with pesticide-grade isopropanol.
5. Rinse the equipment with deionized water.
6. To the extent possible, allow components to air dry.
7. If the equipment is to be used immediately after decontamination, screen it with a calibrated PID/FID to ensure that all solvents (if they were used) and trace contaminants have been adequately removed.
8. After the equipment has dried, wrap it in aluminum foil for storage until use.

Dredges employed in sediment sampling are typically decontaminated as follows:

- Remove the sediment sample from the sampling device
- If sufficient associated surface water is available at the sampling site, place the dredge in the water and flush to remove visible sediment.
- Extract the dredge and wash it in soap and water per the project-specific planning documents.

CAUTION

When handling dredges, the primary safety concern is trapping fingers or extremities in the larger dredge samplers within the jaws or pinch points of the mechanical jaws. Keep hands, fingers, and extremities away from these pinch and compression points. Either handle the device by the rope or preferably lock the jaws in place to control the potential for closing during maintenance and/or cleaning.

Subject DECONTAMINATION OF FIELD EQUIPMENT	Number SA-7.1	Page 12 of 16
	Revision 5	Effective Date 04/07/2008

7.3 Contact Waste/Materials

During the course of field investigations, disposable/single-use equipment becomes contaminated. These items include tubing, trowels, PPE (gloves, overboots, splash suits, etc.), and broken sample containers.

With the exception of the broken glass, single-use articles should be cleaned (washed and rinsed) of visible materials and disposed as normal refuse. The exception to this rule is that extremely soiled materials that cannot be cleaned shall be containerized for disposal in accordance with applicable federal, state, and local regulations.

7.3.1 Investigation-Derived Wastes - Decontamination Wash Waters and Sediments

NOTE

Requirements for waste storage may differ from one facility to the next. Facility-specific directions for waste storage areas will be provided in project-specific documents, or separate direction will be provided by the Project Manager.

1. Assume that all investigation-derived waste (IDW) generated from decontamination activities contains the hazardous chemicals associated with the site unless there are analytical or other data to the contrary. Waste solution volumes could vary from a few gallons to several hundred gallons in cases where large equipment required cleaning.
2. Where possible, use filtering systems to extend the use of water within a closed system wash unit to recycle water and to reduce possible waste amounts.

NOTE

Containerized waste rinse solutions are best stored in 55-gallon drums (or equivalent containers) that can be sealed until ultimate disposal at an approved facility.

3. Label waste storage containers appropriately labeled (see Attachment A).
4. Ensure that the IDW storage area is configured to meet the following specifications to permit access to the containers and to conduct spill/leak monitoring, sampling, and extraction when the disposal route is determined:
 - Enclose areas accessible by the general public using construction fencing and signs.
 - Stored materials in 55-gallon drums on pallets with four (or fewer) drums per pallet.
 - Maintain the retaining bolt and label on the outside of storage containers where readily visible.
 - Provide at least 4 feet of room between each row of pallets to allow access to containers for sampling, drum removal, and spill response.
 - As directed in project-specific planning documents, maintain an IDW Inventory List and provide the list to the site Point of Contact at the termination of each shift.
 - Maintain spill response equipment at the IDW storage area in case it is required for immediate access.

Subject DECONTAMINATION OF FIELD EQUIPMENT	Number SA-7.1	Page 13 of 16
	Revision 5	Effective Date 04/07/2008

- Where possible, use equipment for moving containers. Where not possible, obtain help to manipulate containers.

Subject	DECONTAMINATION OF FIELD EQUIPMENT	Number	SA-7.1	Page	14 of 16
		Revision	5	Effective Date	04/07/2008

CAUTION

Each container of water can weigh up to 490 pounds. Each 55-gallon drum of wet soil can weigh more than 750 pounds. Fill drums and temporary containers to 80 percent capacity to minimize spill and handling difficulties. Use drum carts to move filled drums.

See safe lifting techniques provided in Section 4.4 of the Tetra Tech NUS, Inc. Health and Safety Guidance Manual.

When placing drums, keep your fingers out of pinch and smash points such as between the drums. In some cases such as well development and/or purge water, you can place the drums to be filled on the pallet and transport materials in smaller easier to handle containers.

7.4 Decontamination Evaluation

Upon decontamination of equipment, determine the effectiveness of the decontamination process in the following manner:

- Visual evaluation – A visual evaluation will be conducted to ensure the removal of particulate matter. This shall be done to ensure that the washing/rinsing process is working as intended.
- Instrument Screening – A properly calibrated PID/FID should be used to evaluate the presence of site contaminants and solvents used in the cleaning process. The air intake of the instrument shall be passed over the article to be evaluated. Avoid placing the instrument probe into residual waters. A PID/FID reading greater than the daily established background level requires a repeat of the decontamination process, followed by rescreening with the PID/FID. This sequence must be repeated until no instrument readings greater than the daily established background level are observed. It should be noted that the instrument scan is only viable if the contaminants are detectable within the instrument's capabilities.

NOTE

When required by project-specific planning documents, collection of rinsate blanks (see next step) shall be completed without exception unless approval to not collect these samples is obtained from the Project Manager.

- Collection of Rinsate Blanks – It is recommended that rinsate samples be collected to:
 - Evaluate the decontamination procedure representing different equipment applications (pumps versus drilling equipment) and different decontamination applications.
 - Single-use disposable equipment – The number of samples should represent different types of equipment as well as different lot numbers of single-use articles.
 - The collection and the frequency of collection of rinsate samples are as follows unless specified differently in the project-specific planning documents:
 - Per decontamination method
 - Per disposable article/batch number of disposable articles

Subject DECONTAMINATION OF FIELD EQUIPMENT	Number SA-7.1	Page 15 of 16
	Revision 5	Effective Date 04/07/2008

NOTE

It is recommended that an initial rinsate sample be collected early in the project to ensure that the decontamination process is functioning properly and to avoid using a contaminated batch of single-use articles. It is recommended that a follow-up sample be collected later during the execution of the project to ensure that those conditions do not change.

Rinsate samples collection may be driven by types of and/or levels of contaminant. Difficult to remove contaminants, oils/greases, some PAHs/PCBs, etc. may also support the collection of additional rinsates due to the obvious challenges to the decontamination process. This is a field consideration to be determined by the FOL.



STANDARD OPERATING PROCEDURES

Number SA-7.1	Page 16 of 16
Effective Date 04/07/2008	Revision 5
Applicability Tetra Tech NUS, Inc.	
Prepared Earth Sciences Department	
Approved Tom Johnston <i>T.E. Johnston</i>	

Subject DECONTAMINATION OF FIELD EQUIPMENT

Attachment A DW Label

INVESTIGATION DERIVED WASTE

GENERATOR INFORMATION:

SITE _____ JOB NO. _____

LOCATION _____

DATE _____

DRUM# _____

CONTENTS _____

VOLUME _____

CONTACT _____

EMERGENCY PHONE NUMBER _____

Project-Specific SAP
Site Name/Project Name: NAS JRB Willow Grove Site 5
Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study
Revision Number: 0
Revision Date: September 2008

APPENDIX B

LABORATORY STANDARD OPERATING PROCEDURES

UNCONTROLLED DOCUMENT: DO NOT Transfer or Print

Method: 02-5035
Revision: 0
Date: January 22, 2007
Page 1 of 32

Document Title: Closed-System Purge-and-Trap and Extraction
for Volatile Organics in Soil and Waste Samples

Document Control Number: _____

Organization Title: ANALYTICAL LABORATORY SERVICE, INC. (ALSI)

Address: 34 Dogwood Lane
Middletown, PA 17057

Phone: (717) 944-5541

Approved by:

Helen MacMinn,
Quality Assurance Manager

Date

Clark Dougherty,
GC/MS Supervisor

Date

Erin Ripka,
Validator

Date

Annual Review:

Reviewed By

Date Reviewed

Approved By

Date Approved

Reviewed By

Date Reviewed

Approved By

Date Approved

Annual Review (continued):

UNCONTROLLED DOCUMENT: DO NOT Transfer or Print

Method: 02-5035
Revision: 0
Date: January 22, 2007
Page 2 of 32

Reviewed By

Date Reviewed

Approved By

Date Approved

Reviewed By

Date Reviewed

Approved By

Date Approved

Reviewed By

Date Reviewed

Approved By

Date Approved

Reviewed By

Date Reviewed

Approved By

Date Approved

Reviewed By

Date Reviewed

Approved By

Date Approved

TABLE OF CONTENTS

1 Scope and Application4

2 Summary of Method5

3 Interferences6

4 Safety6

5 Apparatus and Materials7

6 Reagents8

7 Instrument Calibration9

8 Quality Control10

9 Sample Collection, Preservation and Handling10

10 Procedure15

11 Calculations21

12 Reporting Results22

13 Waste Disposal22

14 Pollution Prevention22

15 Definitions22

16 Troubleshooting22

APPENDIX A - Bottle Preparation Log-Sheet23

APPENDIX B - Sample Collection Procedure24

APPENDIX C - Sample Label29

SOP Change History Summary30

SOP Concurrence Form31

1 Scope and Application

- 1.1 This standard operating procedure is adapted from SW-846, Method 5035A-1, Draft Revision 1, July 2002, "Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples".
- 1.2 Method 5035A-1 was designed for use with solid materials (e.g., soils, sediments, and solid waste) containing low levels of volatile organic compounds (VOCs) using a closed-system purge-and-trap process. Procedures for collecting and preparing solid and oily waste samples containing high levels of VOCs are also included in the method. In regards to the high level concentration of VOCs, sample introduction is performed using Method 5030, which is used in conjunction with method 02-8260B and 8021B. For a list of compounds that are determined by this method, refer to the SOP for method 02-8260B or 8021B.
- 1.3 This sample vial preparation procedure is restricted for use by or under the supervision of analysts trained for volumetric and gravimetric procedures. The analysis portion of the procedure is restricted for use by or under the supervision of analysts trained on the use of the GC and/or GC/MS.
- 1.4 This method covers the determination of volatile organics compounds in soil and waste samples.
- 1.5 The applicable concentration range of the low-concentration procedure is dependent on the determinative method, matrix, and compound; however, it will generally fall in the 0.5 to 200 µg/kg range. The high-concentration procedure is intended for samples containing VOCs greater than 200 µg/kg.
- 1.6 Method Detection Limits can be found on the ALSI network in the GCMS folder and are maintained and updated by the QA department. The detection limits for a specific sample may differ from those listed due to the nature of interferences in a particular sample matrix. MDL studies must be performed according to SOP 99-MDL or the reference method, whichever is more frequent.
- 1.7 This document states the laboratory's policies and procedures established in order to meet requirements of all certifications/accreditations currently held by the laboratory, including the most current NELAC standards.
- 1.8 Individual project requirements may override criteria listed in this SOP.

2 Summary of Method

- 2.1 Volatile organic compounds are determined by collecting a 5 gram solid sample and placing it in a pre-weighed vial. For low-concentration samples, the vial contains a stirring bar and 5 mL of sodium bisulfate preservation solution. For high-concentration samples, the vial contains 5 mL of methanol only. The sample may be collected as a bulk sample in a soil jar without preservative. Once in the laboratory, an aliquot of that sample would be weighed and extracted in the proper preservative within 48 hours of collection.
- 2.2 For low-concentration solid samples, the entire vial is then placed into the instrument auto-sampler. Immediately before analysis, organic-free water, surrogates, and internal standards are automatically added without opening the sample vial.
- 2.3 The vial containing the sample is heated to 40°C and the volatiles purged into an appropriate trap using an inert gas combined with agitation of the sample. When purging is complete, the trap is heated and back flushed with helium to desorb the trapped sample components into a gas chromatograph for analysis by an appropriate determinative method such as 8021 or 8260.
- 2.4 For high concentration solid samples, the sample is extracted with a water-miscible solvent such as methanol or polyethylene glycol and purged via method 5030C. Surrogates will be added prior to analysis to determine if matrix effects are present. An aliquot of the solution is added to 5 mL of reagent water and the sample is purged under the same conditions as described under method 8260B.

NOTE: The method allows surrogates to be spiked into the solvent at the time of extraction or into the reagent water containing the aliquot prior to analysis. Since we are concerned with matrix effect or extraction efficiencies more than analytical efficiencies, we spike the surrogates into the solvent containing the soil and then take an aliquot out to add to reagent water for purging. The only exception to this would be an oily solvent, which could require dilutions large enough to dilute the added surrogates out of solution. In this case, the Archon Autosampler would add the surrogates at the time of analysis.

- 2.5 For high concentration oily waste samples, the sample is collected as a bulk sample without any type of preservation. The sample is tested for solubility with a water-miscible solvent like methanol. The sample is then diluted with the appropriate solvent, and an aliquot of the dilution is added to 5 mL of reagent water. The sample is then analyzed using the conditions described in method 8260B.

Method: 02-5035
Revision: 0
Date: January 22, 2007
Page 6 of 32

- 2.6 Samples that contain oily materials that are not soluble in water-miscible solvents must be extracted according to Method 3585 prior to the determinative procedure.

3 Interferences

- 3.1 Impurities in the purge gas and from organic compounds out-gassing from the plumbing ahead of the trap are a potential contamination problem. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running method blanks.
- 3.2 Samples can be contaminated by diffusion of volatile organics through the septum seal of the sample vial during shipment and storage. A trip blank prepared from organic-free reagent water and carried through sampling and handling protocols serves as a check on such contamination. Storage blanks are also analyzed monthly to ensure absence of contamination in the storage location.
- 3.3 Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed in sequence. Where practical, samples with unusually high concentrations of analytes shall be followed by an analysis of organic-free reagent water to check for cross-contamination.
- 3.4 Sample bottle preparation and volatile analysis shall be performed in a laboratory area that is free of solvents which interfere with the analytes being measured.

4 Safety

- 4.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical shall be regarded as a potential health hazard and exposure shall be as low as reasonably achievable. Cautions are included for known extremely hazardous materials or procedures.
- 4.2 Each analyst shall become familiar with the reagents used by referencing the Material Safety Data Sheets (MSDS) for each reagent. In doing so, the analyst will become familiar with the appropriate precautions for each reagent. MSDS are available to all staff and are located in hard copy format in the QA office and electronically on the ALSI public network, F:\MSDS - Material Safety Data Sheets.
- 4.3 The laboratory operates under a formal safety plan. All laboratory personnel must be familiar with the laboratory environmental health and safety plan described in the ALSI Quality Assurance Plan.

- 4.4 The following chemicals have the potential to be highly toxic or hazardous. Consult MSDS: methanol, polyethylene glycol, sodium bisulfate, benzene, carbon tetrachloride, chloroform, 1,4-dichlorobenzene and vinyl chloride
- 4.5 Analysts must wear a buttoned lab coat, safety glasses, and latex gloves at all times during the analysis.

5 Apparatus and Materials

- 5.1 40-mL, screw-cap, PTFE lined, septum-sealed vials. VWR #IRS136-0040, or equivalent
- 5.2 4 or 8 ounce wide-mouth glass bottle with PTFE lined screw-cap. VWR #IRV220-0125 or IRV220-0250, or equivalent
- 5.3 Balance - ACCULAB VI-200; 200 gm capacity; 0.01 gm resolution, or equivalent.
- 5.4 Volumetric flasks: Class A, various volumes, with ground-glass stoppers.
- 5.5 Disposable pipettes: Pasteur, VWR 5 3/4" #14672-200, or equivalent.
- 5.6 Magnetic stir bars – PTFE or glass coated, methanol rinsed. VWR #58949-036, or equivalent.
- 5.7 Syringes: 5 and 1 mL Hamilton Gastight: VWR #60376-321/285, or equivalent.
- 5.8 Microsyringes: Hamilton gastight, various volumes between 10 µL and 100 µL: VWR #60376-220,230,241,252,263,274, or equivalent.
- 5.9 Disposable pipettes: Pasteur, VWR 5 3/4" #14672-200, or equivalent.
- 5.10 Spatulas – stainless steel. VWR #57952-107, or equivalent.
- 5.11 Vials: 20 mL I-CHEM: VWR #IRS126-0020, or equivalent.
- 5.12 Purging device – closed system–Archon 5100. The purging device must be capable of accepting a vial large enough to contain a 5-gram soil sample plus a magnetic stirring bar and up to 10 mL of water. The device must also be capable of heating a soil vial to 40°C and holding it at that temperature while the inert purge gas is allowed to pass through the sample. The device shall also allow for the addition of internal and surrogate standards via the addition of 5

Method: 02-5035
Revision: 0
Date: January 22, 2007
Page 8 of 32

mL of reagent water while trapping the displaced headspace vapors. The device must be able to agitate the sample during the purging process. The analytes being purged must be quantitatively transferred to an absorbent trap. (See SOP for the determinative procedure for the types of traps used, GC/MS, and data system information.)

- 5.13 Pre-weighed 40-mL vials with 5 mL of methanol: C&G Containers, Inc. #R-B-1, or equivalent.

NOTE: In the above listings, the item noted may be replaced by a similar item of equivalent quality and function by another supplier if the need arises.

6 Reagents

- 6.1 Reagent grade inorganic chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all inorganic reagents shall conform to the specifications of The Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.
- 6.2 Organic-Free Reagent Water- Reagent water is water in which an interference is not observed with the analytes of interest. For this purpose, tap water is used. De-ionized water may also be used for this method if it has been shown to be free from interferences due to cartridge bleed.

NOTE: If interferences are detected in the de-ionized source, tap water must be used until the water source is again clean.
- 6.3 Methanol, Purge-and-Trap quality or equivalent. EM Science Purge & Trap grade, #MX0482-6, or equivalent. This methanol is stored at room temperature in the solvent storage cabinet in the GC department away from other solvents. See manufacturer's expiration dating, up to five years maximum for expiry.
- 6.4 Polyethylene Glycol (PEG) - free of interferences at the detection limit of the target analytes. Fisher #P167-1, or equivalent. PEG is stored at room temperature in the solvent storage cabinet in the GC department away from other solvents. See manufacturer's expiration dating, up to five years maximum for expiry.
- 6.5 Sodium Bisulfate - ACS reagent grade or equivalent. J.T. Baker #3534-01, or equivalent. Sodium Bisulfate is stored at room temperature in proximity to the GC/MS soil scale. See manufacturer's expiration dating, up to five years

maximum for expiry.

- 6.6 Hexadecane - free of interferences at the detection limit of the target analytes, for oily samples processed by method 3585. Aldrich #H6703-500ML, or equivalent. Hexadecane is stored at room temperature in the solvent storage cabinet in the GC department away from other solvents. See manufacturer's expiration dating, up to five years maximum for expiry.
- 6.7 Internal Standards and Surrogates are specified in the determinative procedure SOP.
 - 6.7.1 8021B Methanol extraction solution: Prepare a 3 µg/mL solution of α, α -trifluorotoluene from the 2000 µg/mL Ultra Scientific stock solution cat. #STS-220 or equivalent. Add 150 mL to 175 mL of purge and trap grade methanol to a 200 mL class A volumetric flask. Using a 500 µL gas tight syringe, transfer 300 µL of the purchased surrogate solution (STS-220) to the flask. When adding the α, α, α -trifluorotoluene stock to the volumetric flask, ensure that the needle of the syringe is below the surface of the methanol. Dilute to volume with methanol.
 - 6.7.2 8260B Methanol extraction solution: Refer to Section 10.4.2.4.
- 6.8 Calibration Solutions are listed in the determinative procedure SOP; 02-8260B for method 8260 and 01-8021 for method 8021.
- 6.9 Quality control sample solutions are listed in the determinative procedure SOP; 02-8260B for method 8260 and 01-8021 for method 8021.

7 Instrument Calibration

- 7.1 Refer to the standard operating procedure for the determinative methodology (SW-846 Method 8021 or 8260).
- 7.2 Instruments processing samples for the low-concentration samples shall be calibrated from approximately 0.5 to 200 µg/kg, when possible to do so without over-saturating the detector.

NOTE: For low level soil analysis by 8260/5035, the calibration range is 2-200 µg/kg for most compounds of interest. At this time, we do not analyze for low level soils using 8021 methodology. The 8260B SOP, 02-8260B, has instructions to calibrate in a range of 1-200 ppb. To prepare a 2 ppb standard, simply double the amount of standard added for the 1 ppb standard (8260B Appendix A, pg. 39)

7.2.1 All calibration samples are prepared by adding 5 mL of the calibration solution to a 40 mL vial with 1 g of sodium bisulfate and a magnetic stirring bar before placing on the Archon auto sampler.

8 Quality Control

8.1 All policies and procedures in the most current revision of the ALSI QA Plan shall be followed when performing this procedure.

8.2 A trip blank must be prepared with each set of bottles. This blank will accompany the samples to the collection site and back to the laboratory. It will be analyzed to determine if any contamination has occurred during preparation of the sample containers. The trip blank shall consist of two (2) 40 mL vials. Prepare one for the low-concentration procedure by adding 5 mL of reagent water, 1 g of sodium bisulfate, and a magnetic stirring bar into a 40 mL vial. The other vial shall be prepared for the high-concentration procedure by adding 5 mL of methanol to a 40 mL vial.

8.3 For information on method blanks, duplicates, matrix spikes, internal standards, surrogates, QC standards, proficiency tests, DOCs, ongoing proficiency and MDLs, refer to the standard operating procedure for the determinative procedure; 02-8260B for method 8260 and 01-8021 for method 8021.

8.3.1 For low level concentration soils, all QC samples are prepared by adding 5 mL of QC solution to a 40 mL vial with 1 g of sodium bisulfate and a magnetic stir bar before placing on the Archon auto sampler.

9 Sample Collection, Preservation and Handling

9.1 Preparation of sample collection containers by the Sample Receiving department

9.1.1 A variety of sample containers are required for “low-concentration” and “high-concentration” samples. Being that the concentration of samples cannot be accurately predicted prior to sample collection, a set of 4 sample containers will be prepared and furnished for each sample to be collected.

9.1.1.1 Two (2) 40 mL vials will be prepared for “low-concentration” samples.

9.1.1.2 For each set of vials being furnished to a customer, an additional two (2) 40 mL low-concentration vials shall be provided in the sample collection kit. These extra 2 vials are for internal QC

measurements.

9.1.1.3 One (1) 40 mL vial will be prepared for “high-concentration” samples, and preserved with 5 mL of methanol.

9.1.1.4 For each set of vials being furnished to a customer, an additional two (2) 40 mL high-concentration vials shall be provided in the sample collection kit. These extra 2 vials are for internal QC measurements.

9.1.1.5 One (1) four ounce wide mouth jar shall be furnished for the moisture determination of solid matrices, and can be used for the collection of “oily” samples.

9.1.1.6 Furnish trip blanks for each set of bottles being prepared. The trip blank shall consist of one 40 mL vial prepared according to the low-concentration procedure. (1 gram of sodium bisulfate and 5 mL of organic-free water). Another trip blank shall be prepared according to the high-concentration procedure. The trip blank bottles shall be labeled “Trip Blank – Do Not Open”.

9.1.1.7 Zip-lock bags shall be furnished to prevent the sample bottle labels from picking up moisture from the ice used for cooling.

9.1.1.8 The customer shall also be provided with a copy of the “Method 5035 Sample Collection Procedure” and a copy of the completed “Method 5035 Bottle Preparation Log-Sheet”.

9.1.1.9 If sample weights will be approximated with plastic syringes, a 5 or 10 mL syringe shall be included for each sample site. The tip of each syringe will be cut off at the 0 mL mark prior to sampling.

9.1.2 Preparation of Low-Concentration Soil Sample Vials Collected and Preserved in the Field (pre-weighed)

NOTE: Wear gloves during all steps in this procedure to ensure that no additional weight is transferred to the sample container from contact.

9.1.2.1 Add a clean, methanol rinsed and dried, magnetic stirring bar to each 40 mL pre-cleaned vial.

9.1.2.2 Add approximately 1 gram of sodium bisulfate to each vial.

Method: 02-5035
Revision: 0
Date: January 22, 2007
Page 12 of 32

NOTE: If samples markedly smaller or larger than 5 g are to be collected, adjust the amount of preservative added to correspond to approximately 0.2 g of preservative for each 1 g of sample to ensure a sample pH of ≤ 2 .

9.1.2.3 Add 5.0 mL of organic-free reagent water to each vial.

9.1.2.4 Seal the vial with the screw-cap and septum seal.

9.1.2.5 Affix a sample bottle label to each vial and number with a unique number. Place the label as far down on each vial as possible to allow for attachment of a second label without obscuring the first label. The labels have spaces for recording: vial-number; initial weight (g); final weight (g); and sample size (g). See Appendix C for an example of an appropriate label. For vials preserved with sodium bisulfate, an "S" shall then be followed by a sequential log number.

9.1.2.6 Indicate on the label that the vial is preserved with sodium bisulfate.

9.1.2.7 Weigh the prepared vial to the nearest 0.01 gram and write the weight on the sample bottle label. (When the vials are returned to the laboratory for analysis, do not add any additional labels to the vial until the final weighing is completed).

NOTE: Purchased pre-weighed vials may be used in certain circumstances. If so, ignore sections prior to Section 9.1.2.8. The purchased pre-weighed vials are labeled with the initial weight.

9.1.2.8 Record the vial number and weight on "Method 5035 Bottle Preparation Log-Sheet". After recording the initial information, a copy of the log-sheet shall be furnished to the customer and to the Billing Department. The original shall be filed in the Sample Receiving Department to await the return of the sample containers. If weights are calculated by the Sample Receiving Department, write the weight on the vial as well as the prep log sheet under sample size.

NOTE: Method 5035 bottle prep log sheets will be kept in a spreadsheet file in the Sample Receiving Department entitled 5035 Bottle Prep.

Method: 02-5035
Revision: 0
Date: January 22, 2007
Page 13 of 32

9.1.2.9 Because volatile organics will partition into the headspace of the vial from the aqueous solution and will be lost when the vial is opened, surrogates, internal standards, and matrix spikes (if applicable) shall only be added to the vials after the sample has been added to the vial. The matrix spike solution needs to be added manually through the septum using a small gauge needle while the surrogates and internal standards will be added by the Archon Autosampler at the time of analysis.

9.1.3 Preparation of High-Concentration Soil Samples Vials collected and preserved in the field (pre-weighed).

9.1.3.1 Use a 40 mL pre-cleaned vial. A 20 mL vial would be preferable to minimize headspace, but due to the size of the attached label, a 40 mL vial is used.

9.1.3.2 Add 5.0 mL of purge-and-trap Methanol to each vial (unless otherwise noted in logbook).

NOTE: For analyses using Method 8021 procedures, 10 mL of methanol gets added to the vial.

9.1.3.3 Seal the vial with the screw cap and septum seal.

9.1.3.4 Affix a sample bottle label to each vial and number with a unique number. Place the label as far down on each vial as possible to allow for attachment of a second label without obscuring the first label. The labels have spaces for recording: vial-number; initial weight (g); final weight (g); and sample size (g). See Appendix C for an example of an appropriate label. For vials preserved with methanol, an "M" shall then be followed by a sequential log number.

9.1.3.5 Indicate on the label that the vial is preserved with methanol.

9.1.3.6 Weigh the prepared vial to the nearest 0.01-gram and write the weight on the sample bottle label. (When the vials are returned to the laboratory for analysis, do not add any additional labels to the vial until the final weighing is completed).

NOTE: Purchased pre-weighed vials may be used in certain circumstances. If so, ignore sections prior to Section 9.1.3.7. The purchased pre-weighed vials are labeled with the initial weight .

Method: 02-5035

Revision: 0

Date: January 22, 2007

Page 14 of 32

NOTE: Vials containing methanol shall be weighed a second time on the day that they are used to determine if methanol has been lost. This step is especially important with purchased pre-weighed vials. If there is a reduction in weight of more than 0.01 g, that vial shall not be used for sample collection. This would require a balance in the field to achieve this, which is not always possible so this is more a recommendation than a requirement.

9.1.3.7 Record the vial number and weight on “Method 5035 Bottle Preparation Log-Sheet”. After recording the initial information, a copy of the log-sheet shall be furnished to the customer and to the Billing Department. The original shall be filed in the sample-receiving department to await the return of the sample containers. If weights are calculated by the Sample Receiving Department, write the weight on the vial as well as the prep log sheet under sample size.

NOTE: Method 5035 bottle prep log sheets will be kept in a spreadsheet file in the Sample Receiving Department entitled 5035 Bottle Prep.

9.1.3.8 Surrogates, internal standards and matrix spikes (if applicable) shall be added to the sample after it is returned to the laboratory and prior to analysis. The exception to this rule would be for New Jersey pre-weighed vials, which require 25 mL of methanol containing surrogates in the vial as it leaves the laboratory.

9.1.4 Preparation of Low and High Concentration Soils, Oily Samples, and Moisture Determination Bottles That Are Not Preserved in the Field.

9.1.4.1 A pre-cleaned, unpreserved, four (4) ounce wide-mouth jar with a PTFE lined screw-cap, shall be furnished. Extraction of these samples occurs within 48 hours of receipt at the laboratory.

9.2 Sample collection:

9.2.1 Samples shall be collected in the containers described in Section 9.1. All customers shall be provided with a copy of the “Method 5035 Sample Collection Procedure”. Field personnel shall add approximately 5 g of soil to each pre-weighed, labeled vial. Approximate weights can be obtained by use of a balance in the field or by using a 5 mL plastic syringe with the tip cut off. In this case the syringe shall be filled to the 3.5 or 4.0 mL mark. **(DO NOT** add more than this as it will affect

Method: 02-5035
Revision: 0
Date: January 22, 2007
Page 15 of 32

recoveries adversely.). Field personnel must not add any additional labels to the sample vials, as the weight of the label will be included in the sample size.

NOTE: When collecting low-level soil samples, be sure not to collect more than indicated. Collection of too much soil may invalidate the test.

- 9.2.2 Samples may also be collected in devices such as the EnCore sampler. See Section 10.4 for more information concerning the preparation of EnCore samples collected.
- 9.2.3 All samples collected for volatiles analysis shall be cooled to above the freezing point of water up to 6°C, packed in a cooler, and shipped to the laboratory on ice. All vials shall be sealed in zip-lock bags to prevent contact with wet ice.
- 9.2.4 When collecting samples that are not in pre-weighed vials, be sure to fill soil to top of jar. Headspace can cause loss of analytes of interest.

10 Procedure

10.1 Low-Concentration Samples

- 10.1.1 Upon return of the samples to the laboratory for analysis, the final sample weights shall be determined by the sample-receiving department. Do not add any additional labels to the vials until the final weight is determined.
- 10.1.2 The final vial weight shall be determined on a top-loading balance accurate to 0.01 grams. The final weight shall be recorded on the “Method 5035 Bottle Preparation Log-Sheet”. Subtracting the initial and final vial weights will yield the sample size. A copy of the completed log-sheet shall be forwarded to the GC and GC/MS departments. The sample size shall be recorded on the vial as well.
- 10.1.3 In the event that the samples were collected and submitted in devices such as the EnCore sampler, the GC or GC/MS department must, within 48 hours of sample collection, transfer the sample to low-concentration vials. See Section 10.4 for the procedure.
- 10.1.4 In the event that the samples were collected and submitted in sample collection jars (Section 9.1.4), the GC or GC/MS department must, within 48 hours of sample collection, transfer 5 g of the sample to the appropriate pre-weighed vial described in Section 9.

Method: 02-5035
Revision: 0
Date: January 22, 2007
Page 16 of 32

NOTE: If the extraction does not occur within the 48 hours of collection, but the soil sample was received by the laboratory within that time, a JAR comment is used in the Horizon LIMS software. If the soil sample was received by the laboratory after 48 hours and extraction occurs, a JAX comment is used in the Horizon LIMS software.

10.1.5 Within 14 days of sample collection, samples must be purged in the closed system purge-and-trap and analyzed by 8260.

10.1.6 The customer shall provide 2 low-concentration vials preserved with sodium bisulfate, containing 5 grams of sample and 1 high-concentration vial preserved with methanol, containing 5 grams of sample. In the event that analyte concentrations exceed 200 µg/kg, the sample shall be re-analyzed using the High-concentration procedure.

10.2 High-Concentration Samples

10.2.1 Samples with analyte concentrations that exceed the calibration range of the low-concentration procedure, or are greater than 200 µg/kg, shall be processed by the methanol extraction procedure, purged and analyzed according to the standard operating procedure for method 8021 or 8260. Polyethylene glycol may be substituted for methanol.

10.2.2 Weigh the prepared vial to the nearest 0.01 gram and write the weight on the sample bottle label. (When the vials are returned to the laboratory for analysis, do not add any additional labels to the vial until the final weighing is completed). Calculate the sample size (g) by subtracting initial weight (g) from final weight (g). Record the sample size (g) on the sample vial label.

10.2.3 Record the vial number, final weight (g) and sample size (g) on “Method 5035 Bottle Preparation Log-Sheet”. After recording the initial information, a copy of the log-sheet shall be furnished to the customer. The original shall be filed in the sample-receiving department to await the return of the sample containers.

10.2.4 Prior to analysis, the GC/MS department will add surrogates to the methanol-preserved vials if the sample requires 8260 methodology or, the GC department will add appropriate surrogates if 8021 methodology is requested.

NOTE: See Section 10.4.2 for amounts and type of surrogate solution to be added.

Method: 02-5035
Revision: 0
Date: January 22, 2007
Page 17 of 32

10.2.5 In the event that the samples were collected and submitted in an EnCore device, the GC or GC/MS department must, within 48 hours of sample collection, transfer the samples to high concentration vials. See Section 10.4 for the appropriate procedure.

10.2.6 In the event that the samples were collected and submitted in sample collection jars (Section 9.1.4), the GC or GC/MS department must, within 48 hours of sample collection, transfer 5 g of the sample to the appropriate pre-weighed vial described in Section 9.

NOTE: If the extraction does not occur within the 48 hours of collection, but the soil sample was received by the laboratory within that time, a JAR comment is used in the Horizon LIMS software. If the soil sample was received by the laboratory after 48 hours and extraction occurs, a JAX comment is used in the Horizon LIMS software

10.2.7 Within 14 days of sample collection, a portion of the methanol extract with surrogates present is added to reagent water to make a 1:50 final dilution. This dilution is added to the Archon auto sampler and analyzed according to the determinative procedure.

NOTE: Larger dilutions of the methanol extract may be required to ensure that the analytes of interest are within the curve limits. See the SOP for the determinative procedure on preparations of dilutions. If the surrogates are diluted below the limit of the curve, and recovery cannot be quantitated, a SDO comment is used in the Horizon LIMS software.

10.3 Oily Samples

10.3.1 Oily, non-water-miscible samples shall be extracted with hexadecane according to the standard operating procedure for method 3585. (See Section 10.4.4)

10.4 EnCore Procedure

10.4.1 Low concentration soil samples

10.4.1.1 Add a clean, methanol rinsed and dried, magnetic stirring bar to each clean, 40 mL vial needed. With cap removed, place vial with stir bar on the balance and tare to 0.

10.4.1.2 Add approximately 1 gram of sodium bisulfate to the vial. Again, tare the vial.

Method: 02-5035

Revision: 0

Date: January 22, 2007

Page 18 of 32

- 10.4.1.3 Open the EnCore sample device with pliers or a screwdriver, and insert the soil plug into the tared 40 mL vial containing sodium bisulfate and a stir bar. Carefully and quickly clean off the lip of the vial and the threads of the vial with a clean cloth (Kimwipe) to remove any dirt, which could cause a leak resulting in lower recoveries than actually present. Weigh the vial and record the weight in the 8260A Extraction Logbook. This must be done within 48 hours of collecting the sample.

NOTE: Soil samples that contain carbonate minerals may effervesce upon contact with the acidic preservative solution. If the amount of gas generated is very small, any loss of volatiles may be minimal if the vial is sealed quickly. However, if larger amounts of gas are generated, the sample may lose significant amounts of VOCs and may even cause the vial to shatter once the vial is closed. To avoid this, when samples are known or are discovered to contain high levels of carbonates, the sample shall be placed in unpreserved reagent water and a comment shall be placed on the lab report stating why the sample is unpreserved. The sample must be analyzed within seven days if unpreserved.

- 10.4.1.4 Label the vial with the corresponding COC# of the sample that is to be prepared. If two EnCore plugs are sent in for low-level analysis, label the COC#s with a B and a C at the end of the number. (Ex: 123456-1B)

- 10.4.1.5 Add 5.0 mL of organic-free reagent water to each vial and snugly cap the vial. The vial will not be reopened until after the analysis is completed. The Archon Auto sampler adds internals and surrogates at the time of the analysis.

- 10.4.1.6 For samples not collected in an EnCore sampling device, an aliquot of soil (approximately 5 g) shall be taken from the soil jar and prepped according to the instructions found in Sections 10.4.1.1 – 10.4.1.5. The prep needs to be done within 14 days of collection as well as the analysis.

NOTE: Sample aliquots taken from the soil jar shall be prepared as soon as possible to avoid loss of volatiles and contamination that may occur during the percent moisture determination step.

Method: 02-5035
Revision: 0
Date: January 22, 2007
Page 19 of 32

10.4.2 High concentration soil samples

10.4.2.1 Remove the cap from a clean 20 mL vial, place the vial on the balance and tare to zero. 20 mL vials are preferred over the 40 mL size because they have less headspace than the 40 mL vials.

10.4.2.2 Label the vial with the corresponding COC# of the sample that is to be prepared. Usually one EnCore plug is sent in for high concentration analysis. Label the COC# with an "A" at the end of the number.

10.4.2.3 Open the EnCore sample device with pliers or a screwdriver, and insert the soil plug into the tared 20 mL vial. Weigh the vial and record the weight in the 8260A Extraction Logbook or the 8021B extraction logbook.

10.4.2.4 Preparation for 8260 analysis. Unlike the low-level soil preparation, the high level preparation gets surrogates added to the vial at the time of preparation, not at the time of analysis if the sample is to be analyzed by GC/MS. This is accomplished using one of the following methods.

10.4.2.4.1 5.0 mL of purge and trap grade methanol containing surrogates, MED826SS, is added to the vial. The portion of methanol must be prepared so that the addition of 5.0 mL of MED826SS will yield a concentration of 30 µg/kg when analyzed at a 50 fold dilution. MED826SS is prepared by adding 0.6 mL of Restek 8260A Surrogate Mix (2500 ppm), catalog #30240, to purge and trap grade methanol to a final volume of 1000 mL. Cap and shake. This is the preferred method of preparation.

10.4.2.4.2 After addition of 5.0 mL of unfortified methanol, add 3 µL of Restek 8260A Surrogate Mix (2500 ppm), catalog #30240, to the vial, which will yield a concentration of 30 ppb when analyzed. Cap and shake. This method shall be used when MED826SS is not available or when pre-weighed methanol vials are submitted by the client. If more than five mL of methanol is added, the

Method: 02-5035

Revision: 0

Date: January 22, 2007

Page 20 of 32

8260A surrogate mix added shall be increased to keep the ratio to 3 μ L added for every 5 mL of methanol.

10.4.2.5 Preparation for 8021B analysis: Unlike the 8260B low level soil preparation, the high level preparation requires surrogates to be added to the sample at the time of preparation, not at the time of analysis. This is accomplished using one of the following methods:

10.4.2.5.1 Add 5.0 mL of the 8021B fortified methanol extraction solution (Section 6.7.1) to the VOA vial containing the weighed soil from the Encore sampler or other sample device or container. Cap the VOA vial and shake. **Store above the freezing point of water up to 6°C and analyze within fourteen days of sample collection.**

10.4.2.5.2 If pre-weighed VOA vials of methanol are submitted to the laboratory with unfortified methanol, use the following procedure. Using a 10 μ L gas tight syringe, transfer 7.5 μ L of 2000 μ g/mL α , α , α -trifluorotoluene purchased stock (Ultra Scientific, catalog #STS-220, or equivalent) to the methanol. Ensure that the needle of the syringe is below the surface of the methanol when adding the surrogate solution. Cap the VOA vial and shake. Store above the freezing point of water up to 6°C and analyze within fourteen days of when the soil sample was collected.

10.4.3 Method for deciding how EnCores shall be prepared:

10.4.3.1 Normally, a client will submit three EnCore plugs per COC#. Two plugs shall be prepared in sodium bisulfate for low level analysis and one plug shall be prepared in methanol for high level analysis to be used in the event that the low level analysis contains over-range compounds. The high level preparation will be labeled as the "A" sample, and the low level preparations will be labeled as the "B" and "C" samples.

10.4.3.2 In the event that the client sends in only two EnCore plugs, prepare one in sodium bisulfate and one in methanol.

Method: 02-5035

Revision: 0

Date: January 22, 2007

Page 21 of 32

NOTE: Some clients sending in 2 EnCores want both prepared in sodium bisulfate to be analyzed as low level soils. See Project Coordinators for information on these specific clients.

10.4.3.3 If only one EnCore plug is submitted, it will be prepared in methanol only.

10.4.4 To determine if the soils submitted get 8260 or 8021, consult the LIMS, the chain of custody, or speak to the project manager. If in doubt, or if low-level detections are required, prepare for analysis by 8260.

10.4.5 Oily Samples

10.4.5.1 For most of the cases this laboratory deals with, oily samples are submitted in 4 oz. jars or larger and therefore this SOP will not cover encore prep directions.

10.4.5.1.1 To prepare an oil sample that is soluble in methanol or PEG (polyethylene glycol), take a 20 mL volatile vial and add 10 mL of methanol. Mark the bottom of the meniscus with a permanent marker to indicate the 10 mL volume mark. Discard the methanol. In the marked vial, weigh out 1 gram of sample (wet weight) and record the weight in the extraction logbook. Add methanol to marked line to achieve an initial dilution of 1:10 (weight: volume) ratio.

10.4.5.1.2 Analyze the sample by diluting up to 1 mL of the extract into a final volume of 50 mL. Put the solution into a 40 mL VOA vial and put the vial into the archon autosampler to be analyzed as per method 8260B. Since many oil samples need dilutions high enough that surrogates would be diluted out of solution, we allow the archon to add the surrogates.

11 Calculations

11.1 Calculations are performed according to the standard operating procedure for method 8021 or 8260; 02-8260B for method 8260 and 01-8021 for method 8021.

UNCONTROLLED DOCUMENT: DO NOT Transfer or Print

Method: 02-5035
Revision: 0
Date: January 22, 2007
Page 22 of 32

Method: 02-5035
Revision: 0
Date: January 22, 2007
Page 23 of 32

12 Reporting Results

12.1 Report results according to the standard operating procedure for methods 8021 or 8260; 02-8260B for method 8260 and 01-8021 for method 8021.

13 Waste Management

13.1 Refer to ALSI SOP 19-Waste Disposal.

14 Pollution Prevention

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. Management shall consider pollution prevention a high priority. Extended storage of unused chemicals increases the risk of accidents. The laboratory shall consider smaller quantity purchases which will result in fewer unused chemicals being stored and reduce the potential for exposure by employees. ALSI tracks chemicals when received by recording their receipt in a traceable logbook. Each chemical is then labeled according to required procedures and stored in assigned locations for proper laboratory use.

15 Definitions

15.1 Refer to ALSI QA Plan under Laboratory Quality Control Checks for general definitions.

16 Troubleshooting

16.1 Refer to maintenance logs and instrument manuals for guidance in troubleshooting specific problems related to the instrumentation used in this method.

Method: 02-5035
 Revision: 0
 Date: January 22, 2007
 Page 24 of 32

APPENDIX A
Method 5035 Bottle Preparation Log-Sheet

Customer/Project Name: _____ COC/Proj. #: _____

Vial Preparation:

Date Prepared: _____
 Prepared By: _____
 Sodium Bisulfate #: _____
 Methanol #: _____

Final Weight:

Date Weighed: _____
 Weighed By: _____

Sample/Vial Cross-Reference

Customer: Please use **“Unshaded”** columns only.

Vial Number	Sample Identification	Vial sent out	8260 or 8021	Lab Sample #	Initial Wt, g	Field Wt, g	Final Lab Wt, g	Sample Size, g
S								
S								
S								
S								
S								
S								
S								
S								
S								
S								
S								
M								
M								
M								
M								
M								
M								

Method: 02-5035
Revision: 0
Date: January 22, 2007
Page 25 of 32

APPENDIX B



Method 5035 Sample Collection Procedure

Samples may be collected by one of two procedures. Below, are instructions for collecting samples with the Encore Sampler and with Pre-weighed vials.

Sampling With the Encore Sampler or Similar Device

Follow the instructions provided by the manufacturer, for collecting the sample.

When Collecting Samples for the UST Soil-Leaded Gas and Land Re-use Projects:

- For each sample being collected, you must collect 3 portions of soil with 3 Encore sampling devices.
- For one sample out of the group, you must collect 7 portions of soil with 7 Encore sampling devices. The extra sample is for laboratory quality control such as the matrix spike and matrix spike duplicate
- Also collect one 4 ounce widemouth jar of each sample being collected.

When Collecting Samples for All UST Samples Other Than Leaded Gas:

- For each sample being collected, you must collect 1 portion of soil with 1 Encore sampling device.
- For one sample out of the group, you must collect 3 portions of soil with 3 Encore sampling devices. The extra sample is for laboratory quality control such as the matrix spike and matrix spike duplicate
- Also collect one 4 ounce widemouth jar of each sample being collected.

When submitting samples to the lab, please specify the purpose of the project. (UST parameters or Land Re-use).

Method: 02-5035
Revision: 0
Date: January 22, 2007
Page: 26 of 32

APPENDIX B



Method 5035 Sample Collection Procedure

Using Pre-weighed Containers for UST Soil-Leaded Gas and Land Re-use Projects

Contents of Sampling Kit:

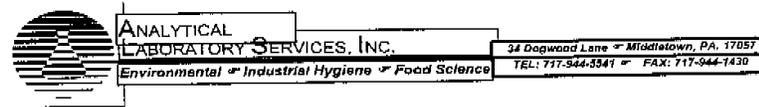
- For each sample being collected, there are two (2) 40ml pre-weighed vials with containing a magnetic spin bar, sodium bisulfate preservative and 5 ml of organic-free water. These vials are numbered and begin with the letter "S".
- For each sample being collected, there is one (1) 40ml pre-weighed vial containing 5ml of Methanol. These vials are numbered and begin with the letter "M".
- For each sample being collected, there is one (1) 4 ounce widemouth jar.
- Two (2) Extra "S" 40 ml vials. These are for internal QC checks such as the matrix spike and matrix spike duplicate. For each set of 20 or less samples being collected, use these extra 2 vials to collect a total of 4 "S" vials for one of the samples. Make sure that all four vials are labeled with the sample location.
- Two (2) Extra "M" 40 ml vials. These are for internal QC checks such as the matrix spike and matrix spike duplicate. For each set of 20 or less samples being collected, use these extra 2 vials to collect a total of 3 "M" vials for one of the samples.
- One (1) 40 ml "S" vial labeled as "Trip Blank". Do not open this vial. Return it to the laboratory.
- One (1) 40 ml "M" vial labeled as "Trip Blank". Do not open this vial. Return it the laboratory.
- A Copy of "Method 5035 Bottle Preparation Log-Sheet"
- This instruction sheet

Sampling Procedure:

- Always wear gloves when handling the pre-weighed vials
- Use new or decontaminated sampling equipment for each new sampling location. (i.e.: new Encore sampler or new syringe)
- Do not add any additional labels to the pre-weighed vials
- For each sample being collected, you will use two(2) of the "S" vials, one (1) of the "M" vials, and one (1) 4 ounce wide-mouth jar.
- Add about 5 grams of soil to each of the pre-weighed sample vial. Be careful not to splash any of the preservative solution out of the vial. If any solution is lost, the vial must be discarded and not used. For most soils, 5 grams is approximately a "sewing thimble" full. (The method also permits the use of a cutoff hypodermic syringe for sampling and estimating the 5 grams of soil by collecting soil to the 3.5 to 4 mL mark.) Be sure not to add more than the necessary amount as this will adversely affect the results.
- Quickly brush any soil off the vial threads and immediately seal the lid.
- When practical, use a portable balance to weigh the sealed vial to ensure that 5 +/- 0.5 grams of soil was added to the vial. If you go over 5 grams, do not remove soil from the vial.

Method: 02-5035
Revision: 0
Date: January 22, 2007
Page: 27 of 32

APPENDIX B



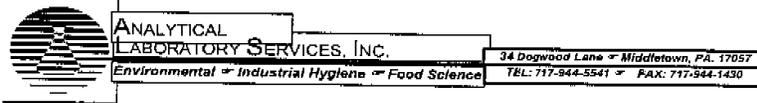
Method 5035 Sample Collection Procedure

Using Pre-weighed Containers for UST Soil-Leaded Gas and Land Re-use Projects Continued...

- The "Method 5035 Bottle Preparation Log-Sheet" lists the serial number of each vial furnished in the sample collection kit. Under the heading "Sample Identification", list your sample identification name next to the correct serial number for each "S" and "M" vial used for each sample.
- If the vial was weighed in the field, record the weight under the column "Field Wt".
- Fill the 4 ounce widemouth jar completely full, with no air space.
- For each group of 20 or less samples being collected, one of the samples must use 2 extra "S" vials and 2 extra "M" vials. This is for internal QC measurements such as the matrix spike and matrix spike duplicate.
- The vials should be sealed in zip-lock bags and stored on ice until delivery to the laboratory.
- Do not get the bottle labels wet, as they will increase in weight from the moisture.

Method: 02-5035
Revision: 0
Date: January 22, 2007
Page: 28 of 32

APPENDIX B



Method 5035 Sample Collection Procedure

Using Pre-weighed Containers for UST Soils Other Than Leaded Gas:

Contents of Sampling Kit:

- For each sample being collected, there is one (1) 40ml pre-weighed vial containing 5ml of Methanol. These vials are numbered and begin with the letter "M".
- For each sample being collected, there is one (1) 4 ounce widemouth jar.
- Two (2) extra "M" 40 ml vials. These are for internal QC checks such as the matrix spike and matrix spike duplicate. For each set of 20 or less samples being collected, use these extra 2 vials to collect a total of 3 "M" vials for one of the samples. Ensure that all three vials are labeled with the sample location.
- One (1) 40 ml "M" vial labeled as "Trip Blank". Do not open this vial. Return it to the laboratory.
- A Copy of "Method 5035 Bottle Preparation Log-Sheet"
- This instruction sheet

Sampling Procedure:

- Always wear gloves when handling the pre-weighed vials
- Use new or decontaminated sampling equipment for each sampling location. (i.e.: new Encore sampling device or syringe)
- Do not add any additional labels to the pre-weighed vials
- For each sample being collected, you will use one (1) of the "M" vials, and one (1) 4 ounce wide-mouth jar.
- Add about 5 grams of soil to the pre-weighed sample vial. Be careful not to splash any of the preservative solution out of the vial. If any solution is lost, the vial must be discarded and not used. For most soils, 5 grams is approximately a "sewing thimble" full. If using a syringe, add 3.5 to 4 mL of soil to approximate 5 grams.
- Quickly brush any soil off the vial threads and immediately seal the lid.
- When practical, use a portable balance to weigh the sealed vial to ensure that 5 +/- 0.5 grams of soil was added to the vial.
- The "Method 5035 Bottle Preparation Log-Sheet" lists the serial number of each vial furnished in the sample collection kit. Under the heading "Sample Identification", list your sample identification name next to the correct serial number for each "M" vial used for each sample.
- If the vial was weighed in the field, record the weight under the column "Field Wt".
- Fill the 4 ounce widemouth jar completely full, with no air space.
- For each group of samples being collected, one of the samples must use 2 extra "M" vials. This is for internal QC measurements such as the matrix spike and matrix spike duplicate.
- The vials should be sealed in zip-lock bags and stored on ice until delivery to the laboratory.
- Do not get the bottle labels wet, as they will increase in weight from the moisture.

Method: 02-5035
 Revision: 0
 Date: January 22, 2007
 Page: 29 of 32

APPENDIX B

**Method 5035 Sample Collection Procedure
 Quick Summary Table**

Encore Sampling Devices	
Project Type:	UST Soil – Leaded Gas Projects Land Re-use Projects
For Each Sample:	3 Encore Samples 1 Four Ounce Wide-Mouth Glass Jar
For One Sample in Each Group of 20 Or Less Samples:	7 Encore Samples 1 Four Ounce Wide-Mouth Glass Jar
All UST Soil Projects Other than Leaded Gas	
Project Type:	All UST Soil Projects Other than Leaded Gas
For Each Sample:	1 Encore Sample 1 Four Ounce Wide-Mouth Glass Jar
For One Sample in Each Group of 20 Or Less Samples:	3 Encore Samples 1 Four Ounce Wide-Mouth Glass Jar
Using Lab-Prepared Pre-Weighed Bottles	
Project Type:	UST Soil – Leaded Gas Projects Land Re-use Projects
For Each Sample:	2 Sodium Bisulfate Preserved "S" Vials 1 Methanol Preserved "M" Vials 1 Four Ounce Wide-Mouth Glass Jar
For One Sample in Each Group of 20 Or Less Samples:	4 Sodium Bisulfate Preserved "S" Vials 3 Methanol Preserved "M" Vials 1 Four Ounce Wide-Mouth Glass Jar
All UST Soil Projects Other than Leaded Gas	
Project Type:	All UST Soil Projects Other than Leaded Gas
For Each Sample:	1 Methanol Preserved "M" Vials 1 Four Ounce Wide-Mouth Glass Jar
For One Sample in Each Group of 20 Or Less Samples:	3 Methanol Preserved "M" Vials 1 Four Ounce Wide-Mouth Glass Jar

Method: 02-5035
Revision: 0
Date: January 22, 2007
Page 30 of 32

APPENDIX C



Company Name: _____
Client Sample ID: _____
Date sampled: _____ Sampled By: _____
Time Sampled: _____ Preserved With: _____
Vial #: _____ Initial Wt: _____
Final Wt: _____ Final Sample Size: _____
Analysis: _____

34 Dagwood Lane + Middletown, PA 17057
TEL: 717-844-8841 + FAX: 717-844-1438

UNCONTROLLED DOCUMENT: DO NOT Transfer or Print

Method: 02-5035
Revision: 0
Date: January 22, 2007
Page 31 of 32

SOP Change History Sheet

<u>Section #</u>	<u>Section Description</u>	<u>Reason for Change</u>
<u>Revision 0: 01/22/2007</u>		New SOP

UNCONTROLLED DOCUMENT: DO NOT Transfer or Print

Method: 02-8260B
Revision: 9
Date: August 17, 2006
Page: 1 of 52

Document Title: Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique

Document Control Number: _____

Organization Title: ANALYTICAL LABORATORY SERVICE, INC. (ALSI)
Address: 34 Dogwood Lane
Middletown, PA 17057

Phone: (717) 944-5541

Approved by:

_____ Helen MacMinn, Quality Assurance Manager	_____ Date
_____ Clark Dougherty, GC/MS Supervisor	_____ Date
_____ Erin Ripka, Validator	_____ Date

Annual Review:

Reviewed By

Date Reviewed

Approved By

Date Approved

Reviewed By

Date Reviewed

Approved By

Date Approved

Method: 02-8260B
Revision: 9
Date: August 17, 2006
Page: 3 of 52

TABLE OF CONTENTS

1 Scope and Application 4

2 Summary of Method 6

3 Interferences 7

4 Safety 7

5 Apparatus and Materials 8

6 Reagents 10

7 Instrument Calibration 15

8 Quality Control 20

9 Sample Collection, Preservation and Handling 25

10 Procedure 27

11 Calculations 36

12 Reporting Results 36

13 Waste Disposal 36

14 Pollution Prevention 37

15 Definitions 37

16 Troubleshooting 37

Table 1 38

APPENDIX A 39

APPENDIX B 42

APPENDIX C 45

APPENDIX D 46

APPENDIX E 47

SOP Change History Summary 48

SOP Concurrence Form 52

Method: 02-8260B

Revision: 9

Date: August 17, 2006

Page: 4 of 52

1 Scope and Application

- 1.1 This standard operating procedure is adapted from "Test Methods for Evaluating Solid Waste Physical/Chemical Methods" (SW-846), Method 8260B, Revision 2, December 1996, Method 5035A, Revision 1, July 2002, and Method 5030C, Revision 3, December 2003. The method detection limits (MDLs) can be found in the most current GC/MS method detection limit book. The detection limits for a specific sample may differ from those listed due to the nature of interferences in a particular sample matrix.
- 1.2 Method 8260B is used to determine volatile organic compounds in a variety of solid waste matrices. This method is applicable to nearly all types of samples, regardless of water content, including ground water, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils and sediments. The following compounds can be determined by this method:

Analyte	CAS No. ^b	Appropriate Technique
		Purge-and-Trap
Acetone	67-64-1	pp
Acetonitrile	75-05-8	pp
Acrolein (Propenal)	107-02-8	pp
Acrylonitrile	107-13-1	pp
Allyl alcohol	107-18-6	ht
Allyl chloride	107-05-1	a
Benzene	71-43-2	a
Benzyl chloride	100-44-7	a
Bromacetone	598-31-2	pp
Bromochloromethane	74-97-5	a
Bromodichloromethane	75-27-4	a
4-Bromofluorobenzene	460-00-4	a
Bromoform	75-25-2	a
Bromomethane	74-83-9	a
n-Butanol	71-36-3	ht
2-Butanone (MEK)	78-93-3	pp
Carbon disulfide	75-15-0	pp
Carbon tetrachloride	56-23-5	a
Chloral hydrate	302-17-0	pp
Chlorobenzene	108-90-7	a
Chlorobenzene	126-99-8	a
Chlorodibromomethane	124-48-1	a
Chloroethane	75-00-3	a
2-Chloroethanol	107-07-3	pp
bis-(2-Chloroethyl) sulfide	505-60-2	pp
2-Chloroethyl vinyl ether	110-75-8	a
Chloroform	67-66-3	a
Chloromethane	74-87-3	a
Chloroprene	126-99-8	a
3-Chloropropene	107-05-1	a
3-Chloropropionitrile	542-76-7	I
1,2-Dibromo-3-chloropropane	96-12-8	pp
1,2-Dibromoethane	106-93-4	a
Dibromomethane	74-95-3	a
1,2-Dichlorobenzene	95-50-1	a
1,3-Dichlorobenzene	541-73-1	a
1,4-Dichlorobenzene	106-46-7	a
cis-1,4-Dichloro-2-butene	1476-11-5	a
trans-1,4-Dichloro-2-butene	110-57-6	pp
Dichlorodifluoromethane	75-71-8	a
1,1-Dichloroethane	75-34-3	a
1,2-Dichloroethane	107-06-2	a
1,1-Dichloroethene	75-35-4	a
trans-1,2-Dichloroethene	156-60-5	a
1,2-Dichloropropane	78-87-5	a
1,3-Dichloro-2-propanol	96-23-1	pp
cis-1,3-Dichloropropene	10061-01-5	a
trans-1,3-Dichloropropene	10061-02-6	a
1,2,3,4-Diepoxybutane	1464-53-5	a
Diethyl ether	60-29-7	a
1,4-Difluorobenzene	540-36-3	a

UNCONTROLLED DOCUMENT: DO NOT Transfer or Print

Method: 02-8260B

Revision: 9

Date: August 17, 2006

Page: 5 of 52

1,4-Dioxane	123-91-1	pp
Epichlorohydrin	106-89-8	i
Ethanol	64-17-5	i
Ethyl acetate	141-78-6	i
Ethylbenzene	100-41-4	a
Ethylene oxide	75-21-8	pp
Ethyl methacrylate	97-63-2	a
Hexachlorobutadiene	87-68-3	a
Hexachloroethane	67-72-1	i
2-Hexanone	591-78-6	pp
2-Hydroxypropionitrile	78-97-7	i
Iodomethane	74-88-4	a
Isobutyl alcohol	78-83-1	pp
Isopropylbenzene	98-82-8	a
Malononitrile	109-77-3	pp
Methacrylonitrile	126-98-7	pp
Methylene chloride (DCM)	75-09-2	a
Methyl methacrylate	80-62-6	a
4-Methyl-2-pentanone (MIBK)	108-10-1	pp
Naphthalene	91-20-3	a
Nitrobenzene	98-95-3	a
2-Nitropropane	79-46-9	a
Pentachloroethane	76-01-7	i
2-Picoline	109-06-8	pp
Propargyl alcohol	107-19-7	pp
β -Propiolactone	57-57-8	pp
Propionitrile (ethyl cyanide)	107-12-0	ht
Propylamine	107-10-8	a
Pyridine	110-96-1	i
Styrene	100-42-5	a
1,1,1,2-Tetrachloroethane	630-20-6	a
1,1,2,2-Tetrachloroethane	79-34-5	a
Tetrachloroethene	127-18-4	a
Toluene	108-88-3	a
1,2,4-Trichlorobenzene	120-82-1	a
1,1,1-Trichloroethane	71-55-6	a
1,1,2-Trichloroethane	79-00-5	a
Trichloroethene	79-01-6	a
Trichlorofluoromethane	75-69-4	a
1,2,3-Trichloropropane	96-18-4	a
Vinyl acetate	108-05-4	a
Vinyl chloride	75-01-4	a
o-Xylene	95-47-6	a
m-Xylene	108-38-3	a
p-Xylene	106-42-3	a

a	Adequate response by this technique.
b	Chemical Abstract Services Registry Number.
ht	Method analyte only when purged at 80°C.
i	Inappropriate technique for this analyte.
pc	Poor chromatographic behavior
pp	Poor purging efficiency resulting in high EQLs.

NOTE: For the preparation of soils and solids, see the 19-5035 SOP.

1.3 Method 8260B can be used to quantitate most volatile organic compounds that have boiling points below 200°C and that are insoluble or slightly soluble in water. Volatile water-soluble compounds can be included in this analytical technique. However, for the more soluble compounds, quantitation limits are approximately ten times higher because of poor purging efficiency. Such compounds include low-molecular-weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers and sulfides. The following analytes are also amenable to analysis by Method 8260B:

Bromobenzene	1-Chlorohexane
n-Butylbenzene	2-Chlorotoluene
sec-Butylbenzene	4-Chlorotoluene
tert-Butylbenzene	Crotonaldehyde
Chloroacetonitrile	Dibromofluoromethane
1-Chlorobutane	cis-1,2-Dichloroethene
1,3-Dichloropropane	Methyl-t-butyl ether
2,2-Dichloropropane	Pentafluorobenzene
1,1-Dichloropropene	n-Propylbenzene
Fluorobenzene	1,2,3-Trichlorobenzene
p-Isopropyltoluene	1,2,4-Trimethylbenzene
Methyl acrylate	1,3,5-Trimethylbenzene

Method: 02-8260B

Revision: 9

Date: August 17, 2006

Page: 6 of 52

- 1.4 The estimated quantitation limit (EQL) of Method 8260B for an individual compound is somewhat instrument dependent. Using standard quadruple instrumentation, limits shall be approximately 5 µg/kg (wet weight) for soil/sediment samples, 0.5 mg/kg (wet weight) for wastes, and 5 µg/L for ground water. EQLs will be proportionately higher for sample extracts and samples that require dilution or reduced sample size to avoid saturation of the detector.
- 1.5 Method 8260B is based upon a purge-and-trap, gas chromatographic/mass spectrometric (GC/MS) procedure. This method is restricted to use by, or under the supervision of, analysts experienced in the use of purge-and-trap systems and gas chromatograph/mass spectrometers, and skilled in the interpretation of mass spectra and their use as a quantitative tool.
- 1.6 An additional method for sample introduction is direct injection. This technique has been tested (by agencies other than ALSI) for the analysis of waste oil diluted with hexadecane 1:1 (vol/vol) and may have application for the analysis of some alcohols and aldehydes in aqueous samples. ALSI does not use the direct injection technique and the technique will not be covered by this standard operating procedure.
- 1.7 This standard operating procedure also describes the preparation of water-miscible liquids, non-water-miscible liquids, solids, wastes and soils/sediments for analysis by the purge-and-trap procedure.
- 1.8 This document states the laboratory's policies and procedures established in order to meet requirements of all certifications/accreditations currently held by the laboratory, including the most current NELAC standards.
- 1.9 The Method Detection Limits can be found in the current GC/MS Volatiles method detection limit book. The detection limits for a specific sample may differ from those listed due to the nature of interferences in a particular sample matrix.
- 1.10 Individual project requirements may override criteria listed in this SOP.

2 Summary of Method

- 2.1 An inert gas is bubbled through a 5-mL water sample contained in a purging chamber at ambient temperature. The purgeables are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent trap where the purgeables are trapped. After purging is completed, the trap is heated and backflushed with helium to desorb the purgeables onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the purgeables, which are then detected with a mass spectrometer.

Method: 02-8260B

Revision: 9

Date: August 17, 2006

Page: 7 of 52

3 Interferences

- 3.1 Impurities in the purge gas, organic compounds outgassing from the plumbing ahead of the trap and solvent vapors in the laboratory, account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.4. The use of non-Teflon plastic tubing, non-Teflon thread sealants, or flow controllers with rubber components in the purge and trap system shall be avoided.
- 3.2 Samples may be contaminated by diffusion of volatile organics (particularly fluorocarbons and methylene chloride) through the septum seal into the sample during shipment and storage. A field reagent blank prepared from organic-free reagent water and carried through the sampling and handling protocol can serve as a check on contamination.
- 3.3 Contamination by carry-over can occur whenever a low level sample is analyzed immediately after a high level sample. To reduce carry-over, the purging device and sample syringe must be rinsed with reagent water between sample analyses. Whenever an unusually concentrated sample is encountered, one or more cleaning blanks shall be analyzed to check for cross contamination. For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds or high purgeable levels, it may be necessary to wash the purging device with a soap solution, rinse with organic-free reagent water, and then dry in an oven at 105°C. The trap and other parts of the system are also subject to contamination; therefore, frequent baking and purging of the entire system may be needed. In extreme situations, the whole purge and trap device may require dismantling and cleaning.
- 3.4 Special precautions must be taken to analyze for methylene chloride. The analytical and sample storage area shall be isolated from all atmospheric sources of methylene chloride. Otherwise random background levels will result. Laboratory clothing worn by analysts shall be clean since exposure to methylene chloride fumes during extraction procedures can contribute to sample contamination.

4 Safety

- 4.1 The toxicity or carcinogenicity of each reagent in this method has not been precisely defined; however, each chemical compound shall be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available.
- 4.2 Analysts shall consult the material safety data sheets (MSDS) for each chemical used in the analysis. ALSI maintains MSDSs on all chemicals used in this procedure.

Method: 02-8260B

Revision: 9

Date: August 17, 2006

Page: 8 of 52

MSDSs are available to all staff and are located in the QA office.

- 4.3 The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: benzene, carbon tetrachloride, chloroform, 1,4-dichlorobenzene and vinyl chloride. Primary standards of these toxic compounds shall be prepared in a hood.
- 4.4 Since the chemical makeup of the samples is not known, analysts shall treat the samples with extreme caution. Precautionary steps would include using Latex gloves, wearing a fully-buttoned lab coat, and safety glasses.

5 Apparatus and Materials

- 5.1 Purge-and-trap device (Example system provided below)
- 5.1.1 Purge and Trap concentrator: Tekmar 3000, Model #14-30000-000, Serial #93133003.
- 5.1.2 Autosampler: Archon-EST, Model #D4-505220-16, Serial #12543.
- 5.1.3 Trap: VocarbJ 3000, Purge Trap K, purchased from Supelco, catalog #2-4920, or equivalent.
- 5.2 Gas chromatography/mass spectrometer/data system. (Example system provided below.)
- 5.2.1 Gas chromatograph: Hewlett Packard 5890 Series II, Serial # 3336A50415.
- 5.2.2 Gas chromatographic column: 75 m x 0.53 mm ID megabore capillary column coated with DB624 (J & W Scientific), 3 um film thickness, or equivalent.
- 5.2.3 Mass spectrometer: Hewlett Packard 5970 Series Mass Selective Detector, Model #5970B, Serial #3004A12574.
- 5.2.4 Electron Multiplier: K and M Model #7596M, purchased from CPI, part #4200-01.
- 5.2.5 GC/MS Interface
- 5.2.5.1 Jet separator if necessary: purchased from SIS, part #13505
- 5.2.5.2 Transfer line: 0.53 ID fused silica guard column, phenyl methyl deactivated, purchased from Restek, catalog #0045. Not needed with

Method: 02-8260B

Revision: 9

Date: August 17, 2006

Page: 9 of 52

split injector and EPC (electronic pressure control).

Note: Currently, none of the GCMS instruments have need of a jet separator but may require one in the future.

- 5.2.6 GC Inlet: O-I Low-Dead-Volume Injector Port kit, O-I-Analytical, Part #176926.
- 5.2.7 Data System
 - 5.2.7.1 Hewlett Packard MS DOS Chemstation, used for instrument tuning and data collection.
 - 5.2.7.2 Hewlett Packard 4920 ChemServer with Envision and Target 4.13 software.
- 5.2.8 See the instrument maintenance logbooks, located in the data review area of the GC/MS laboratory, for serial number and all pertinent information relating to all other GC/MS instruments used for the analysis of 8260B volatile samples.
- 5.3 Microsyringes: Hamilton gastight, various volumes between 10 μ L and 100 μ L: VWR #60376-220,230,241,252,263,274, or equivalent.
- 5.4 Syringe valve: two-way, with Luer ends.
- 5.5 Syringes: 5 mL Hamilton Gastight: VWR # 60376-321, or equivalent.
- 5.6 Balance – ACCULAB VI-200; 200 gm capacity; 0.01 gm resolution or equivalent.
- 5.7 Micro Reaction Vessels: 1.0 mL, Supelco #3-3293 or equivalent.
- 5.8 Mininert Valves: 15 mm, Supelco #614160 or equivalent.
- 5.9 Vials: 40 mL I-CHEM: VWR # IRS136-0040, or equivalent.
- 5.10 Vials: 20 mL I-CHEM: VWR # IRS126-0020, or equivalent.
- 5.11 Teflon faced liners: VWR # 66001-236, or equivalent.
- 5.12 Disposable pipettes: Pasteur, VWR 5 3/4" # 14672-200, or equivalent.
- 5.13 Volumetric flasks: Class A, various volumes, with ground-glass stoppers.

Method: 02-8260B

Revision: 9

Date: August 17, 2006

Page: 10 of 52

- 5.14 Spatula: stainless steel VWR # 57952-107, or equivalent
- 5.15 NOTE: In the above listings, the item noted may be replaced by a similar item of equivalent quality and function by another supplier if the need arises.

6 Reagents

- 6.1 Reagent grade inorganic chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all inorganic reagents shall conform to the specifications of The Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.
- 6.2 Reagent water: Reagent water is water in which an interference is not observed at the analyte of interest. For this purpose, tap water is used. De-ionized water shall not be used for this method as it has been shown to contain interferences due to cartridge bleed. NOTE: Once resolved, DI water may be used.
- 6.3 Compressed Helium gas: ultrahigh purity grade.
- 6.4 Methanol: EM Science Purge & Trap grade, # MX0482-6 or equivalent. This methanol is stored in the solvent storage cabinet in the GC department.
- 6.5 Primary Stock Solutions: Primary stock solutions may be prepared from pure standard materials or purchased as certified solutions. Standards for all 8260B compounds are purchased as certified solutions. These certified solutions are stored in flame-sealed ampules in the small freezer marked "Volatile Standards" located in the GCMS volatile area of the laboratory at -10°C to -20°C. Each certified solution has an expiration date and needs to be properly discarded if that date is exceeded. For all secondary working solutions, vials are labeled with the name of the standard, the date prepared, the expiration date, the preparer's initials and the reference to the volume and page number in the GCMS VOC Standards Logbook. All standard preparations must be documented in the GCMS VOC Standards Logbook. Storage location is the volatile standards freezer.
 - 6.5.1 If the primary stock solutions are to be prepared from pure standard materials, follow the instructions in Section 5.7 of Method 8260B.
 - 6.5.2 As an alternative to Section 5.7 of Method 8260B, the following procedure may be used to prepare standards from pure standard materials that are a liquid at room temperature.

Method: 02-8260B

Revision: 9

Date: August 17, 2006

Page: 11 of 52

6.5.2.1 Determine the desired concentration, **C**, in $\mu\text{g/mL}$ of the stock solution.

6.5.2.2 Determine the desired volume, **V**, in mL of the stock solution.

6.5.2.3 Lookup the density, **D**, of the liquid analyte.

6.5.2.4 Find the purity, **P**, in percent of the chemical. If it is 96% or greater, assume the purity is 100%.

6.5.2.5 Determine the volume, **VA**, in μL of chemical necessary to prepare the standard using the following equation.

$$\mathbf{VA} = (\mathbf{C*V})/(\mathbf{10*P*D})$$

6.5.2.6 Partially fill a **V** mL volumetric flask with purge and trap methanol.

6.5.2.7 Add **VA** μL of the chemical. Note more than one chemical may be added to a solution using this procedure.

6.5.2.8 Dilute to volume and invert three times to mix.

6.6 Tuning Solution: The tuning solution is prepared containing 50 $\mu\text{g/mL}$ of 4-Bromofluorobenzene in P+T (purge and trap) Methanol.

6.6.1 Place about 48 mL of P+T methanol in a 50 mL Class A volumetric flask.

6.6.2 Add 1.25 mL of Restek's 2000 $\mu\text{g/mL}$ 4-Bromofluorobenzene mix, Cat. #30026, to the methanol, dilute mixture to volume, and invert 3 times to mix.

6.6.3 Note: Preparations of varied amounts are acceptable for this standard as long as the ratio of BFB to methanol is 1:40 (50 $\mu\text{g/mL}$).

6.6.4 This solution has a six-month expiration date from the time prepared and is stored in the volatile standards freezer.

6.7 Internal Standard (IS) solution: The internal standard solution is prepared containing 150 $\mu\text{g/mL}$ of the 8260A internal standards. This standard is abbreviated as 826IS. Note, since the auto sampler adds the spiking solution, the sample loop must be calibrated according to manufacturer's instructions and the IS solution may need to be prepared at a slightly different concentration to spike the standards at 30 ppb. Record the preparation of the alternate solution in the standard logbook with the identification of the system it will be used with.

Method: 02-8260B

Revision: 9

Date: August 17, 2006

Page: 12 of 52

- 6.7.1 The IS solution contains the following compounds at 150 µg/mL: chlorobenzene-d5, fluorobenzene, and 1,4-dichlorobenzene-d4.
- 6.7.2 IS solution preparation. (Note: The ratio of standard to methanol will remain the same if different volumes are prepared.)
- 6.7.2.1 Place about 23 mL of P+T methanol in a 25 mL Class A volumetric flask.
- 6.7.2.2 Add 1.5 mL of Restek's 2500 µg/mL 8260A Internal Standard Mix, cat. #30241, dilute mixture to volume, and invert 3 times to mix.
- 6.7.2.3 This solution has a six-month expiration from the date prepared and is stored in the volatile standards freezer.
- 6.8 8260 Surrogate Solution. The 8260 surrogate solution is prepared containing 150 µg/mL of the 8260A surrogate compounds. This solution is used to prepare samples. This standard is abbreviated as **826SS**. Note: since the auto sampler adds the spiking solution, the sample loop must be calibrated according to manufacturer's instructions and the SS solution may need to be prepared at a slightly different concentration to spike the standards at 30 ppb. Record the preparation of the alternate solution in the standard logbook with the identification of the system it will be used with.
- 6.8.1 The 8260 surrogate solution contains the following compounds at 150 µg/mL: 4-bromofluorobenzene, 1,2-dichloroethane-d4, dibromofluoromethane, and toluene-d8.
- 6.8.2 8260 surrogate solution preparation. Note: Any volume can be prepared as long as the ratio of surrogate mix to methanol is 1:50.
- 6.8.3 Place about 48 mL of P+T methanol in a 50 mL Class A volumetric flask.
- 6.8.4 Add 1.0 mL of Restek's 2500 µg/mL 8260A Surrogate Standard Mix, cat. #30240, dilute mixture to volume, and invert 3 times to mix.
- 6.8.5 This solution has a six-month expiration date from the time prepared and is stored in the volatile standards freezer.
- 6.9 High 8260 Surrogate Solution. The 8260 surrogate solution is prepared containing 250 µg/mL of the 8260A surrogate compounds. This solution is used to prepare initial calibrations. This standard is abbreviated as **H826SS**.

Method: 02-8260B
Revision: 9
Date: August 17, 2006
Page: 13 of 52

6.9.1 The H8260 surrogate solution contains the following compounds at 250 µg/mL: 4-bromofluorobenzene, 1,2-dichloroethane-d4, dibromofluoromethane, and toluene-d8.

6.9.2 H8260 surrogate solution preparation. Note: Any volume can be prepared as long as the ratio of surrogate mix to methanol is 1:10.

6.9.2.1 Place slightly less than 9 mL of P+T methanol in a 10 mL volumetric flask.

6.9.2.2 Add 1.0 mL of Restek's 2500 µg/mL 8260A surrogates mix, Cat. #30240, to the methanol.

6.9.2.3 Dilute mixture to volume.

6.9.2.4 Invert the flask 3 times to mix and place the solution into labeled sub-vials.

6.9.2.5 This solution has a six-month expiration date from the time prepared and is stored in the volatile standards freezer.

6.10 Calibration Solutions.

6.10.1 The following stock solutions are purchased to prepare the calibration and calibration checkstandards:

<i>Name</i>	<i>Catalog #</i>	<i>Abbreviation</i>
502.2 CAL2000 MEGA MIX	30431	VCSMEGA
Custom V standard Acrolein	54588	VCS Acrolein
8260B Acetates Mix	30489	VCS Acetates
Custom Ketones Mix (10,000 µg/mL)	559848	VCS Ketones
2-Chloroethylvinylether Standard	30265	VCS 2 CEVE
502.2 Calibration mix 1A	30439	V Gas
Custom VOA Additions Mix (2000-50,000 µg/mL)	559847	VCS Additions

6.10.2 The concentrations of the stock solutions and their concentration in the calibration solutions are recorded in appendix A.

6.10.3 Preparation of VOA NEW, a secondary stock standard.

6.10.3.1 Add approximately 3.0 mL (but no more than 4.0 mL) of P+T methanol to a 10 mL volumetric flask.

Method: 02-8260B
Revision: 9
Date: August 17, 2006
Page: 14 of 52

6.10.3.2 Add 1.0 mL of the following stock standards: VCSMEGA, VCS Acetates, VCS Ketones, VCS Additions, VCS Acrolein, and VCS 2 CEVE.

6.10.3.3 Dilute mixture to volume.

6.10.3.4 Invert the flask 3 times to mix and place the solution into labeled sub vials.

6.10.3.5 This solution has a two-month expiration date from the time prepared and is stored in the volatile standards freezer.

6.11 Quality control sample solutions.

6.11.1 The following stock solutions are purchased to prepare the matrix spikes and blank spikes:

<i>Name</i>	<i>Catalog #</i>	<i>Abbreviation</i>
502.2 CAL200 MEGA Mix	30432	QCS MEGA
Custom Acetates Mix	560215	QCS Acetates
Custom Q Acrolein	54589	QCS Acrolein
Custom 2-Chloroethylvinylether Std	560216	QCS2CEVE
Custom Q Gases	52911	QGAS
Custom Ketones Mix (1000µg/mL)	560214	QCS Ketones
Custom VOA Additions Mix (200-5000 µg/mL)	560213	QCS Additions

6.11.2 The concentrations of the stock solutions and their concentration in the calibration verification solution are recorded in Appendix B.

6.11.3 Preparation of QVOALCS, a secondary stock standard.

6.11.3.1 Add approximately 6.0 mL (but no more than 7.0 mL) of P+T methanol to a 10 mL volumetric flask.

6.11.3.2 Add 0.5 mL of the following stock standards: QCSMEGA, QCS Acetates, QCS Ketones, QCS Additions, QCS Acrolein, and QCS2CEVE.

6.11.3.3 Dilute mixture to volume.

6.11.3.4 Invert flask 3 times and place into labeled sub-vials.

Method: 02-8260B

Revision: 9

Date: August 17, 2006

Page: 15 of 52

6.11.3.5 This solution has a two-month expiration from the date prepared and is stored in the volatile standards freezer.

6.12 Antifoam Agent (Sigma – SE-15, product #A8582 or equivalent).

6.12.1 10 μ L of antifoam is added to 5 mL of sample. An antifoam blank shall precede any samples run with antifoam to prove that there are no target analytes present in the antifoam solution. If using an Archon sampler, add 100 μ L to the 40mL vial.

6.13 DPD Free Chlorine Reagent, HACH # 21055-60, or equivalent.

7 Instrument Calibration

7.1 The specific configuration of each volatile instrument (ms01, ms03, ms05, and ms07) is recorded in the instrument's maintenance logbook.

7.2 The purge and trap program of each volatiles instrument is recorded in the instrument maintenance logbook.

7.3 The GC/MS methods are printed out with each analytical batch. This includes the tune reports and the methods. NOTE: If no changes have been made to the methods, photocopies of the latest updated method can be produced from the originals. These copies are archived with the associated raw data.

7.4 Tuning Requirements. Before the beginning of the analysis of samples, blanks, MS/MSDs, duplicates, or standards; the instrument must be hardware tuned to meet the requirements stated below.

7.4.1 Inject or purge 1.0 μ L (50 ng of BFB) of the tuning solution into the instrument.

7.4.2 When the run is complete, process the data on the Target system using the BFB method.

7.4.3 BFB performance may be evaluated using the following scans: apex, left of the apex, right of the apex, average of the apex (left, apex, and right), average of the entire BFB peak, or any of the preceding with background subtraction. Note: if background subtraction is performed, the background scan must elute within 10 scans before BFB begins to elute.

Method: 02-8260B
Revision: 9
Date: August 17, 2006
Page: 16 of 52

NOTE: The current software criteria set up in our BFB method uses the average of three scans: the apex, scan left of the apex, and scan right of the apex. These criteria satisfy DoD specifications.

7.4.4 Compare the performance of BFB to the following table:

<u>Mass (m/z)</u>	<u>Abundance Criteria</u>
50	15 to 40% of mass 95
75	30-60% of mass 95
95	Base peak, 100% Relative abundance
96	5-9% of m/z 95
173	<2% of mass 174
174	>50% of mass 95
175	5 to 9% of mass 174
176	>95% but <101% of mass 174
177	5 to 9% of mass 176

7.4.5 When the instrument meets the above requirements analysis may begin and continue for 12 hours from the injection of the tuning solution. For example, if the tuning solution is injected at 0100 on 12/1/98, the last sample may inject at 1300 on 12/1/98.

7.5 Initial calibration.

7.5.1 Each instrument in the laboratory could have a specific initial calibration curve analyzed on it but mainly use the following concentrations.

7.5.1.1 The following calibration standards are analyzed for the initial calibration, VSTD001, VSTD005, VSTD020, VSTD050, VSTD100, and VSTD200.

7.5.2 Prepare calibration solutions following the table in appendix A. For the Archon auto sampler, simply place these solutions in a 40 mL VOC vials, the auto sampler will add the IS solution automatically.

NOTE: Appendix A allows for surrogate standards to be elevated along with the same concentration of each individual calibration standard resulting in a multi-point calibration.

Method: 02-8260B

Revision: 9

Date: August 17, 2006

Page: 17 of 52

7.5.4 Calibration Criteria.

7.5.4.1 The average fit must have a %RSD of less than 15% over at least five calibration points (Six points are normally analyzed). It is acceptable to drop calibration points from either end of the calibration. Note: if the high calibration standard is dropped for a compound, the limit of quantitation must be lowered to the next calibration concentration for that compound.

7.5.4.2 The calibration curves are generated with the Target software. A primary or secondary curve fit may also be used for any compound with a %RSD greater than 15%, but shall be less than 50%.
(DoD requires less than %30)

7.5.4.2.1 Power and quadratic fits require 6 points. The R value must be 0.99 or greater. **(DoD requires ≥ 0.995)** Our Target software uses R^2 so it must be ≥ 0.98 and 0.99 for DoD.

7.5.4.2.2 The linear primary fit requires at least 5 points. The R value must be 0.99 or greater. **(DoD requires $\geq .995$)** Our Target software uses R^2 so it must be ≥ 0.98 and 0.99 for DoD.

7.5.4.2.3 A %RSD of <30% must be achieved for the Calibration Check Compounds (CCCs) before the curve passes. If it is between 15% and 30% RSD, a primary or secondary curve fit must be used. The CCCs are as follows:

1,1-Dichloroethane
Chloroform
1,2-Dichloropropane
Toluene
Ethylbenzene
Vinyl Chloride

7.5.5 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 isomers in a standard is acceptable. Acceptable resolution is achieved if the baseline to valley height between isomers is less than 25 % of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs. In other words, sum the isomers and report any results as total of the isomer X and Y. Isomers of this type need to be calibrated as a sum also.

Method: 02-8260B

Revision: 9

Date: August 17, 2006

Page: 18 of 52

7.5.5.1 After an initial calibration, a blank spike (LCS) must be analyzed before the analysis of samples can begin. The recoveries of the LCS must fall within laboratory acceptable limits for each compound of interest. If these recovery limits are met, samples may be run under the initial calibration to finish the 12-hour tune. **DoD requires** the second source verification meet 25% recovery of expected value.

7.6 GC/MS calibration verification

7.6.1 Prior to the analysis of samples, inject the BFB standard. The resultant mass spectra for the BFB must meet all of the criteria as stated in the standard operating procedure for tuning the GC/MS system. These criteria must be demonstrated each twelve hours of operation.

7.6.1.1 When the analysis of **DoD samples** is to take place, BFB must meet criteria by the average of three (3) scans. The scans include the apex, the scan left of the apex, and the scan right of the apex.

7.6.2 The initial calibration curve for each compound of interest must be checked and verified once every twelve hours during analysis with the introduction technique used for samples. Analyzing a 50 ppb calibration standard and checking the SPCCs and CCCs accomplish this.

Note: When DoD samples are to be analyzed for 15 or less analytes, all of the target analytes shall meet the same criteria as the CCCs.

7.6.2.1 When the analysis of **DoD samples** is to take place, a continuing calibration standard may need to be analyzed following the samples in a 12-hour period.

7.6.3 System Performance Check Compounds (SPCCs) - Once the continuing calibration check standard is analyzed (Section 7.6.2), the SPCCs are checked. If the SPCC criteria are met, a comparison of relative response factors is made for all compounds. This is the same check applied during the initial calibration. If the minimum relative 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.

Method: 02-8260B

Revision: 9

Date: August 17, 2006

Page: 19 of 52

7.6.3.1 The minimum relative response factor for volatile SPCCs are as follows:

Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

7.6.4 Calibration Check Compounds (CCCs) - After the system performance check is met, CCCs listed below are used to check the validity of the initial calibration. If the %RSD for each CCC is less than or equal to 20%, the initial calibration is assumed valid. If the criterion is not met ($> 20\%$ RSD), for any one CCC, corrective action must be taken. Problems similar to those listed under SPCCs could affect this criterion. If no source of the problem can be determined after corrective action has been taken, a new six-point calibration MUST be generated. This criterion MUST be met before quantitative sample analysis begins.

1,1-Dichloroethane
Chloroform
1,2-Dichloropropane
Toluene
Ethylbenzene
Vinyl Chloride

7.6.5 The internal standard responses and retention times in the check calibration standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the midpoint (50) standard of the last initial calibration check (12 hours), the chromatographic system must be inspected for malfunctions and corrections must be made, as required. If the EICP area for any of the internal standards changes by a factor of two (-50% to $+100\%$) from the midpoint (50) standard of the last initial calibration, the mass spectrometer must be inspected for malfunctions and corrections made. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is necessary. NOTE: During the course of a 12-hour tune period, all samples and blanks must also follow these criteria when referenced against the continuing calibration standard run in that tune period.

Method: 02-8260B

Revision: 9

Date: August 17, 2006

Page: 20 of 52

8 Quality Control

- 8.1 All policies and procedures in the most current revision of the ALSI QA Plan shall be followed when performing this procedure.
- 8.2 ALSI operates a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document data quality. The laboratory shall maintain records to document the quality of the data generated. Ongoing quality checks are compared to established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a quality control check standard shall be analyzed to confirm that the measurements were performed in an in-control mode of operation. (i.e.: If the MS/MSD fails, an LCS shall be analyzed.) It is the practice of the GC/MS department to analyze a laboratory control sample in every 12-hour tune period.
- 8.3 The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.9. If the analyst meets the acceptance criteria, they are now capable of running actual samples. Ongoing proficiency must be established annually as specified in the QA plan, Technical Training.
- 8.4 Each day a reagent water blank must be analyzed to demonstrate that interferences from the analytical system are under control.
- 8.5 The method blank shall be performed at a frequency of one per 12-hour tune period per matrix type or preparation method. The results of this analysis shall be one of the QC measures to be used to assess tune period acceptance. The source of method blank contamination shall be investigated, and measures taken to correct, minimize, or eliminate the problem if the concentration exceeds one-half the reporting limit. If one-half the reporting limit (RL) is exceeded, the laboratory shall evaluate whether reanalysis of the samples are necessary, based on the following criteria:
- 8.5.1 The blank contamination exceeds a concentration greater than 1/10 of the measured concentration of any sample in the associated preparatory batch, or
- 8.5.2 The blank contamination is greater than 1/10 of the project specified limit.
- 8.5.3 Any samples associated with a blank that fail these criteria shall be reanalyzed, except when the sample analysis resulted in a non-detect. If no sample volume remains for reanalysis, the results shall be reported with appropriate data qualifying codes.

Method: 02-8260B

Revision: 9

Date: August 17, 2006

Page: 21 of 52

- 8.5.4 The current laboratory practice is to comment on a sample associated with a method blank in which one or more analytes were detected at or above the reporting limit in the blank and also in the sample. (i.e. If j-values are detected, no comment is necessary)
- 8.6 The laboratory must, on an ongoing basis, analyze a spike and spike duplicate on a minimum of 5 % of all samples to monitor and evaluate laboratory data quality. It is the policy of the GC/MS department to spike one sample per every 20 samples. Samples selected for duplicate and matrix spike analysis shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a duplicate or spike may indicate a problem with the sample composition and shall be reported to the client whose sample produced the poor recovery.
- 8.6.1 Analyze one 5-mL sample aliquot to determine the background concentration (B) of each parameter.
- 8.6.2 Spike a second 40 mL sample vial with 86 μ L and 43 μ L of the QVOALCS/QGAS(see Section 6.11 and Appendix B) using an 100 μ L gastight syringe and analyze it twice, and determine the concentration after spiking (A) of each parameter. Calculate each percent recovery (P) as:
- $$P = \frac{\text{Spiked sample conc.} - \text{unspiked sample conc.}}{T} \times 100\%$$
- where T = the known true value of the spike
- 8.6.3 Compare the percent recovery (P) for each parameter with the corresponding QC acceptance criteria found in the most current listing of QC recovery limits.
- 8.6.4 If any individual P falls outside the designated range for recovery, that parameter has failed the acceptance criteria. However, since some failures may occur due to sample matrix interferences, if the LCS (Section 8.7) passes the set criteria for those failing compounds, the system performance is acceptable. In this case, a comment needs to go on the background sample stating that one or more compounds failed in the MS/MSD associated but passed in the associated LCS.
- 8.7 If any parameter fails the acceptance criteria for recovery in Section 8.6, a Laboratory Control Sample (LCS) containing each parameter that failed must be prepared and analyzed. Note: The current practice is to run an LCS at the same frequency as the method blank, which is one per 12-hour tune period. This is more than the method requires but allows for QC to be more closely associated with each sample in that 12-

Method: 02-8260B

Revision: 9

Date: August 17, 2006

Page: 22 of 52

hour tune period.

- 8.7.1 Prepare a Laboratory Control Sample (LCS) by using a 100 or 250 μL gastight microliter syringe and adding 50 μL of QGAS and 100 μL of QVOALCS listed in Section 6.11 to 50 mL of reagent water and analyze.
 - 8.7.2 Analyze the Laboratory Control Sample (LCS) to determine the concentration measured (A) of each failed parameter. Calculate each percent recovery (P_s).
 - 8.7.3 Compare the percent recovery (P_s) for each failed parameter with the corresponding LCS acceptance criteria found in the latest control charts generated for the Method 8260 LCS. **DoD requires** specific acceptance criteria found in the DoD Quality Systems Manual. If the recovery of any such parameter, which failed in the MS/MSD, falls outside the designated range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter, if detected in any samples associated with that LCS, is suspect and shall not be reported for regulatory compliance purposes. If any results are reported, a comment must accompany that result stating the failure and the possibility of a low or high bias to the data. If the LCS fails for one or more compounds that met criteria in the associated MS/MSD, the MS/MSD can prove that the instrument performance is valid as long as the acceptance criteria are as stringent as the criteria for the LCS.
 - 8.7.4 It will be the judgment of the analyst and/or supervisor to approve the data acquired using the initial calibration. If evaluation of the system in addition to the failed QCs indicates a lack of integrity of the data, it will be reanalyzed.
- 8.8 As a quality control check, the laboratory must spike all samples with the surrogate standard spiking solution and calculate the percent recovery of each surrogate compound. Recoveries must fall within the calculated limits. See Section 8.13 for the development of surrogate control limits. If the surrogate recoveries do not fall within the calculated limits, the sample shall be re-analyzed and the system shall be evaluated for malfunctions. If the surrogate recovery is acceptable in the re-analysis, report the data from the re-analysis. If data must be reported in which a surrogate(s) is out, report with a comment.
- 8.8.1 HORIZON LIMS standard verbiage comment V8L - One or more 8260 surrogates were recovered outside of the recovery limits. Then it lists the current limits.

Method: 02-8260B

Revision: 9

Date: August 17, 2006

Page: 23 of 52

8.8.2 HORIZON LIMS standard verbiage comment VSM - One or more volatile surrogate(s) was recovered outside of the recovery limits. Its recovery was confirmed by re-analysis indicating a significant matrix effect.

NOTE: A sample with a surrogate out will normally be re-analyzed followed by reporting with a comment VSM. However, if holding time is expired, comment V8L will be used, and the sample will not be re-analyzed.

8.9 To establish the ability to generate accuracy and precision, the analyst must perform the following operation as an initial demonstration of capability.

8.9.1 A quality control (QC) check sample concentrate is prepared containing each parameter of interest. The concentrate must be from an external source, different from the source used for the calibration standards.

8.9.2 Using a 100 or 250 μL gastight syringe, inject 50 μL and 100 μL of the QGAS/QVOALCS solutions into a 50 mL volumetric flask containing reagent water. This is done four (4) times. See Appendix B.

8.9.3 Calculate the average recovery (\bar{X}) in $\mu\text{g/L}$, and the standard deviation of the recovery (s) in $\mu\text{g/L}$, for each parameter of interest using the four results.

8.9.4 For each parameter, compare s and \bar{X} with the DOC forms of the corresponding acceptance criteria in the latest control charts generated for 8260B MS/MSDs, respectively. If s and \bar{X} for all parameters of interest meet the acceptance criteria, the system performance is acceptable and analysis of samples can begin. If any individual s exceeds the precision limit or any individual \bar{X} falls outside the range for accuracy, the system performance is unacceptable for that parameter.

8.10 If one or more of the parameters tested fail at least one of the acceptance criteria from Section 8.9, the analyst must proceed according to Section 8.10.1 or 8.10.2.

8.10.1 Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with Section 8.9.2.

8.10.2 Beginning with Section 8.9.2, repeat the test only for those parameters that failed. Repeated failure will confirm a general problem with the measurement system. If this occurs, locate and correct the problem and repeat the test for all compounds of interest beginning with Section 8.9.2.

8.11 The laboratory must, on an ongoing basis, demonstrate through the analysis of

UNCONTROLLED DOCUMENT: DO NOT Transfer or Print

Method: 02-8260B

Revision: 9

Date: August 17, 2006

Page: 24 of 52

Laboratory Control Samples (LCSs) that the operation of the measurement system is in control. The procedure is described in Section 8.7. The frequency of the check standard analysis is equivalent to 5 % of all samples analyzed. This 5% is equivalent to 1 in 20 samples, which is what the GCMS department defines as a batch. This may be reduced if spike recoveries from samples (see Section 8.6) meet all specific control criteria. It is the practice of the GCMS department to analyze a LCS every 12-hour tune period even though the method does not require it. A batch will typically consist of two or more 12-hour tune periods until 20 samples have been analyzed including all QC.

- 8.12 The laboratory must maintain performance records to document the quality of data generated.
- 8.13 As part of the QC program, control limits for samples must be assessed and records must be maintained. After the analysis of at least 20 spiked samples as in Section 8.6, calculate the average percent recovery (P) and the standard deviation of the percent recovery (s_p). Express the accuracy assessment as a percent recovery interval from $P - 3s_p$ to $P + 3s_p$ (i.e., If $P = 100\%$ and $s_p = 10\%$, the accuracy interval is expressed as 70 - 130%). Update the accuracy assessment for each parameter at least annually.
- 8.14 MDL Studies. Method detection limit studies are performed annually to statistically determine the concentration levels an analytical system is capable of determining. MDL studies must be performed according to SOP 99-MDL or the reference method, whichever is more frequent. Reporting limits are set approximately 3-5 times the method detection limit, but not lower than the lowest initial calibration standard. (For DoD reporting purposes, reporting limits are set at least 3 times the MDL, and not more than 10 times the MDL.)
- 8.14.1 The group leader will determine at what level (concentration) the MDL study will be performed. At least seven replicates are to be analyzed. All replicates analyzed must be included in the MDL study. Note: The current concentration used for MDL studies is 0.8 µg/L.
- 8.14.2 The QLCS and the QGAS standards can be used to prepare the MDL studies, but using the initial calibration standards, VOANEW and VGAS, is a more commonly used method.
- 8.14.3 Use the Target software to generate an MDL study report.

Method: 02-8260B

Revision: 9

Date: August 17, 2006

Page: 25 of 52

9 Sample Collection, Preservation and Handling

9.1 Sample Collection

9.1.1 Standard 40 mL glass screw-cap VOA vials with Teflon-lined silicone septa may be used for liquid samples.

9.1.2 When collecting the samples, liquids shall be introduced into the vials gently to reduce agitation, which might drive off volatile compounds. In general, liquid samples shall be poured into the vial without introducing any air bubbles within the vial as it is being filled. Should bubbling occur as a result of violent pouring, the sample must be poured out and the vial refilled. The vials shall be completely filled at the time of sampling, so that when the septum cap is fitted and sealed, and the vial inverted, no headspace is visible. The sample shall be hermetically sealed in the vial at the time of sampling, and must not be opened prior to analysis to preserve their integrity.

9.1.2.1 Due to differing solubility and diffusion properties of gases on liquid matrices at different temperatures, it is possible for the sample to generate some headspace during storage. This headspace will appear in the form of micro bubbles, and shall not invalidate a sample for volatile analysis.

9.1.2.2 The presence of a macro bubble in a sample vial generally indicates either improper sampling technique or a source of gas evolution within the sample. The latter case is usually accompanied by a buildup of pressure within the vial (e.g., carbonate-containing samples preserved with acid). Studies conducted by the USEPA (EMSL-Ci, unpublished data) indicate the Pea-sized bubbles (i.e., bubbles not exceeding 1/4 inch or 6 mm in diameter) did not adversely affect volatiles data. These bubbles were generally encountered in wastewater samples, which are more susceptible to variations in gas solubility than are groundwater samples. NOTE: **For DoD samples**, any size air bubble will be commented on the lab report. See Section 9.3.2.

9.1.3 If the aromatic compounds benzene, toluene, and ethyl benzene are to be determined; a second separate sample shall be collected as follows because refrigeration alone may not preserve these compounds for more than seven days. Collect about 500 mL of sample in a clean container. Adjust the pH to about 2 while stirring vigorously by adding 1+ 1 HCl. Check the pH with narrow range (1.4 to 2.8) pH paper. Fill the sample vial as described in Section 9.1.2.

Method: 02-8260B

Revision: 9

Date: August 17, 2006

Page: 26 of 52

9.2 Sample Preservation

- 9.2.1 Preserve aqueous samples using HCl to a pH <2. Sample preservation shall be performed immediately upon sample collection. The sample shall then be iced above the freezing point of water up to 6°C in transport.
- 9.2.2 Ascorbic acid preservative is added to the vial prior to shipping to the sample site if the sample contains residual chlorine. Immediately following collection of the sample, shake the vial vigorously for one minute. Maintain the hermetic seal until the time of sample analysis.
- 9.2.3 Once samples are received, they must be refrigerated above the freezing point of water up to 6°C until analysis.

9.3 Sample Handling

- 9.3.1 All samples must be analyzed within 14 days of collection. All samples not analyzed within this time frame must be discarded and re-sampled for analysis, unless permission is given by the client to run the sample past its hold time. If this occurs, it must be clearly noted on the laboratory report.
 - 9.3.1.1 If a dilution of the sample was analyzed after the hold time due to compounds exceeding the calibration range in the initial (and reportable) analysis, the standard verbiage comment VDL in the HORIZON LIMS may be used.
- 9.3.2 Check the run logbook to see if there was headspace in the sample. Add a comment to the report if it was present. The standard verbiage code for this in the HORIZON LIMS is HSP.
- 9.3.3 Check the run logbook to see if the pH of the sample was greater than 2. Add comment to the report if it exceeded 2. The standard verbiage code for this in the HORIZON LIMS is PH>.
- 9.3.4 Chlorine: Check the run logbook to see if there was free chlorine in the sample. Add a comment to the report if it was present.

Method: 02-8260B

Revision: 9

Date: August 17, 2006

Page: 27 of 52

10 Procedure

10.1 Daily tuning criteria and GC/MS calibration verification criteria must be met before analyzing samples.

10.2 All samples must be allowed to warm to ambient temperature before analysis.

10.3 Sample preparation and analysis.

10.3.1 Blanks must be free from target analytes above the established reporting limits.

10.3.1.1 Using the Archon autosampler, fill a 40 mL VOC vial with reagent water and cap, load on the autosampler and run. The Archon will add the IS/SS automatically.

10.3.2 Laboratory Control Samples (LCSs).

10.3.2.1 Using the Archon autosampler, place slightly less than 50 mL of reagent water in a 50 mL volumetric flask. Add 100 μ L of QVOA LCS and add 50 μ L of QGAS. Dilute to volume, invert 3 times, and place in a 40 mL VOC vial. The Archon will add the IS/SS automatically.

10.3.3 Samples:

10.3.3.1 Note in the run logbook whether or not the sample has headspace. Mark a Y in the HSP column if there is an air bubble bigger than a large pea (one quarter inch in diameter). Otherwise, mark an N in this column.

10.3.3.2 In order to prevent system overload it is a good practice to check each sample's history in the LIMS before analyzing the sample. If the sample has no history, then immediately before loading the instrument, open the vial, lift to within approximately 2-3 inches of the nose, wave a hand across the top of the sample towards the nose. Run the sample at a dilution if it has a polluted or organic chemical odor.

10.3.3.3 To composite samples, gently pour the sample containers into a clean appropriately sized beaker. Swirl the beaker gently to mix, and pour contents back into the sample vials. Mark the sample vials with a "C" to denote that they were composited. NOTE: Only at a client's request are 8260 samples ever composited.

Method: 02-8260B

Revision: 9

Date: August 17, 2006

Page: 28 of 52

10.3.3.4 Prepare sample dilutions according to the following table (Note: This table may not be inclusive of every dilution that will need to be performed. It may be necessary to perform intermediate dilutions or to use a larger volumetric flask to perform the larger dilutions): Note: **For DoD samples;** dilutions shall be limited to steps that are less than 100-fold.

<i>Dilution factor</i>	<i>Water volume</i>	<i>Sample added</i>	<i>Final Volume</i>
2	2.5mL	2.5mL	5.0mL
2	25mL	25mL	50mL
4	3.75mL	1.25mL	5.0mL
4	3.75mL	12.5mL	50mL
5	4.0mL	1.0mL	5.0mL
5	40mL	10mL	50mL
10	4.5mL	0.5mL	50mL
10	45mL	5.0mL	50mL
20	4.75mL	250µl	5.0mL
20	47.5mL	2.5mL	50mL
50	4.9mL	100µl	5.0mL
50	49mL	1.0mL	50mL
100	4.95mL	50µl	5.0mL
100	49.5mL	500µl	50mL
200	4.975mL	25µl	5.0mL
200	49.75mL	250µl	50mL
500	4.99mL	10µl	5.0mL
500	49.9mL	100µl	50mL
1000	5.0mL	5.0µl	5.0mL
1000	50mL	50µl	50mL
2000	5.0mL	2.5µl	5.0mL
2000	50mL	25µl	50mL
5000	5.0mL	1.0µl	5.0mL
5000	50mL	10µl	50mL
10000	50mL	5.0µl	50mL
20000	50mL	2.5µl	50mL
50000	50mL	1.0µl	50mL

10.3.3.5 Dilutions with a 5.0 mL final volume are prepared in the following manner.

10.3.3.5.1 Rinse a 5.0 mL syringe several times with reagent water.

10.3.3.5.2 Fill the syringe with reagent water and adjust the water volume to the appropriate mark.

10.3.3.5.3 Move the plunger back to 5 mL.

10.3.3.5.4 Quickly add the volume of sample, then prepare for the appropriate autosampler.

Method: 02-8260B

Revision: 9

Date: August 17, 2006

Page: 29 of 52

10.3.3.6 Prepare dilutions in Class A volumetric flasks in the following manner.

10.3.3.6.1 Partially fill a clean volumetric with reagent water.

10.3.3.6.2 Quickly add the appropriate volume of sample.

10.3.3.6.3 Quickly dilute to volume. Invert 3 times to mix. Prepare for the appropriate autosampler.

10.3.3.7 Using the Archon autosampler, place the sample vial (it may be necessary to remove the label) or a capped 40 mL VOC vial filled with diluted sample on the autosampler, and run. The Archon will add the IS/SS automatically.

10.3.3.8 Antifoam may be added to prevent foaming. It is added 100 μ L at a time to a 40 mL vial or 50 mL flask, or 10 μ L to a 5 mL syringe.

10.3.3.9 Check the pH of the unused sample (this includes sample spikes if they were taken from a different vial) with pH paper. Record in column of the run logbook either the pH or whether or not the pH was less than or equal to 2 denoted "<2", if greater than 2 it is denoted ">2".

10.3.3.10 Check for the presence of free chlorine with an aliquot of DPD free chlorine reagent added to 10 mL of sample. It will turn pink if chlorine is present. In the Cl column of the run logbook, record a "Y" if free chlorine was detected, record an "N" if it was not. Note: We use a dispenser purchased from HACH to determine the aliquot size.

10.3.3.11 If running low or med level soils by 8260B, see the SOP for the 5035 Method (19-5035).

10.3.4 Sample spikes (MS/MSD).

10.3.4.1 Using the Archon auto sampler, inject 86 μ L of QVOALCS and 43 μ L of QGAS through the vial's septum. Shake or roll the vial to mix contents. Place the sample vial (it may be necessary to remove the label) or a capped 40 mL VOC vial filled with diluted sample on the auto sampler and run. The Archon will add the IS/SS automatically.

Method: 02-8260B

Revision: 9

Date: August 17, 2006

Page: 30 of 52

10.3.5 Duplicates.

10.3.5.1 Prepare duplicates the same way as normal samples. Duplicates shall only be prepared on samples that have historically had target hits. Otherwise, matrix spike duplicates shall be analyzed. The RPD acceptable limit is <40 %. **DoD requires** the RPD acceptable limit is <30%.

10.4 Data analysis.

10.4.1 Tune time. All analyses must have an injection time within 12 hours of the injected 50 ng bfb tuning solution, which met method criteria. The only exception to this is ending calibration checks that are requested by some clients. They shall run within 24 hours of the bfb (immediately after the last sample in the sequence).

10.4.2 Internal standard areas and retention times.

10.4.2.1 The internal standard areas in each analysis shall be within a factor of two of their abundance in the calibration verification standard. Note, this is not only a method requirement, but also a good laboratory practice as it helps to ensure that the instrument system is functioning normally.

10.4.2.2 The retention times of internal standards in each analysis shall be within 0.5 minutes of their retention times in the calibration verification standard. Note, this is not only a method requirement, but also a good laboratory practice as it helps to ensure that the instrument system is functioning normally.

10.4.2.3 The internal standard areas may vary due to the following reasons: instrument malfunction, wrong amount of IS/SS mix added, partially open sample valve, sample overload (i.e., shall have been run very diluted), etc.

10.4.2.3.1 ALSI LIMS standard verbiage comment VIS – One or more volatile internal(s) was recovered outside of the recovery limits. Its recovery was confirmed by re-analysis indicating a significant matrix effect.

10.4.2.4 The retention times may vary due to the following reasons: sample overload, sample foaming, plug or obstruction in the instrument, instrument malfunction, unstable room temperature, etc.

Method: 02-8260B

Revision: 9

Date: August 17, 2006

Page: 31 of 52

10.4.3 Surrogates.

10.4.3.1 The surrogates shall be recovered within the recovery limits. If they are not the sample shall be re-analyzed and the system shall be evaluated for malfunctions. If the surrogate recovery is acceptable in the re-analysis, report the data from the re-analysis. If data must be reported in which a surrogate(s) is out, report with a comment.

10.4.3.1.1 HORIZON LIMS standard verbiage comment V8 - One or more 8260 surrogates recovered outside of the recovery limits. Then it lists the current limits.

10.4.3.1.2 HORIZON LIMS standard verbiage comment VSM - One or more volatile surrogate(s) was recovered outside of the recovery limits. Its recovery was confirmed by re-analysis indicating a significant matrix effect.

NOTE: A sample with a surrogate out will normally be re-analyzed followed by reporting with a comment VSM. However, if holding time is expired, comment V8 will be used, and the sample will not be re-analyzed.

10.4.4 Target hits.

10.4.4.1 Positive hits. Identified by comparing the mass spectrum of the compound with a reference mass spectrum of the compound from a standard, which was analyzed on the same instrument. Obtain EICPs (the overlays on the right of the Target review window) for the primary (quantitating) mass and at least two secondary masses for each parameter of interest. The following criteria must be met to make a qualitative identification.

10.4.4.1.1 The characteristic masses of each parameter of interest must maximize in the same scan or within one scan of each other. Beware of co eluting interferences.

10.4.4.1.2 The retention time must fall within ± 30 seconds of the retention time of the compound in the daily QC calibration verification check.

10.4.4.1.3 The relative peak heights of the characteristic masses in the EICPs must fall within ± 20 % of the relative intensities of the masses in a reference mass spectrum.

Method: 02-8260B

Revision: 9

Date: August 17, 2006

Page: 32 of 52

10.4.4.2 Negative hits. Hits which do not meet the requirements for a positive hit are marked as unknown on Target.

10.4.4.3 Over-range hits. If a requested target analyte is a positive hit (be aware that if an analyte is present at a high enough concentration, its mass spectrum may be distorted) and exceeds the instrument's initial calibration range, the sample shall be rerun at a dilution until the analyte is within the instrument's calibration range (preferably in the upper half of the calibration range). If internal and surrogate recoveries are acceptable in the original analysis, all compounds except those that exceeded the calibration range will be reported from this run. Those that did exceed the calibration range will be reported from the dilution analysis (DL).

10.4.4.4 If manual integrations are required, the procedure in SOP 99-Integration is followed.

10.4.5 Blanks: Blanks shall have no target hits present at the reporting limit. A blank's surrogates shall be within the surrogate recovery limits. If a requested analyte is present above the reporting limit in the blank and it is present above the reporting limit in a sample(s), the sample shall be rerun. If that is not possible fill out a corrective action form explaining the situation and report the data with a comment similar to the following: "This sample had a hit of 8 µg/L of TCE which was present in the associated method blank at 1 µg/L." **NOTE: For DoD samples,** blanks shall have no target hits present above 2 times the calculated MDL. Blanks must not exceed one-half the reporting limit. See Section 8.5 under quality control for additional information.

10.4.6 QC samples (MS/MSD, blank spikes, duplicates).

10.4.6.1 Generate a MS/MSD report (form 3) using Quickforms in the Target software.

10.4.6.2 Compare the percent recovery, P, of each parameter with the corresponding QC acceptance criteria. If the spike sub-list MS/MSD.spk is used, these shall be the limits present on MS/MSD report generated with Quickforms.

10.4.6.3 If any individual P falls outside the designated range for recovery in either the MS or MSD, that parameter has failed the acceptance criteria. A blank spike containing each parameter that failed shall have been analyzed.

Method: 02-8260B

Revision: 9

Date: August 17, 2006

Page: 33 of 52

10.4.6.3.1 Analyze the blank spike to determine the concentration measured (A) of each failed parameter. Compounds which did not fail in the MS/MSD are not considered. The target software will calculate the percent recoveries of the blank spike. Use the spike sub-list WATERQC. The equation to calculate percent recovery (P) in a blank spike follows. T is the known true value of the spike.

$$P = A * 100\% / T$$

10.4.6.3.2 Compare the percent recovery, P, of each parameter with the corresponding QC acceptance criteria found in Appendix C. If the spike sub-list QATERQC is used, these shall be the limits present on blank spike report generated with Quickforms.

10.4.6.3.3 If the recovery of any such parameter, P, falls outside the designated range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked sample is suspect and may not be reported for regulatory compliance purposes. If the data must be reported due to either the samples hold time or a lack of sufficient sample for re-analysis, fill out a corrective action form explaining the problem, and comment on the sample report.

10.4.6.3.4 It will be the judgment of the analyst and/or supervisor to approve the data acquired using this initial calibration. If evaluation of the system in addition to the failed QCs indicates a lack of integrity of the data, the samples will be reanalyzed.

10.4.6.4 Duplicates (including MSD) shall have a % Repeatability of 40 % or less. NOTE: **For DoD samples**, control limits calculated by using LCS data will be used to determine acceptable Relative Percent Difference.

10.4.7 Library searches.

10.4.7.1 The selection and quantitation of non-target peaks is performed automatically by the Target software. Note: The sample shall be processed for client specific compounds only. Otherwise, the Target

Method: 02-8260B

Revision: 9

Date: August 17, 2006

Page: 34 of 52

software will not pick any other target compounds as library peaks. If a sublist containing client specific analytes is not used, the analyst must add, to the library search form, any compounds that were detected in the analysis that the client does not want reported. Also, if any changes have been made to the sample such as deleting or integrating peaks, the library search must be redone by selecting "process unknowns" in Target.

- 10.4.7.2 If the software selects any internal standard, surrogate, or target compounds, delete them in Target. The air peak (the first large peak on the chromatogram usually, its primary m/e will be 44) shall be deleted also. Also, early eluters with mass 40 can be deleted.
- 10.4.7.3 The Target software will perform a library search on and estimate the concentration of the 20 largest non-target peaks.
- 10.4.7.4 Only after visual comparison of sample spectra with the nearest library searches shall a tentative identification be assigned to the peak. The analyst will need to search each non-target peak on the Target system to view the library searches. Consider the following sets of guidelines before making a tentative identification.
- 10.4.7.5 Guidelines for making tentative identification of non-target compounds. In other words, making a specific identification of non-target compounds, i.e., limonene, hexamethylbenzene. These guidelines are from a contract laboratory program statement of work.
- 10.4.7.5.1 Major ions (ions greater than 10% of the most abundant ion) in the reference spectrum shall be present in the sample spectrum.
- 10.4.7.5.2 The relative intensities of the major ions shall agree within plus or minus 20%. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding sample ion abundance must be between 30 and 70%.)
- 10.4.7.5.3 Molecular ions present in the reference spectrum shall be present in the sample spectrum.
- 10.4.7.5.4 Ions present in the reference spectrum but not in the sample spectrum shall be reviewed for possible

Method: 02-8260B

Revision: 9

Date: August 17, 2006

Page: 35 of 52

subtraction from the sample spectrum because of background contamination or co eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

10.4.7.5.5 If in the analyst's technical judgment, no valid identification is possible, the compound shall be reported as unknown. If possible give an additional classification to the compound (i.e., unknown phthalate, unknown hydrocarbon, unknown acid type, unknown chlorinated compound, etc.). If the probable molecular weights can be determined, include them.

10.4.7.6 Guidelines for making tentative ID based on match quality.

10.4.7.6.1 If a non-target compound is present in the calibration mixes it can be identified as that compound no matter how good or bad the match quality is as long as the criteria for identifying the spectra of target compounds are met for those compounds, i.e., hexane, benzyl chloride, 1,2,4-trimethylbenzene, etc.

10.4.7.6.2 If a tentatively identified compound has a match of greater than 90% and the next closest match is greater than 30 % less, tentatively identify the peak as that compound.

10.4.7.6.3 If the match quality of 2 or more isomers are very close together and greater than 70% with no other unrelated compounds within 10 %, identify that peak as _____ isomer. Be as specific as possible.

10.4.7.6.4 Identify classes of compounds if all the compounds above 50 % match belong to the same class, or if the 2 or 3 closest matches belong to one chemical class and the next matches have significantly different match quality.

10.4.7.6.5 Use the analyst's experience when possible. Also, be consistent throughout a group of samples, referring back to retention times as a guide.

Method: 02-8260B

Revision: 9

Date: August 17, 2006

Page: 36 of 52

11 Calculations

11.1 All calculations are performed by the Target software.

12 Reporting Results

12.1 Horizon LIMS results are reported to three significant figures but limited to the number of decimal places in the reporting limit for the individual compound or analyte.

12.2 When entering data into the Horizon LIMS do not round off results: Horizon will automatically round off to 3 significant figures after all internal calculations are completed.

12.3 Any sample compound with a result less than the reporting limit is reported with the actual result in the Horizon LIMS. Any sample with a result less than the reporting limit is reported as ND (non-detectable); LIMS will automatically report the appropriate detection limit. The client may request that "J values" be reported. J values are hits between the reporting limit and the method detection limit. They are reported with a "J" flag.

12.4 If the primary analysis of a sample was diluted, the reporting limits must be raised proportionate to the dilution factor. The following standard verbiage comments in the LIMS may be added to explain to the client why the reporting limits are elevated.

12.4.1 VLE - Sample was run at a dilution due to late eluting non-target compounds.

12.4.2 VNT - Sample was run at a dilution due to the level of non-target compounds.

12.4.3 VTC - Sample was run at a dilution due to the level of target compounds.

12.5 Any errors must be marked through with a single line with the analyst's initial, the date, and the correction.

12.6 All raw data used for reporting results must be dated and initialed by the qualified laboratory personnel performing first and second review.

13 Waste Disposal

13.1 Refer to ALSI SOP 19-Waste Disposal.

Method: 02-8260B

Revision: 9

Date: August 17, 2006

Page: 37 of 52

14 Pollution Prevention

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. Management shall consider pollution prevention a high priority. Extended storage of unused chemicals increases the risk of accidents. The laboratory shall consider smaller quantity purchases which will result in fewer unused chemicals being stored and reduce the potential for exposure by employees. ALSI tracks chemicals when received by recording their receipt in a traceable logbook. Each chemical is then labeled according to required procedures and stored in assigned locations for proper laboratory use.

15 Definitions

15.1 Refer to ALSI QA Plan under Laboratory Quality Control Checks for general definitions.

16 Troubleshooting

16.1 Refer to maintenance logs and instrument manuals for guidance in troubleshooting specific problems related to the instrumentation used in this method.

Method: 02-8260B

Revision: 9

Date: August 17, 2006

Page: 38 of 52

**TABLE 1
BFB KEY M/Z ABUNDANCE CRITERIA**

MASS	M/Z ABUNDANCE CRITERIA
50	15 TO 40% OF MASS 95
75	30 TO 60% OF MASS 95
95	BASE PEAK, 100% RELATIVE ABUNDANCE
96	5 TO 9% OF MASS 95
173	<2% OF MASS 174
174	>50% OF MASS 95
175	5 TO 9% OF MASS 174
176	>95% BUT <101% OF MASS 174
177	5 TO 9% OF MASS 176

Method: 02-8260B

Revision: 9

Date: August 17, 2006

Page: 39 of 52

APPENDIX A**Theoretical Standard Concentrations**Initial Calibration
SW846 Method 8260B

VOANEW = 1.0 mL of VCSMEGA, VCS Acetates, VCS Ketones, VCS Additions, VCS Acrolein, and VCS2CEVE to a final volume of 10 mL in P & T methanol.

Prepare the 6 initial calibration standards from the following table:

Standard ID	Volume Added			Flask Volume (mL)
	VOANEW	VGas	H826SS	
VSTD200	50 µL	50 µL	40 µL	50
VSTD100	25 µL	25 µL	20 µL	50
VSTD050	25 µL	25 µL	20 µL	100
VSTD020	20 µL	20 µL	16 µL	200
VSTD005	12.5 µL	12.5 µL	10 µL	500
VSTD001	2.5 µL	2.5 µL	2 µL	500

Compound Name	Standard Mix	Stock (ppm)	VSTD200	VSTD100	VSTD050	VSTD020	VSTD005	VSTD001
Benzene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Bromobenzene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Bromochloromethane	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Bromodichloromethane	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Bromoform	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
n-Butylbenzene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Sec-Butylbenzene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Tert-Butylbenzene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Carbon tetrachloride	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Chlorobenzene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Chloroform	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
2-Chlorotoluene (o)	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
4-Chlorotoluene (p)	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Dibromochloromethane	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
1,2-Dibromo-3-chloropropane	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
1,2-Dibromoethane (EDB)	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Dibromomethane	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
1,2-Dichlorobenzene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
1,3-Dichlorobenzene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
1,4-Dichlorobenzene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
1,1-Dichloroethene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Cis-1,2-Dichloroethene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Trans-1,2-Dichloroethene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb

UNCONTROLLED DOCUMENT: DO NOT Transfer or Print

Method: 02-8260B

Revision: 9

Date: August 17, 2006

Page: 40 of 52

Compound Name	Standard Mix	Stock (ppm)	VSTD200	VSTD100	VSTD050	VSTD020	VSTD005	VSTD001
1,2-Dichloropropane	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
1,3-Dichloropropane	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
2,2-Dichloropropane	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
1,1-Dichloropropene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Cis-1,3-Dichloropropene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Trans-1,3-Dichloropropene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Ethylbenzene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Hexachlorobutadiene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Isopropylbenzene (Cumene)	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
p-Isopropyltoluene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Methylene Chloride	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Naphthalene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
n-Propylbenzene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Styrene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
1,1,1,2-Tetrachloroethane	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
1,1,2,2-Tetrachloroethane	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Tetrachloroethene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Toluene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
1,2,3-Trichlorobenzene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
1,2,4-Trimethylbenzene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
1,3,5-Trimethylbenzene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
m-xylene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
p-xylene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
o-xylene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
1,1-Dichloroethane	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
1,1,1-Trichloroethane	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
1,2-Dichloroethane	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
1,2,3-Trichloropropane	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
1,2,4-Trichlorobenzene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
1,1,2-Trichloroethane	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Trichloroethene	VCSMEGA	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb

Vinyl Acetate	VCS Acetates	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Methyl acetate	VCS Acetates	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Ethyl acetate	VCS Acetates	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb

Acetone	VCS Ketones	10000	1000 ppb	500 ppb	250 ppb	100 ppb	25 ppb	5 ppb
2-Butanone	VCS Ketones	10000	1000 ppb	500 ppb	250 ppb	100 ppb	25 ppb	5 ppb
4-Methyl-2-pentanone	VCS Ketones	10000	1000 ppb	500 ppb	250 ppb	100 ppb	25 ppb	5 ppb
2-Hexanone	VCS Ketones	10000	1000 ppb	500 ppb	250 ppb	100 ppb	25 ppb	5 ppb
1,1-Dichloro-2-propanone	VCS Ketones	10000	1000 ppb	500 ppb	250 ppb	100 ppb	25 ppb	5 ppb

Pentane	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
3-Chloroprene (allyl chloride)	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Di-isobutylene	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
1-Chlorohexane	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Methyl-tert-butyl ether	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb

UNCONTROLLED DOCUMENT: DO NOT Transfer or Print

Method: 02-8260B

Revision: 9

Date: August 17, 2006

Page: 41 of 52

Compound Name	Standard Mix	Stock (ppm)	VSTD200	VSTD100	VSTD050	VSTD020	VSTD005	VSTD001
Ethyl ether	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Freon 113 (1,1,2-TCTFE)	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Hexane	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Heptane	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Cyclohexane	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Benzyl chloride	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Iodomethane	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Carbon Disulfide	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Chloroprene	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Octane	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Acrylonitrile	VCS Additions	10000	1000 ppb	500 ppb	250 ppb	100 ppb	25 ppb	5 ppb
2-Nitropropane	VCS Additions	10000	1000 ppb	500 ppb	250 ppb	100 ppb	25 ppb	5 ppb
Tetrahydrofuran	VCS Additions	10000	1000 ppb	500 ppb	250 ppb	100 ppb	25 ppb	5 ppb
Tert-Butyl alcohol	VCS Additions	10000	1000 ppb	500 ppb	250 ppb	100 ppb	25 ppb	5 ppb
Trans-1,4-Dichloro-2-butene	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Methyl methacrylate	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Isobutyl alcohol	VCS Additions	20000	2000 ppb	1000 ppb	500 ppb	200 ppb	50 ppb	10 ppb
Hexachloroethane	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Ethyl methacrylate	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
2-Propanol	VCS Additions	10000	1000 ppb	500 ppb	250 ppb	100 ppb	25 ppb	5 ppb
1-Propanol	VCS Additions	20000	2000 ppb	1000 ppb	500 ppb	200 ppb	50 ppb	10 ppb
Propionitrile	VCS Additions	10000	1000 ppb	500 ppb	250 ppb	100 ppb	25 ppb	5 ppb
Methacrylonitrile	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
1,4-Dioxane	VCS Additions	50000	5000 ppb	2500 ppb	1250 ppb	500 ppb	125 ppb	25 ppb
Pentachloroethane	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Nitrobenzene	VCS Additions	20000	2000 ppb	1000 ppb	500 ppb	200 ppb	50 ppb	10 ppb
Methyl acrylate	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Chloroacetonitrile	VCS Additions	10000	1000 ppb	500 ppb	250 ppb	100 ppb	25 ppb	5 ppb
1-Chlorobutane	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Dichlorodifluoromethane	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
tert-amyl methyl ether	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Ethyl tert-butyl ether	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Di-isopropyl ether	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Methyl cyclohexane	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Acetonitrile	VCS Additions	10000	1000 ppb	500 ppb	250 ppb	100 ppb	25 ppb	5 ppb
1,2,3-Trimethylbenzene	VCS Additions	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb

Acrolein	V Acrolein	50000	5000 ppb	2500 ppb	1250 ppb	500 ppb	125 ppb	25 ppb
----------	------------	-------	----------	----------	----------	---------	---------	--------

2-Chloroethyl vinyl ether	VCS2CEVE	2000	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
---------------------------	----------	------	---------	---------	--------	--------	-------	-------

Bromomethane	V Gases	200	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Chloroethane	V Gases	200	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Chloromethane	V Gases	200	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Dichlorodifluoromethane	V Gases	200	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Trichlorofluoromethane	V Gases	200	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb
Vinyl Chloride	V Gases	200	200 ppb	100 ppb	50 ppb	20 ppb	5 ppb	1 ppb

Method: 02-8260B

Revision: 9

Date: August 17, 2006

Page: 42 of 52

APPENDIX BTheoretical Standard Concentrations
Quality Control Standard / Spike
EPA Method 524.2

QVOALCS = 0.5 mL of QCS MEGA, QCS Acetates, QCS Ketones, QCS Additions, QCS Acrolein, and QCS 2CEVE to a final volume of 10.0 mL in P & T methanol.

Stock mix ID	Volume Added		
	5 mL Final Volume	50 mL Final Volume	100 mL Final Volume
QVOALCS	10 µL	100 µL	200 µL
QGASES	5 µL	50 µL	100 µL

Compound Name	Standard Mix	Stock (ppm)	Working Conc.
Benzene	QCSMEGA	200	20 ppb
Bromobenzene	QCSMEGA	200	20 ppb
Bromodichloromethane	QCSMEGA	200	20 ppb
Bromoform	QCSMEGA	200	20 ppb
n-Butylbenzene	QCSMEGA	200	20 ppb
Sec-Butylbenzene	QCSMEGA	200	20 ppb
Tert-Butylbenzene	QCSMEGA	200	20 ppb
Carbon tetrachloride	QCSMEGA	200	20 ppb
Chlorobenzene	QCSMEGA	200	20 ppb
Chloroform	QCSMEGA	200	20 ppb
2-Chlorotoluene (o)	QCSMEGA	200	20 ppb
4-Chlorotoluene (p)	QCSMEGA	200	20 ppb
Dibromochloromethane	QCSMEGA	200	20 ppb
1,2-Dibromo-3-chloropropane	QCSMEGA	200	20 ppb
1,2-Dibromoethane (EDB)	QCSMEGA	200	20 ppb
Dibromomethane	QCSMEGA	200	20 ppb
1,2-Dichlorobenzene	QCSMEGA	200	20 ppb
1,3-Dichlorobenzene	QCSMEGA	200	20 ppb
1,4-Dichlorobenzene	QCSMEGA	200	20 ppb
1,1-Dichloroethene	QCSMEGA	200	20 ppb
Cis-1,2-Dichloroethene	QCSMEGA	200	20 ppb
Trans-1,2-Dichloroethene	QCSMEGA	200	20 ppb
1,2-Dichloropropane	QCSMEGA	200	20 ppb
1,3-Dichloropropane	QCSMEGA	200	20 ppb
2,2-Dichloropropane	QCSMEGA	200	20 ppb
1,1-Dichloropropene	QCSMEGA	200	20 ppb
Cis-1,3-Dichloropropene	QCSMEGA	200	20 ppb
Trans-1,3-Dichloropropene	QCSMEGA	200	20 ppb
Ethylbenzene	QCSMEGA	200	20 ppb
Hexachlorobutadiene	QCSMEGA	200	20 ppb
Isopropylbenzene (Cumene)	QCSMEGA	200	20 ppb

UNCONTROLLED DOCUMENT: DO NOT Transfer or Print

Method: 02-8260B

Revision: 9

Date: August 17, 2006

Page: 43 of 52

Compound Name	Standard Mix	Stock (ppm)	Working Conc.
p-Isopropyltoluene	QCSMEGA	200	20 ppb
Methylene Chloride	QCSMEGA	200	20 ppb
Bromochloromethane	QCSMEGA	200	20 ppb
1,1-Dichloroethane	QCSMEGA	200	20 ppb
1,1,1-Trichloroethane	QCSMEGA	200	20 ppb
1,2-Dichloroethane	QCSMEGA	200	20 ppb
1,2,3-Trichloropropane	QCSMEGA	200	20 ppb
1,2,4-Trichlorobenzene	QCSMEGA	200	20 ppb
1,1,2-Trichloroethane	QCSMEGA	200	20 ppb
Trichloroethene	QCSMEGA	200	20 ppb
Naphthalene	QCSMEGA	200	20 ppb
n-Propylbenzene	QCSMEGA	200	20 ppb
Styrene	QCSMEGA	200	20 ppb
1,1,1,2-Tetrachloroethane	QCSMEGA	200	20 ppb
1,1,2,2-Tetrachloroethane	QCSMEGA	200	20 ppb
Tetrachloroethene	QCSMEGA	200	20 ppb
Toluene	QCSMEGA	200	20 ppb
1,2,3-Trichlorobenzene	QCSMEGA	200	20 ppb
1,2,4-Trimethylbenzene	QCSMEGA	200	20 ppb
1,3,5-Trimethylbenzene	QCSMEGA	200	20 ppb
m-xylene	QCSMEGA	200	20 ppb
p-xylene	QCSMEGA	200	20 ppb
o-xylene	QCSMEGA	200	20 ppb

Methyl acetate	QCS Acetates	200	20 ppb
Ethyl acetate	QCS Acetates	200	20 ppb
Vinyl Acetate	QCS Acetates	200	20 ppb

Acetone	QCS Ketones	1000	100 ppb
2-Butanone	QCS Ketones	1000	100 ppb
4-Methyl-2-pentanone	QCS Ketones	1000	100 ppb
2-Hexanone	QCS Ketones	1000	100 ppb
1,1-Dichloro-2-propanone	QCS Ketones	1000	100 ppb

Pentane	QCS Additions	200	20 ppb
3-Chloroprene (allyl chloride)	QCS Additions	200	20 ppb
Di-isobutylene	QCS Additions	200	20 ppb
1-Chlorohexane	QCS Additions	200	20 ppb
Methyl-tert-butyl ether	QCS Additions	200	20 ppb
Ethyl ether	QCS Additions	200	20 ppb
Freon 113 (1,1,2-TCTFE)	QCS Additions	200	20 ppb
Hexane	QCS Additions	200	20 ppb
Heptane	QCS Additions	200	20 ppb
Cyclohexane	QCS Additions	200	20 ppb
Benzyl chloride	QCS Additions	200	20 ppb
Iodomethane	QCS Additions	200	20 ppb
Carbon Disulfide	QCS Additions	200	20 ppb
Chloroprene	QCS Additions	200	20 ppb

UNCONTROLLED DOCUMENT: DO NOT Transfer or Print

Method: 02-8260B

Revision: 9

Date: August 17, 2006

Page: 44 of 52

Compound Name	Standard Mix	Stock (ppm)	Working Conc.
Octane	QCS Additions	200	20 ppb
Acrylonitrile	QCS Additions	1000	100 ppb
2-Nitropropane	QCS Additions	1000	100 ppb
Tetrahydrofuran	QCS Additions	1000	100 ppb
Tert-Butyl alcohol	QCS Additions	1000	100 ppb
Trans-1,4-Dichloro-2-butene	QCS Additions	200	20 ppb
Methyl methacrylate	QCS Additions	200	20 ppb
Isobutyl alcohol	QCS Additions	2000	200 ppb
Hexachloroethane	QCS Additions	200	20 ppb
Ethyl methacrylate	QCS Additions	200	20 ppb
2-Propanol	QCS Additions	1000	100 ppb
1-Propanol	QCS Additions	2000	200 ppb
Propionitrile	QCS Additions	1000	100 ppb
Methacrylonitrile	QCS Additions	200	20 ppb
1,4-Dioxane	QCS Additions	5000	500 ppb
Pentachloroethane	QCS Additions	200	20 ppb
Nitrobenzene	QCS Additions	2000	200 ppb
Methyl acrylate	QCS Additions	200	20 ppb
Chloroacetonitrile	QCS Additions	1000	100 ppb
1-Chlorobutane	QCS Additions	200	20 ppb
Dichlorofluoromethane	QCS Additions	200	20 ppb
tert-amyl methyl ether	QCS Additions	200	20 ppb
Ethyl tert-butyl ether	QCS Additions	200	20 ppb
Di-isopropyl ether	QCS Additions	200	20 ppb
Methyl cyclohexane	QCS Additions	200	20 ppb
Acetonitrile	QCS Additions	1000	100 ppb

Acrolein	Q Acrolein	1500	150 ppb
----------	------------	------	---------

2-Chloroethyl vinyl ether	QCS2CEVE	200	20 ppb
---------------------------	----------	-----	--------

Bromomethane	Qgas mix	20	20 ppb
Chloroethane	Qgas mix	20	20 ppb
Chloromethane	Qgas mix	20	20 ppb
Dichlorodifluoromethane	Qgas mix	20	20 ppb
Trichlorofluoromethane	Qgas mix	20	20 ppb
Vinyl Chloride	Qgas mix	20	20 ppb

Method: 02-8260B
Revision: 9
Date: August 17, 2006
Page: 45 of 52

APPENDIX C

TABLE 2

CALIBRATION AND QC ACCEPTANCE CRITERIA

PARAMETER	RANGE FOR Q (ug/L)	LIMIT FOR S (ug/L)	RANGE FOR X (ug/L)	RANGE FOR P, P (%)
Benzene	12.8-27.2	6.9	15.2-26.0	37-151
Bromodichloromethane	13.1-26.9	6.4	10.1-28.0	35-155
Bromoform	14.2-25.8	5.4	11.4-31.1	45-169
Bromomethane	2.8-37.2	17.9	D-41.2	D-242
Carbon Tetrachloride	14.6-25.4	5.2	17.2-23.5	70-140
Chlorobenzene	13.2-26.8	6.3	16.4-27.4	37-160
Chloroethane	7.6-32.4	11.4	8.4-40.4	14-230
2-Chloroethylvinyl ether	D-44.8	25.9	D-50.4	D-305
Chloroform	13.5-26.5	6.1	13.7-24.2	51-138
Chloromethane	D-40.8	19.8	D-45.9	D-273
Dibromochloromethane	13.5-26.5	6.1	13.8-26.6	53-149
1,2-Dichlorobenzene	12.6-27.4	7.1	11.8-34.7	18-190
1,3-Dichlorobenzene	14.6-25.4	5.5	17.0-28.8	59-156
1,4-Dichlorobenzene	12.6-27.4	7.1	11.8-34.7	18-190
1,1-Dichloroethane	14.5-25.5	5.1	14.2-28.5	59-155
1,2-Dichloroethane	13.6-26.4	6.0	14.3-27.4	49-155
1,1-Dichloroethene	10.1-29.9	9.1	3.7-42.3	D-234
trans-1,2-Dichloroethene	13.9-26.1	5.7	13.6-28.5	54-156
1,2-Dichloropropane	6.8-33.2	13.8	3.8-36.2	D-210
cis-1,3-Dichloropropene	4.8-35.2	15.8	1.0-39.0	D-227
trans-1,3-Dichloropropene	10.0-30.0	10.4	7.6-32.4	17-183
Ethylbenzene	11.8-28.2	7.5	17.4-26.7	47-150
Methylene chloride	12.1-27.9	7.4	D-41.0	D-221
1,1,2,2-Tetrachloroethane	12.1-27.9	7.4	13.5-27.2	46-157
Tetrachloroethene	14.7-25.3	5.0	17.0-26.6	64-148
Toluene	14.9-25.1	4.8	16.6-26.7	47-150
1,1,1-Trichloroethane	15.0-25.0	4.6	13.7-30.1	52-162
1,1,2-Trichloroethane	14.2-25.8	5.5	14.3-27.1	52-150
Trichloroethene	13.3-26.7	6.6	18.6-27.6	71-157
Trichlorofluoromethane	9.6-30.4	10.0	8.9-31.5	17-181
Vinyl chloride	0.8-39.2	20.0	D-43.5	D-251

Method: 02-8260B
Revision: 9
Date: August 17, 2006
Page: 48 of 52

SOP Change History Sheet

<u>Section No.</u>	<u>Section</u>	<u>Reason for Change</u>
5.2.5	Apparatus and Materials	Correction to current SOP
6.10 – 6.11	Reagents	Correction to current SOP
7.5.1	Instrument Calibration	Correction to current SOP
7.5.3.2	Instrument Calibration	DoD audit response
7.6.2	Instrument Calibrations	Correction to current SOP
8.6.4	Quality Control	Correction to current SOP
8.7.3	Quality Control	Correction to current SOP
8.5.4	Quality Control	Correction to current SOP
8.11	Quality Control	Correction to current SOP
10.3.1	Procedure	Correction to current SOP
10.3.2	Procedure	Correction to current SOP
10.3.3.4	Procedure	DoD audit response
10.3.7.7 & 9	Procedure	Correction to current SOP
10.3.7.9	Procedure	Correction to current SOP
10.3.3.12	Procedure	Correction to current SOP
10.3.4.1 & 3	Procedure	Correction to current SOP
A & B	Appendix	Correction to current SOP
Revision 8:03/06/2006		(Revisions made throughout to update Section references)
1.1	Scope and Application	Updated method revision
1.10	Scope and Application	Added project criteria requirements verbiage

Method: 02-8260B

Revision: 9

Date: August 17, 2006

Page: 49 of 52

SOP Change History Sheet (continued)

<u>Section No.</u>	<u>Section</u>	<u>Reason for Change</u>
4.2	Safety	Added MSDS availability
5.3, 5.5, 5.12	Apparatus and Materials	Added vendor information
5.9-5.11	Apparatus and Materials	Revised vendor information
5.14	Apparatus and Materials	Removed pipette reference
6.4	Reagents	Added vendor information
6.5	Reagents	Revised temperature range
6.6.2, 6.7.2.2, 6.8.4, 6.9.2.3, 6.10.3.3, 6.11.3.1, 6.11.3.3	Reagents	Added word "mixture"
6.7, 6.9	Reagents	Added abbreviation verbiage
6.8	Reagents	Added abbreviation verbiage and note
6.10.1	Reagents	Added calibration check standard verbiage, revised catalog #'s
6.10.3.1	Reagents	Revised preparation volumes
6.10.3.2, 6.11.3.2	Reagents	Revised preparation volumes, added standards
6.10.4.2	Reagents	Added volume amount of Acrolein
6.11.1	Reagents	Removed calibration check standard verbiage, revised catalog #'s
6.13	Reagents	Added DPD Free Chlorine to list
7.1	Instrument Calibration	Added instrument and recording location

Method: 02-8260B

Revision: 9

Date: August 17, 2006

Page: 50 of 52

SOP Change History Sheet (continued)

<u>Section No.</u>	<u>Section</u>	<u>Reason for Change</u>
7.4.2, 10.4.4.2, 10.4.7.2, 10.4.7.4, 11.1	Instrument Calibration Procedure Calculations	Updated reference from “Chemserver” to read “Target”
7.4.3, 7.6.1.1, 7.6.2.1, 8.14	Instrument Calibration Quality Control	Revised “USACE” to read “DoD”
7.5.4.2, 7.5.5.1, 8.7.3, 10.3.5.1, 10.4.5	Instrument Calibration Quality Control Procedure	Added DoD requirements
7.6.1	Instrument Calibration	Revised wording from “purge” to “inject”
7.6.1.1	Instrument Calibration	Added verbiage concerning scanning
8.3	Quality Control	Added verbiage about ongoing proficiency
8.6.2	Quality Control	Removed reference to third aliquot, added/revised volumes
8.7.1, 8.9.2	Quality Control	Added LCS and QC check sample preparation details
8.14	Quality Control	Added reference to SOP 99-MDL/reference method
9.3, 10.4.3	Sample Collection... Procedure	Replaced “AMS” with “Horizon”, revised comment
10.3.3.5	Procedure	Removed references for performing dilutions in 50mL flasks
10.3.3.6	Procedure	Added reference to Class A flasks
10.3.3.10	Procedure	Removed reference to vendor
10.3.4	Procedure	Added mixing directions

Method: 02-8260B
Revision: 9
Date: August 17, 2006
Page: 51 of 52

SOP Change History Sheet (continued)

<u>Section No.</u>	<u>Section</u>	<u>Reason for Change</u>
10.4.4.3	Procedure	Added sample reruns to be rerun "at a dilution"
10.4.4.4	Procedure	Added reference to SOP 99-Integration
10.4.6.3.1, 10.4.6.3.2	Procedure	Revised spike sublist reference
10.4.7.5	Procedure	Added verbiage to include "guidelines"
12.3	Reporting Results	Added verbiage for reporting limits and J-values
12.6	Reporting Results	Added instructions to date and initial 1 st and 2 nd reviews
16	Troubleshooting	Added Section
A, B	Appendix	Substantial revisions throughout both tables

Revision 9: 08/17/2006

6.10-6.11	Reagents	Made current with lab practice as per internal audit findings
A, B, E	Appendix	Made current with lab practice as per internal audit findings

Method: 03-6010B
Revision: 12
Date: February 13, 2007
Page 1 of 47

Document Title: Analysis of Total Metals by Inductively Coupled Plasma Using the TJA Trace ICP

Document Control Number: _____

Organization Title: ANALYTICAL LABORATORY SERVICE, INC. (ALSI)

Address: 34 Dogwood Lane
Middletown, PA 17057

Phone: (717) 944-5541

Approved by:

_____	_____
Helen MacMinn, Quality Assurance Manager	Date
_____	_____
Anna Milliken, Operations Manager	Date
_____	_____
Jason Knight, Validator	Date

Annual Review:

_____	_____
Reviewed By	Date Reviewed
_____	_____
Approved By	Date Approved

_____	_____
Reviewed By	Date Reviewed
_____	_____
Approved By	Date Approved

Method: 03-6010B
Revision: 12
Date: February 13, 2007
Page 3 of 47

TABLE OF CONTENTS

1	Scope and Application	4
2	Summary of Method	4
3	Interferences.....	5
4	Safety	6
5	Apparatus and Materials	6
6	Reagents.....	7
7	Instrument Calibration	16
8	Quality Control	18
9	Sample Collection, Preservation and Handling	27
10	Procedure	28
11	Calculations	31
12	Reporting Results.....	32
13	Waste Disposal.....	33
14	Pollution Prevention.....	33
15	Definitions	34
16	Troubleshooting.....	34
	Appendix A.....	36
	TABLE 1.....	37
	TABLE 2.....	38
	TABLE 3.....	39
	TABLE 4.....	40
	TABLE 5.....	41
	SOP Change Summary	42
	SOP Concurrence Form.	47

Method: 03-6010B
Revision: 12
Date: February 13, 2007
Page 4 of 47

1 Scope and Application

- 1.1 This method is adapted from the U.S. EPA SW846 Method 6010B Revision 2, December 1996, "Inductively Coupled Plasma - Atomic Emission Spectroscopy." This method is applicable to a wide variety of matrices including ground water, aqueous samples, TCLP, SPLP, ASTM extracts, industrial and organic wastes, soils, sludges, sediments, industrial hygiene paints, wipes, airs, and other solid wastes.
- 1.2 The specific elements which ALSI analyzes by this method are listed in Table 1. This table lists the wavelengths used for the appropriate elements.
- 1.3 This method is restricted to use by or under the supervision of analysts experienced in the use of an ICP. Each analyst must also be skilled in the interpretation of raw data, including quality control data.
- 1.4 All samples are digested using appropriate sample preparation techniques.
- 1.5 This document states the laboratory's policies and procedures established in order to meet requirements of all certifications/accreditations currently held by the laboratory, including the most current NELAC standards.
- 1.6 Method Detection Limits can be found on the ALSI network in the Metals folder and are maintained and updated by the QA department. The detection limits for a specific sample may differ from those listed due to the nature of interferences in a particular sample matrix. MDL studies must be performed according to SOP 99-MDL or the reference method, whichever is more frequent.
- 1.7 Individual project requirements may override criteria listed in this SOP.

2 Summary of Method

- 2.1 This method measures element-emitted light by optical spectrometry. The ICP instrument contains a torch through which flows argon gas. A spark is used to initiate a plasma of ionized argon, which is then maintained by a radio-frequency field. Samples are pulled into the system by a peristaltic pump and nebulized. The resulting aerosol is transported into the plasma torch. Element specific atomic- and ionic-line emission spectra are produced by the excited atoms or ions on their return to the ground state. The emission spectra are dispersed by a grating spectrometer, and the intensities of the lines are monitored by photomultiplier tubes. The average of two intensity exposures, along with inter-element corrections yields the final result.

(Note: For all sequence runs that involve samples from the Dept. of Defense (DoD), an average of three separate exposures is used to calculate the data.)

Method: 03-6010B

Revision: 12

Date: February 13, 2007

Page 5 of 47

- 2.2 Background correction is used for all element determinations. Background intensity is measured adjacent to the analytical lines from the samples during analysis in an area free from spectral interferences. It's then subtracted from the intensity measured at these lines.
- 2.3 The possibility for additional interferences such as spectral, chemical, and physical interferences, does exist. Appropriate corrections for these interferences must be made.

3 Interferences

- 3.1 Spectral interferences are caused by (1) overlap of a spectral line from another element at the analytical or background measurement wavelengths; (2) unresolved overlap of molecular band spectra; (3) background from continuum or recombination phenomena; and (4) stray light from the line emission of high concentration elements.
 - 3.1.1 Background contribution and stray light are normally compensated for by the background correction.
 - 3.1.2 Unresolved overlap requires the selection of an alternate wavelength or an alternate method of analysis such as graphite furnace or flame AA.
 - 3.1.3 Spectral overlap is compensated for in the Trace instrument by automatic correction of the raw data after monitoring and measuring the interfering elements. A linear relationship between the interferant levels and the false interferences they cause can be assumed. Inter-element correction factors are checked daily by analysis of an interference check solution and updated every six months, or whenever indicated by failure of the interference check solution.
- 3.2 Physical interferences are effects associated with the sample introduction and flow through the instrument. These interferences are brought about due to differences in viscosity and surface tension. Physical interferences are most commonly seen in samples containing high dissolved solids or high acid concentrations. To reduce physical interferences, ALSI uses a peristaltic pump for sample introduction, dilutions of problem samples, and matrix matching of standards to samples. Internal standard addition and the method of standard additions may also be used to compensate for physical interferences.
 - 3.2.1 Another problem that can occur while analyzing samples with high dissolved solids is salt build-up at the tip of the nebulizer, which affects aerosol flow rate and causes instrument drift. This problem is controlled by regular maintenance and by wetting the argon prior to nebulization using a humidifier.
- 3.3 Chemical interferences include molecular compound formation, ionization effects, and

Method: 03-6010B
Revision: 12
Date: February 13, 2007
Page 6 of 47

solute vaporization effects. Normally, these are not significant with the ICP. If they are encountered, they can be minimized by carefully selecting the operating conditions (RF power, torch position, and nebulizer flow rate), buffering the sample, matrix matching, and performing standard addition procedures. Chemical interferences are highly dependant on matrix type and the specific analyte of interest.

- 3.4 Memory Interferences result when analytes from a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from the build up of sample material in the plasma torch and spray chamber. This problem can be minimized by flushing the system with rinse blank between samples. If a memory interference is suspected, the sample must be reanalyzed after a rinse period.

4 Safety

- 4.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully defined; however, each chemical compound shall be treated as a potential health hazard.
- 4.2 ALSI maintains material safety data sheets (MSDS) on all chemicals used in this procedure. MSDS are available to all staff and are located in the lower level conference room in hard copy and electronically on the ALSI public file server: F:\MSDS - Material Safety Data Sheets.
- 4.3 Precautions shall be taken when handling samples and/or chemicals in the lab. The use of gloves, safety glasses, and lab coats is required when working with samples.

5 Apparatus and Materials

- 5.1 Thermo Jarrell Ash 61E Trace Simultaneous ICP Emission Spectrophotometer with the following options:
- 5.1.1 IBM compatible 386 computer
ThermoSPEC/AE v5.06 software
Axial Torch
Computer controlled emission with background correction.
Computer inter-element correction ability
Radio frequency generator coupled to a water-cooled induction coil
Water cooler
Vacuum Pump
Polychromatic optic system under vacuum
Adjustable variable speed peristaltic pump
Mass flow controllers for argon flow rate
HP LaserJet Printer
TJA 300 automatic liquid sampler
Argon humidifier

Method: 03-6010B

Revision: 12

Date: February 13, 2007

Page 7 of 47

- 5.1.2 Sample pump tubing - CPI, catalog #4062-535 or equivalent.
- 5.1.3 Internal Standard pump tubing - CPI, catalog #4062-5015 or equivalent.
- 5.1.4 Rinse pump tubing - CPI, catalog #4062-545 or equivalent.
- 5.1.5 13 x 100 polystyrene tubes - Perfector Scientific, catalog #2110 or equivalent.
- 5.1.6 Internal Standard Mixing Kit - CPI, catalog #4062-910 or equivalent.
- 5.2 Various Class A volumetric dispensing pipettes. – VWR, catalog #'s 53515-020, 53515-044, 53515-050 or equivalent.
- 5.3 Disposable Pasteur pipettes - VWR, catalog #14670-103 or equivalent.
- 5.4 Class A volumetric flasks (100 mL) – VWR, catalog # 29620-142 or equivalent.

6 Reagents

- 6.1 Concentrated Nitric Acid (HNO_3) - Baker Instra-analyzed Reagent Grade, VWR, catalog #JT9598-34 or equivalent. Store at room temperature and dispose of by the manufacturer's expiration date. If the manufacturer does not supply an expiration date, the reagent must be labeled as expiring one year from when opened. The analyst must write this date directly on the outside of the bottle using a permanent marker.
- 6.2 Concentrated Hydrochloric Acid (HCl) - Baker Analyzed Reagent Grade, VWR catalog #JT9535-33 or equivalent. Store at room temperature and dispose of by the manufacturer's expiration date. If the manufacturer does not supply an expiration date, the reagent must be labeled as expiring one year from when opened. The analyst must write this date directly on the outside of the bottle using a permanent marker.
- 6.3 Reagent water - Reagent water is water in which an interferant is not observed at the analyte of interest. For this purpose, ALSI uses a Filson Water Purification system, which provides analyte free, greater than 18.0 megohm-cm deionized water on demand. This water is used for preparation of all reagents and standards.

Method: 03-6010B
Revision: 12
Date: February 13, 2007
Page 8 of 47

6.4 Liquid Argon Supply - High purity grade, purchased from AIRGAS, or an equivalent supplier.

6.5 Stock Standard Solutions are purchased as commercially prepared NIST traceable certified solutions. When received in the lab each is assigned a unique log number and is recorded in the Standard Preparation Logbook along with the manufacturer, date of receipt, expiration date, and analyst's initials. These standards are stored at room temperature and disposed of on or before the manufacturer's expiration date.

6.5.1 SM-1339-002 (STD MIX #2) Stock Solution in 5% HNO₃/Trace HF. High Purity, or equivalent NIST certified standard. This standard contains the following elements:

<u>ELEMENT</u>	<u>CONCENTRATION</u>
Antimony (Sb)	20 mg/L
Bismuth (Bi)	100 mg/L
Boron (B)	100 mg/L
Cobalt (Co)	100 mg/L
Cooper (Cu)	100 mg/L
Iron (Fe)	100 mg/L
Manganese (Mn)	10 mg/L
Molybdenum (Mo)	100 mg/L
Nickel (Ni)	100 mg/L
Silver (Ag)	10 mg/L
Thallium (Tl)	10 mg/L
Tin (Sn)	10 mg/L
Titanium (Ti)	10 mg/L
Zinc (Zn)	50 mg/L

6.5.2 SM-1339-001 (STD MIX #1-R) Stock Solution in 5% HNO₃. High Purity Express, or equivalent NIST certified standard. This standard contains the following elements:

<u>ELEMENT</u>	<u>CONCENTRATION</u>
Aluminum (Al)	100 mg/L
Arsenic (As)	10 mg/L
Barium (Ba)	100 mg/L
Beryllium (Be)	20 mg/L
Calcium (Ca)	100 mg/L
Cadmium (Cd)	10 mg/L
Chromium (Cr)	10 mg/L
Lead (Pb)	10 mg/L
Magnesium (Mg)	100 mg/L
Potassium (K)	2000 mg/L
Selenium (Se)	100 mg/L
Sodium (Na)	2000 mg/L
Strontium (Sr)	10 mg/L
Vanadium (V)	5 mg/L

Method: 03-6010B
Revision: 12
Date: February 13, 2007
Page 9 of 47

6.5.3 QC26 Stock Solution in 5% HNO₃. QCD, catalog #QCS26K or equivalent. This standard contains the following elements:

<u>ELEMENT</u>	<u>CONCENTRATION</u>
Sb, As, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Mo, Ni, Se, Tl, Ti, V, Zn, Al, Ba, B, Si, Ag, Na, K	all at 100 mg/L

6.5.4 Strontium Stock Standard (1000 ppm) in 4% HNO₃. SCP, catalog #140-051-382 or equivalent NIST certified standard.

6.5.5 Tin Stock Standard (1000 ppm) in 20% HCl. SCP, catalog #140-052-502 or equivalent NIST certified standard.

6.5.6 Bismuth Stock Standard (1000 ppm) in 4% HNO₃. SCP, catalog #140-051-832 or equivalent NIST certified standard.

6.5.7 Scandium Stock Standard (1000 ppm) in 4% HNO₃. SCP, catalog #140-051-215 or equivalent NIST certified standard.

6.5.8 INTER18 (Solution A) in 5% HNO₃. CPI, Cat. #4400-INTR18-100 - Solution A, or equivalent NIST certified standard. This standard contains the following elements:

<u>ELEMENT</u>	<u>CONCENTRATION</u>
K	20000 mg/L
Se	500 mg/L
As, Pb, Tl	1000 mg/L
Ba, Cd, Cr	300 mg/L
Co, Cu, Ni	
V, Zn	
Mn	200 mg/L
Be	100 mg/L
Hg	50 mg/L

6.5.9 INTR18 (Solution B) in 5% HNO₃. CPI, Cat. #4400-INTR18-100 – Solution B, or equivalent NIST certified standard. This standard contains the following elements:

<u>ELEMENT</u>	<u>CONCENTRATION</u>
Ag	300 mg/L

Method: 03-6010B
Revision: 12
Date: February 13, 2007
Page 10 of 47

6.5.10 INTER5 in 2% HNO₃. CPI, Cat. #4400-INTR5-500 or equivalent NIST certified standard. This standard contains the following elements:

<u>ELEMENT</u>	<u>CONCENTRATION</u>
Al	1200 mg/L
Ca	6000 mg/L
Fe	5000 mg/L
Mg	3000 mg/L
Na	1000 mg/L

6.5.11 Arsenic Stock Standard (1000 ppm) in 4% HNO₃. SCP, catalog # 7697-37-2 or equivalent NIST certified standard.

6.6 Working Standard Solutions. Prepare in an acid matrix similar to the samples being analyzed. This is most often a 10% HNO₃ matrix, but is dependent on the type of digestion performed on the samples, and shall be adjusted to match the samples being analyzed. After preparation, each standard is assigned a unique log number and is recorded in the standard preparation logbook along with the stock solution used, the concentration of that stock, the volume used, the final volume, the matrix, the date prepared, the date it will expire, and the preparer. Prior to analysis of **DoD** QSM samples, the following calibration standards (Sections 6.6.2 through 6.6.8) are to be prepared fresh prior to analysis. (*Note: The associated working standards are prepared daily, and held at room temperature. The maximum storage life is no longer than one day.*)

6.6.1 High Calibration Standard. To a 100 mL volumetric flask containing 10 mL HNO₃ (or that which matches the sample) in reagent water, add 5 mL SM-1339-002 (STD MIX #2)(Section 6.5.1) Stock Solution and 5 mL SM-1339-001 (STD MIX # 1-R)(Section 6.5.2) Stock Solution. Bring up to volume using reagent water. The formulation of this standard must be documented in the trace analysis logbook. This standard must be prepared daily, and is stable for no longer the 24 hrs.

6.6.2 Mid Calibration Standard. Add 4 mL of High Calibration Standard to 16 mL of calibration blank in a standard vessel for the Trace ICP. Mix solution by placing cap on vessel and inverting several times. The formulation of this standard must be documented in the trace analysis logbook. This standard must be prepared daily, and is stable for no longer the 24 hrs.

6.6.3 Low Calibration Standard. Add 2 mL of High Calibration Standard to 18 mL of calibration blank in a standard vessel for the Trace ICP. Mix solution by placing a cap on the vessel and inverting several times. The formulation of this standard

Method: 03-6010B
Revision: 12
Date: February 13, 2007
Page 11 of 47

must be documented in the trace analysis logbook. This standard must be prepared daily, and is stable for no longer the 24 hrs.

- 6.6.4 Calibration Blank (<detection limit (DL)). To 1000 mL volumetric flask, add 100 mL HNO₃ (or that which matches the samples) and bring up to volume using reagent water. Transfer to a labeled polyethylene bottle. The calibration blank standard is stable for 3 months.

(Note: For all sequence runs that involve samples from the Dept. of Defense (DoD), the calibration reagent blank must have a concentration less than 2x the method detection limit for each analyte.

Calibration Standard Concentrations (mg/L)

Element	High Cal. Std.	Mid Cal. Std.	Low Cal. Std.
Ag	0.5	0.10	0.05
Al	5.0	1.0	0.5
As	0.5	0.10	0.05
Ba	5.0	1.0	0.5
Be	1.0	0.20	0.10
Ca	5.0	1.0	0.5
Cd	0.5	0.10	0.05
Co	5.0	1.0	0.5
Cr	0.5	0.10	0.05
Cu	5.0	1.0	0.5
Fe	5.0	1.0	0.5
Mg	5.0	1.0	0.5
Mn	0.5	0.10	0.05
Mo	5.0	1.0	0.5
Ni	5.0	1.0	0.5
Pb	0.5	0.10	0.05
Se	5.0	1.0	0.5
Sn	0.5	0.10	0.05
Sr	0.5	0.10	0.05
Ti	0.5	0.10	0.05
Tl	0.5	0.10	0.05
V	0.25	0.05	0.025
Sb	1.0	0.20	0.10
Bi	5.0	1.0	0.5
B	5.0	1.0	0.5
Zn	2.5	0.50	0.25

Method: 03-6010B**Revision: 12****Date: February 13, 2007****Page 12 of 47**

- 6.6.5 Working Profile Solution (5.0 mg/L). To a 1000 mL volumetric flask containing 100 mL HNO₃ (it is not critical for this standard to be matrix matched to the samples being analyzed) in reagent water, add 5 mL Arsenic Stock Standard (Section 6.13). Bring up to volume using reagent water. This profile solution must be documented in the metals Standard Reagent Logbook and transferred to a labeled polyethylene bottle. Working standard solutions are stable for 3 months, when stored at room temperature.
- 6.6.6 Initial Calibration Verification Standard QC26 (1.0 mg/L). To a 100 mL volumetric flask containing 10 mL HNO₃ (or that which matches the sample) in reagent water, add 1 mL QC26 Stock Solution, 0.1 mL Strontium Stock Standard (Section 6.5.4), 0.1 mL Tin Stock Standard (Section 6.5.5), and 0.1 mL Bismuth Stock Standard (Section 6.5.6). Bring up to volume using reagent water. . The formulation of this standard must be documented in the trace analysis logbook. This standard must be prepared daily, and is stable for no longer the 24 hrs.
- 6.6.7 Working Interference Check Solution. To a 1000 mL volumetric flask containing 100 mL HNO₃ (or that which matches the sample) in reagent water, add 2.5 mL INTER18 Stock Solution A, 2.5 mL INTER18 Stock Solution B, and 25 mL INTER5 Stock Solution. Bring up to volume using reagent water. Transfer to a labeled polyethylene bottle. Working standard solutions are stable for 3 months, when stored at room temperature.

<u>ELEMENT</u>	<u>CONCENTRATION</u>
Ag	0.75 mg/L
Al	30 mg/L
As	2.5 mg/L
Ba	0.75 mg/L
Be	0.25 mg/L
Ca	150 mg/L
Cd	0.75 mg/L
Co	0.75 mg/L
Cr	0.75 mg/L
Cu	0.75 mg/L
Fe	125 mg/L
Mg	75 mg/L
Mn	0.25 mg/L
Ni	0.75 mg/L
Tl	2.5 mg/L
V	0.75 mg/L
Zn	0.75 mg/L
Pb	2.5 mg/L
Se	1.25 mg/L
Na	25 mg/L
K	25 mg/L

Method: 03-6010B
Revision: 12
Date: February 13, 2007
Page 13 of 47

- 6.6.8 RPL Standard (CRI). To a 25 mL polyethylene analysis tube add 0.1 mL of RPL Stock Standard (Section 6.10). Using the 10 mL Finn pipette, add 19.9 mL 10% HNO₃ reagent blank (or that which matches the samples). This standard shall be prepared daily, and documented in the sample analysis logbook. The RPL / CRI concentrations are listed in Table 2.
- 6.7 Rinse Solution. To the rinse reservoir containing reagent water, add 300 mL HNO₃ (or that which matches the samples). Bring up to volume (3 L) using reagent water. Prepare as needed.
- 6.8 Working Internal Standard Solution (10.0 mg/L Sc). To a 2000 mL volumetric flask containing 40 mL HNO₃ in reagent water, (or that which matches the samples), add 10 mL of Scandium Stock Solution (Section 6.5.7), and 50 grams of solid cesium chloride (Section 6.34). Bring up to volume using reagent water. Transfer to a labeled polyethylene jug. Working standard solutions are stable for 3 months when stored at room temperature.
- 6.9 Instrument Performance Check Solution (IPC). To a 25 mL polyethylene analysis tube add 10 mL of 10% HNO₃ reagent blank (or that which matches the sample), and 10 mL of High Calibration Standard (Section 6.6.1). This standard will need to be prepared throughout the analysis run depending upon how many samples are being analyzed. This Working Calibration standard solution needs to be prepared daily with every analysis calibration. This standard contains the following elements:

<u>ELEMENT</u>	<u>CONCENTRATION</u>
Ag	0.25 mg/L
Al	2.5 mg/L
As	0.25 mg/L
Ba	2.5 mg/L
Be	0.50 mg/L
Ca	2.5 mg/L
Cd	0.25 mg/L
Co	2.5 mg/L
Cr	0.25 mg/L
Cu	2.5 mg/L
Fe	2.5 mg/L
Mg	2.5 mg/L
Mn	0.25 mg/L
Mo	2.5 mg/L
Ni	2.5 mg/L
Pb	0.25 mg/L
Se	2.5 mg/L
Sn	0.25 mg/L

Method: 03-6010B
Revision: 12
Date: February 13, 2007
Page 14 of 47

<u>ELEMENT</u>	<u>CONCENTRATION</u>
Sr	0.25 mg/L
Ti	0.25 mg/L
Tl	0.25 mg/L
V	0.125 mg/L
Zn	1.25 mg/L
Sb	0.50 mg/L
Bi	2.5 mg/L
B	2.5 mg/L

- 6.10 Report Limit Stock Standard (RPL / CRI) is prepared by adding the following amounts of single element standard to a 200 mL volumetric flask containing 5 mL conc. HNO₃. This working standard is stable for three months when stored at room temperature.

<u>ELEMENT</u>	<u>Volume of 1000 ppm Single Element Solution in 200 mL.</u>	<u>Final Concentration (mg/L)</u>
Aluminum	4.00 mL	20
Antimony	1.00 mL	5.0
Arsenic	0.40 mL	2.0
Barium	0.40 mL	2.0
Beryllium	0.16 mL	0.8
Bismuth	2.00 mL	10.0
Boron	4.00 mL	20.0
Cadmium	0.08 mL	0.40
Calcium	4.00 mL	20.0
Chromium	0.20 mL	1.0
Cobalt	0.20 mL	1.0
Copper	0.40mL	2.0
Iron	2.40mL	12.0
Lead	0.24 mL	1.2
Magnesium	4.00 mL	20.0
Manganese	0.20 mL	1.0
Molybdenum	0.80 mL	4.0
Selenium	0.80 mL	4.0
Silver	.16 mL	0.8
Nickel	0.80 mL	4.0
Tin	0.80 mL	4.0
Strontium	0.20 mL	1.0
Titanium	0.80 mL	4.0
Thallium	0.80 mL	4.0
Vanadium	0.20 mL	1.0
Zinc	0.80 mL	4.0

- 6.11 Silver Stock Standard (1000 ppm) in 4% HNO₃. SCP catalog #140-051-472 or

Method: 03-6010B
Revision: 12
Date: February 13, 2007
Page 15 of 47

- equivalent NIST certified standard.
- 6.12 Aluminum Stock Standard (1000 ppm) in 4% HNO₃. SCP catalog #140-051-132 or equivalent NIST certified standard.
- 6.13 Arsenic Stock Standard (1000 ppm) in 4% HNO₃. SCP catalog #140-051-332 or equivalent NIST certified standard.
- 6.14 Barium Stock Standard (1000 ppm) in 4% HNO₃. SCP catalog #140-051-562 or equivalent NIST certified standard.
- 6.15 Beryllium Stock Standard (1000 ppm) in 4% HNO₃. SCP catalog #140-051-042 or equivalent NIST certified standard.
- 6.16 Calcium Stock Standard (1000 ppm) in 4% HNO₃. SCP catalog #140-051-202 or equivalent NIST certified standard.
- 6.17 Cadmium Stock Standard (1000 ppm) in 4% HNO₃. SCP catalog #140-051-482 or equivalent NIST certified standard.
- 6.18 Cobalt Stock Standard (1000 ppm) in 4% HNO₃. SCP catalog #140-051-272 or equivalent NIST certified standard.
- 6.19 Chromium Stock Standard (1000 ppm) in 4% HNO₃. SCP catalog #140-051-242 or equivalent NIST certified standard.
- 6.20 Copper Stock Standard (1000 ppm) in 4% HNO₃. SCP catalog #140-051-292 or equivalent NIST certified standard.
- 6.21 Iron Stock Standard (1000 ppm) in 4% HNO₃. SCP catalog #140-051-262 or equivalent NIST certified standard.
- 6.22 Magnesium Stock Standard (1000 ppm) in 4% HNO₃. SCP catalog #140-051-122 or equivalent NIST certified standard.
- 6.23 Manganese Stock Standard (1000 ppm) in 4% HNO₃. SCP catalog #140-051-252 or equivalent NIST certified standard.
- 6.24 Molybdenum Stock Standard (1000 ppm) in H₂O. SCP catalog #140-050-422 or equivalent NIST certified standard.
- 6.25 Nickel Stock Standard (1000 ppm) in 4% HNO₃. SCP catalog #140-051-282 or equivalent NIST certified standard.

Method: 03-6010B
Revision: 12
Date: February 13, 2007
Page 16 of 47

- 6.26 Lead Stock Standard (1000 ppm) in 4% HNO₃. SCP catalog #140-051-822 or equivalent NIST certified standard.
- 6.27 Selenium Stock Standard (1000 ppm) in 4% HNO₃. SCP catalog #140-051-342 or equivalent NIST certified standard.
- 6.28 Titanium Stock Standard (1000 ppm) in H₂O/Trace HF. SCP catalog #140-050-222 or equivalent NIST certified standard.
- 6.29 Thallium Stock Standard (1000 ppm) in 4% HNO₃. SCP catalog #140-051-812 or equivalent NIST certified standard.
- 6.30 Vanadium Stock Standard (1000 ppm) in 4% HNO₃. SCP catalog #140-051-232 or equivalent NIST certified standard.
- 6.31 Zinc Stock Standard (1000 ppm) in 4% HNO₃. SCP catalog #140-051-302 or equivalent NIST certified standard.
- 6.32 Antimony Stock Standard (1000 ppm) in 4% HNO₃. SCP catalog #140-051-512 or equivalent NIST certified standard.
- 6.33 Boron Stock Standard (1000 ppm) in H₂O. SCP catalog #140-050-052 or equivalent NIST certified standard.
- 6.34 Cesium Chloride (100%). VWR catalog #4042-02 or equivalent NIST certified standard.

7 Instrument Calibration

- 7.1 Immediately preceding calibration, each spectral line must be centered on its exit slit which is positioned in front of each photomultiplier tube. Maintaining this optical alignment during operation is called profiling.
 - 7.1.1 Only the scandium spectral line is profiled. All other lines are preset relative to scandium.
 - 7.1.2 To profile the instrument, aspirate the Working Profile Solution. From the main menu, enter Analysis. Enter the method name to be used and press Enter. Press F5, Profile. The profile line shall read Sc 361.384.
 - 7.1.3 Press F3, Automatic. When the Working Profile solution has reached the plasma, press F1, Run. After scanning is complete, the scandium peak is displayed on the screen, along with the peak position, peak intensity and peak width.

Method: 03-6010B
Revision: 12
Date: February 13, 2007
Page 17 of 47

- 7.1.4 In order to get the peak position as close to the center as possible, the peak position number shall be as close to $0.00 \pm .02$ as possible. This is accomplished by choosing F1, calc SS, and pressing enter. The new vernier (Hg profile) position will be displayed on the screen. Adjust the vernier knob on the instrument manually to the value displayed on the screen. Then press F9, Done/Keep.
- 7.1.5 Go to Section 7.1.3 and repeat the process until the peak position is as close to 0.00 ± 0.02 as possible. Then press F9, Done/Keep and F9, Done. Allow the system to rinse for 5 minutes before starting calibration.
- 7.1.6 Re-profiling may be performed periodically throughout the analysis to compensate for diurnal changes, which are apparent by drifting QCs. It must only be adjusted, however, after instrument check standards and calibration blank have been analyzed.
- 7.2 The instrument prepares a standard curve by analyzing three levels of calibration standards and a calibration blank. Starting with the blank and working toward the high standard, the standards are aspirated and emission intensity readings are recorded by the data system.
- 7.3 All calibration standards are analyzed in duplicate and an average intensity is reported and used by the data system to prepare the calibration curve.
(Note: All runs involving Dept. of defense (DoD) samples require three replicates for calibration standards.)
- 7.4 A daily calibration curve is created by plotting the average intensity readings on the y-axis and concentration readings on the x-axis. The software of the data system plots the curve in a linear configuration. The calibration curve occurring most immediately preceding a particular sample is used to calculate the concentration for that sample. The acceptance criteria for a calibration curve for all analytes is a correlation coefficient of 0.995 or greater.
- 7.5 The calibration curve is validated using instrument check solutions prepared at known concentrations from a different source than that of the calibration standards. Validation occurs immediately following calibration and then at a frequency of 10% throughout the analysis run.

Method: 03-6010B
Revision: 12
Date: February 13, 2007
Page 18 of 47

8 Quality Control

8.1 Initial demonstration of performance

8.1.1 Linear Dynamic Range (LDR) - The upper limit of linearity must be established for each element being analyzed. Analyze successively higher concentrations of the analyte until the percent recovery falls under 90%. The last concentration maintaining greater or equal to 90% recovery is considered the upper limit of linearity. Samples containing analytes greater than 90% of the upper limit of linearity must be diluted and reanalyzed for those analytes. The LDRs are verified every six months or any time a change in operating conditions occurs that may change the LDR.

(Note: When analyzing samples from DoD (Dept. of Defense), the upper linearities must be verified in every analytical run. A verification standard can be analyzed either before or after the DoD samples in your analysis run. If the analyte recoveries are within 10% of their known value, the upper linear ranges can be used. Otherwise, all samples must be diluted to analyte concentrations, which fall within the calibration curve.)

8.1.2 Method Detection Limits (MDL) - MDLs must be established and verified annually, and any time a change in operating conditions occurs that may change the MDL (reference 99-MDL). When analyzing Dept. of Defense (DoD) samples, MDL studies must be performed annually followed by an MDL Verification Check Standard analyzed at 2X the MDL. If The MDL verification standard is not recovered within at least 3X the instruments noise level, a new set of MDLs at a higher concentration will be analyzed. Once the MDL levels have been established, the Lower Quantitation Limits / Reporting Limits (RPL) can be set. The RPL shall be at least 3X the MDL.

8.1.3 Demonstration of Capability (DOC) – DOCs must be performed yearly by each analyst prior to performing this method and repeated at any time there is a significant change in instrument type (See: QA Plan, technical Training). To perform DOCs, four consecutive Laboratory Control Samples (LCS), with a matrix matching that of the calibration standards are analyzed. The recoveries obtained must be within 80-120% of the known values for each associated metal, and consecutive reads must have an RSD less than 20%. If the DOCs are outside these acceptance limits, a new calibration curve must be established, and the LCS's reanalyzed. This process is repeated until the DOCs are completed successfully.

8.1.4 Interelement correction factors must be verified and updated every six months or at any time a change in instrument operating conditions occurs which may change the interelement correction requirements. The IECs are calculated while performing the bi-yearly linear dynamic ranges (Section 8.1.1). When the upper

Method: 03-6010B
Revision: 12
Date: February 13, 2007
Page 19 of 47

limit for each analyte is established, the recovery effects on the remaining analytes must be updated. Any effect, whether positive or negative the analyte detection limit, must be corrected in the method IEC table. The IEC correction is calculated by dividing the affected analytes recovery by the concentration of the linear range standard being analyzed. The resulting correction must then be manually added or subtracted to the existing IEC. (ex.: An upper limit is established for Fe at 900 ppm. The Pb recovery is -0.006 ppm when Fe is 900 ppm. Since the Pb reporting limit is 0.005 ppm, the IEC is in need of correction. The -0.006 ppm result is divided by 900 yielding a correction factor of -0.000000666.)

- 8.1.5 Prior to performing analysis on NLLAP samples (IH), analysts will have read through the latest AALA lead requirements, and have demonstrated ability to produce reliable results through accurate analysis of standard reference material (i.e. PAT rounds or ELPAT studies), or in-house quality control samples. Their performance must be documented in their training logs.
 - 8.1.6 Analysts/Technicians involved in IH Lead analysis shall demonstrate their ability to adequately analyze certified reference materials (i.e. PAT rounds or ELPAT studies), on a bi-yearly basis. Their performance must be documented in their training log.
 - 8.1.7 Contamination Control – Lead dust wipe sampling must be performed in all associated areas of the lab on a quarterly basis to determine surface concentrations of lead. Sample preparation and analysis is not to proceed until surface contamination is less than the specified maximum allowable limit of 40 micrograms per square foot.
 - 8.1.8 Instrument Detection Limit (IDL). The IDLs for the Trace will be performed Quarterly. This study is performed following a standard calibration, and includes analyzing ten calibration blanks on three non-consecutive days. Each measurement must be performed as though it were a separate analytical sample. (i.e., each measurement must be followed by a rinse and/or any other procedure normally performed between the analyses of separate samples. The IDL responses are then manually entered off the raw data into an excel spreadsheet which then calculates the Standard Deviation. This number shall then be used as a starting point for setting up your MDL standards. (Note: the IDL limits shall be less than the MDL limits. If they are not, the IDL and MDL studies must be reviewed for errors, and redone if necessary.)
- 8.2 Daily demonstration of instrument performance
- 8.2.1 Quality Control Sample (QC26) - Initial and periodic verification of calibration standards is necessary to verify instrument performance. To verify the calibration

Method: 03-6010B
Revision: 12
Date: February 13, 2007
Page 20 of 47

standards, the Working Quality Control Sample QC26 must be within $\pm 10\%$ of the true value immediately following the daily calibration. If outside of this acceptable range for target elements, the problem must be corrected by re-analysis, preparation, recalibration, or instrument maintenance. Samples may not be analyzed until the problem has been corrected and a QCS has been recovered within acceptable range.

- 8.2.2 A laboratory method blank (MB) is prepared with every batch of samples or one per every 20 samples digested, if the batch contains more than 20 samples. The method blank must be less than 1/10 of the concentration of any sample in the associated batch or less than 1/10 of the specific regulatory limit (MCL) if the concentration exceeds the concentration of the sample. Otherwise, the source of the contamination must be investigated and measures taken to correct, minimize, or eliminate the problem. Any sample associated with a contaminated method blank shall be reprocessed or the results reported with an appropriate data qualifier.

(Note: The method blank result must be equal to or less than 1/2 the reporting limit for all sequence batches that include samples from the Dept. of Defense (DoD))

8.2.2.1 IH Lead Wipe QC. The wipe media used for the quality control samples (i.e. MB / LCS / LCSD), shall be of the same lot number or manufacturer as the wipes used for sample collection. If the samples are collected by an outside source, the lab is requesting that extra wipe media is provided to perform the required QC. If the wipe media is not provided by the client, then ALSI will use in-house wipe media purchased from Environmental Express.

- 8.2.3 A laboratory fortified blank (LCS) is processed with every batch of samples, or one every 20 samples if the batch contains more than 20 samples. The LCS must be subjected to all sample preparation steps, such as digestion, if necessary. The percent recovery must be 80-120% of the true spike value. If the recovery falls outside of this range, the source of the problem shall be identified and resolved before continuing analyses. LCS results are documented in the ALSI LIMS system, which can produce Control Charts that will show method recovery trends over time.

8.2.3.1 To prepare a laboratory fortified blank while analyzing samples which do not require digestion, add 1 mL SM-1339-002 (STD MIX #2) (Section 6.5.1) Stock Solution and 1 mL SM-1339-001 (STD MIX # 1-R) (Section 6.5.2) Stock Solution to 100 mL of sample. This will result in a mid-level concentration for the LCS.

8.2.3.2 Calculating LCS Recoveries:

Method: 03-6010B
Revision: 12
Date: February 13, 2007
Page 21 of 47

$$\% \text{ Recovery} = (C_m/C_n) \times 100$$

where: C_m = measured concentration of LCS

C_n = spiking concentration

- 8.2.4 Instrument Performance Check Solution (IPC) is analyzed following the calibration, after every 10 samples and at the end of the run. The results of the analytes in the check solution immediately following calibration must be within $\pm 10\%$ of the true value with $<5\%$ RSD between replicates. Subsequent analyses of the IPC solution must be within $\pm 10\%$ of the true value. If the result falls outside of this range for any element, the IPC may be rerun for that element. If the result still falls outside of this range, the problem must be corrected by re-profiling, preparation of new IPC, recalibration, or instrument maintenance. Samples following the last acceptable IPC requiring the elements that failed must be reanalyzed after correction of the problem and successful analysis of the IPC. All results of IPCs are documented in the ALSI LIMS system.
- 8.2.5 The Working Interference Check Solution (ICS) is analyzed to provide an adequate test of the inter-element and background correction factors. It must be analyzed at the beginning and end of an analytical run or twice during every 8-hour shift, whichever is more frequent. The result must be within $\pm 20\%$ of the true value. If the results fall outside of this range, the interference check solution may be rerun. If it is still outside this range for any element, the problem must be corrected by preparation of new working interference solution, recalibration, or adjustment of inter-element and background correction factors. Any sample requiring an element that fails must be rerun for that element after the problem had been corrected and the interference check successfully meets the criteria.
- 8.2.6 Calibration reagent blanks are analyzed directly after each IPC. The result for every element being analyzed must be less than $2.2 \times$ the MDL. If the result does not meet this requirement for any element, the calibration blank may be rerun for that element. If the result is still not acceptable, the problem must be corrected by preparation of new blank, recalibration, or instrument maintenance. Samples following the last acceptable calibration reagent blank requiring the elements that failed must be reanalyzed after correction of the problem and successful analysis of the calibration reagent blank. If reanalysis is not possible, data shall be reported with a qualifying statement.

Note: For analysis runs involving samples from The Dept. of Defense (DoD), the calibration blanks must have analyte concentrations less than $2x$ the MDL. Otherwise, the source of the contamination must be investigated, and measures taken to minimize or eliminate the problem before the samples can be reanalyzed. If reanalysis is not possible, data shall be reported with a qualifying statement.

Method: 03-6010B
Revision: 12
Date: February 13, 2007
Page: 22 of 47

8.3 Daily demonstration of data quality

8.3.1 A matrix spike sample is processed at a frequency of 10% of the routine samples. Per EPA Method 6010 requirements the percent recovery must be 75-125% of the true spike value. Recovery calculations are not required if the concentration of the spike added is greater than ten times the sample background and a comment must be placed on the lab report. If the recovery falls outside of the acceptable range, and the system is found to be in control (Section 8.2), the recovery problem is judged to be matrix related and not system related. To determine if the method of standard additions is necessary, the sample must be post spiked or diluted as described below. Results of all matrix spikes are documented in the ALSI LIMS system.

(Note: When analyzing samples from the Dept. of Defense (DoD) the matrix spike must be recovered at 80-120% of the true spiked value.)

$$\% \text{ Recovery} = \frac{MS \text{ Conc.} - \text{Sample Conc.}}{\text{Actual Spike Concentration}} \times 100$$

8.3.2 Analyte Addition Test. To prepare a post spike, add an amount of high standard that will produce a minimum level of 20x and a maximum level of 100x the MDL and analyze. The percent recovery must be 75-125% of the true value as determined by the following formula. If the result is outside of this range, make successive dilutions of the sample and re-spike until the recovery falls within this range. The reporting limit of the sample must then be raised to reflect the dilution used. If this raises the reporting limit higher than the client needs, the method of standard additions shall be performed. NOTE: If the analyte concentration is greater than ten times the matrix spike concentration, post-spikes are not required, but a comment must be added to the lab report stating "No Spike Calculated".

$$A = \frac{B - \left(\frac{E}{F}\right)(C)}{\left(\frac{E}{F}\right)(D)} \times 100$$

where:

- A = Post spike percent recovery
- B = Resulting spike concentration
- C = Sample concentration
- D = Working calibration standard concentration
- E = Amount of spike added (mL)
- F = Final volume spike solution + sample

(Note: When analyzing samples from the Dept. of Defense (DoD) the post digestion spikes must be recovered at 80-120% of the true spiked value.)

8.3.3 Dilution Test. If $(100 \times \text{MDL}) < 20\%$ of the sample concentration, prepare a 1/5 dilution on the sample and reanalyze. The resulting corrected concentration shall be within $\pm 10\%$ of the original sample concentration. If not, a matrix effect shall

Method: 03-6010B
Revision: 12
Date: February 13, 2007
Page: 23 of 47

be suspected, and the sample diluted until the matrix problem has been eliminated. If this raises the reporting limit higher than the client needs, the method of standard additions shall be performed.

- 8.3.4 A matrix spike duplicate or sample duplicate is processed at a frequency of 5% of the routine samples. The relative percent difference (RPD) must be within 20%. If the RPD falls outside of the acceptable range, and the system is found to be in control, the precision problem is judged to be matrix related and not system related. A comment must be added to the lab report. Results of all duplicate analyses are documented in the ALSI LIMS system.

$$RPD = \text{Difference} / \text{Average} \times 100$$

- 8.4 Method of Standard Additions - The method of standard additions is used when sample dilution and spikes fail to produce good recoveries. In the standard addition technique, two identical aliquots (Volume V_x) of the sample solution are taken. To the first, (labeled A), is added a small a volume (V_s) of a standard analyte solution of concentration C_s . To the second (labeled B), is added the same volume V_s of the matrix blank. The intensity counts of A and B are measured and corrected for non-analyte intensity counts. The unknown sample concentration (C_x) is calculated as follows:

$$C_x = \frac{S_B V_s C_s}{(S_A - S_B) V_x}$$

where:

S_A = intensity counts of A corrected for the blank

S_B = intensity counts of B corrected for the blank

V_s and C_s shall be chosen so that S_A is roughly twice S_B on the average. It is best if V_s is much less than V_x , and thus C_s is much greater than C_x , to avoid excess dilution of the sample matrix. If a concentration or separation step is used, the additions are best made first and carried through the entire procedure. For results from this technique to be valid, the following limitations must be taken into consideration:

- 8.4.1 The analytical curve must be linear.
- 8.4.2 The chemical form of the analyte added must respond the same as the analyte in the sample.
- 8.4.3 The interference effect must be constant over the working range of concern.
- 8.4.4 The signal must be corrected for any additive interference.
- 8.5 Samples resulting in high negative ($|\text{conc.}| > \text{reporting limit}$) concentrations must be post-

Method: 03-6010B
Revision: 12
Date: February 13, 2007
Page 24 of 47

spiked (Section 8.3.2) to determine if there exists a negative interference. If multiple samples containing the same matrix (from the same source or client) show the same negative trend, only one sample of this matrix needs to be post-spiked.

- 8.6 When analyzing using a scandium internal standard, the intensity of scandium in each sample must be within $\pm 30\%$ of the intensities in the initial calibration blank. If the intensities fall outside of this range, re-analyze the sample at a dilution to eliminate matrix interferences.
- 8.7 Each sample, QC check, and calibration standard is analyzed in duplicate and the results averaged. The relative standard deviation (RSD) between sample replicates must be less than 20% for all concentrations greater than the reporting limit to be accepted. If the RSD is greater than 20% and the sample concentration is above the reporting limit, the sample must be reanalyzed.
(Note: All runs involving DoD samples require three replicates for standards, samples, and QC Checks.)
- 8.8 It is recommended that whenever a new or unusual sample matrix is encountered, either of the following tests be performed to determine if either positive or negative matrix interferences are present to distort the accuracy of the reported values.
- 8.8.1 Serial dilution. If the analyte concentration is at least 40x the detection limit, a 1:4 dilution shall agree within $\pm 10\%$ of the original determination. If not, a chemical or physical interference shall be suspected and must either be diluted out and the detection level raised or the sample may be analyzed by the method of standard addition.
- 8.8.2 Post digestion spike. A post digestion spike prepared as directed in Section 8.3.2, shall be recovered within 75 to 125% of the true value. If the spike is not recovered and the necessary sample dilution to recover the analyte concerned raises the detection limit of the sample above the limit needed by the client, the method of standard additions must be used.
- 8.9 ALSI participates regularly in applicable performance evaluation studies conducted by various certifying organizations.
- 8.10 All policies and procedures in the most current revision of the ALSI QA Plan shall be followed when performing this procedure.
- 8.11 Ongoing proficiency must be established annually as specified in the QA Plan.

Quality Control Requirements

(Specific Project Requirements may override these requirements)

UNCONTROLLED DOCUMENT: DO NOT Transfer or Print

Method: 03-6010B

Revision: 12

Date: February 13, 2007

Page 25 of 47

Parameter	Concentration	Frequency	Control Limits	Corrective Action
Calibration Reagent Blank	--	Beginning of run, after every 10 samples, and at the end of the run.	< 2.2 x MDL < 2X the MDL for runs involving samples from the Dept. of Defense (DoD)	Reanalyze the blank, prepare new blank and analyze, perform maintenance on instrument, recalibrate, reanalyze any samples since the last acceptable blank. If reanalysis is not possible, report with a qualifying comment.
Method Blank (MB)	--	One per batch of no more than 20 samples. Analyze with associated sample batch.	< Reporting Limit or < 1/2 reporting limit for DoD QSM samples	Reanalyze the blank. Samples in the batch must be < the reporting limit or ≥ 10x the method blank. If not, samples must be redigested and reanalyzed. If reanalysis is not possible, report with a qualifying comment. For DoD QSM samples the method blank must be 1/2 the reporting limit
High Calibration Standard	See Section 6.6.1	After calibration and before analysis of samples.	90-110%	Reanalyze the High Standard. If the standard is still not acceptable, perform instrument maintenance, and prepare a new calibration.
*Laboratory Fortified Blank (LFB or LCS)	Listed in Table 3	One per batch of no more than 20 samples. Analyze with associated sample batch.	80-120%	Reanalyze the LFB. If still outside of acceptable range, samples must be redigested and reanalyzed. If reanalysis is not possible, report with a qualifying comment.
Quality Control Sample (QCS) Second Source Standard	1.0 mg/L	Immediately after calibration.	90-110%	Reanalyze the QCS. If the standard is still not acceptable, perform instrument maintenance, and prepare a new calibration.
Instrument Performance Check Solution (IPC) Same Source	Listed in Table 4	Beginning of run, after every 10 samples, and at the end of the run.	90-110%	Reanalyze the IPC. If the standard is still not acceptable, perform instrument maintenance, and prepare a new calibration. Reanalyze any samples since the last acceptable IPC. If reanalysis is not possible, report with a qualifying comment.
Reporting Limit Standard (RPL)	Listed in Table 2	Beginning of run, after calibration.	50-150% Dept. of Defense samples (DoD) (80-120%)	Reanalyze the RPL. If the standard is still not acceptable, perform instrument maintenance, prepare a new calibration, and reanalyze.

UNCONTROLLED DOCUMENT: DO NOT Transfer or Print

Method: 03-6010B
Revision: 12
Date: February 13, 2007
Page 26 of 47

Parameter	Concentration	Frequency	Control Limits	Corrective Action
* Matrix Spike (MS)	Listed in Table 3	One every 10 samples with at least one per batch.	75-125% <i>Dept. of Defense samples (DoD)</i> 80-120% <i>50-150% for TCLP leachates</i>	Post Spike the sample for the failing analyte. If the post spike recovery is outside of the acceptable 75-125% limit, the sample will need to be analyzed at a 1:1 dilution and post spiked at a 1:1 dilution. If the post spike recovery is still outside acceptable limits, the sample will need to be further diluted and post spiked until an acceptable recovery has been reached. Report the results with a qualifying comment. If the matrix interference is identified, and the batch LCS was recovered within acceptable limits, report with a qualifying statement. (example: The sample in question contains % level iron.)
*Duplicate or matrix spike duplicate (MSD)	--	One every 10 samples with at least one per batch.	RPD ≤ 20%	Reanalyze the duplicate. If the RPD is still >20% or if reanalysis is not possible, report the results with a qualifying comment.
Sample replicates	--	Every sample	RSD < 20% for all samples > reporting limit	Reanalyze the sample. If the RSD is still > 20%, check to see if the relative percent difference of the four obtained exposures is less than 20%. If so, average the four exposures, and report the result with a qualifying comment. If the RPD is greater than 20%, reanalyze the sample a third time, and average all six exposures. Report the results with a qualifying comment.
Samples with high negative concentration	--	--	(conc. < reporting limit	Post-spike sample to determine if there exists a negative interference. If multiple samples containing the same matrix (from the same source or client) show the same negative trend, only one sample of this matrix needs to be post-spiked.
Scandium Internal Standard	--	Every sample	Intensity must be within 30% of the intensity of the initial calibration blank	Reanalyze the sample at the lowest possible dilution to eliminate sample matrix interference. The detection limits then need to be raised accordingly.

Method: 03-6010B
Revision: 12
Date: February 13, 2007
Page 27 of 47

Parameter	Concentration	Frequency	Control Limits	Corrective Action
Interference Check Solution	See Section 6.6.7	Beginning and end of every run, and at least once every 8 hours.	80-120%	Rerun the interference check solution, preparation of new solution, recalibrate, or adjustment of interelement correction factors. Rerun any samples requiring an element that fails, or if re-analysis is not possible, report the results with a qualifying comment.

- * Samples selected for duplicate and matrix spike analysis shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a duplicate or spike may indicate a problem with the sample composition and shall be reported to the client whose sample produced the poor recovery.
- LCS recoveries and duplicate precision limits stated in the QC Chart are also used for all IH analyses.

9 Sample Collection, Preservation and Handling

9.1 Sample Collection.

9.1.1 Samples must be collected in plastic or glass containers.

9.1.2 Soil/Sediment/Solid samples can be collected in plastic or glass containers.

9.1.3 A minimum of 3.00 g for soil samples, and 150 mL for water samples must be supplied by the sampler in order for the laboratory to perform analysis.

9.2 Sample Preservation

9.2.1 Preserve aqueous samples using HNO₃ to a pH<2. Sample preservation shall be performed immediately upon sample collection. If this is not possible, then samples shall be preserved ASAP when received by the laboratory.

9.3 Sample Handling

9.3.1 All samples must be analyzed within 180 days of collection. All samples not analyzed within this time frame must be discarded and re-sampled for analysis, unless permission is given by the client to run the sample past its hold time. If this occurs, it must be clearly noted on the laboratory report.

9.3.2 Soil samples must be preserved above the freezing point of water up to 6° C until analysis.

9.3.3 Water samples and sample digestates shall be stored at room temperature both

Method: 03-6010B
Revision: 12
Date: February 13, 2007
Page 28 of 47

before and after analysis.

9.3.4 All samples and digestates must be held by the laboratory for a minimum of two weeks after the lab reports have been sent to the client.

9.3.5 For samples requiring digestion, refer to the Sample Preparation SOPs for procedures.

10 Procedure

10.1 Initial Set-up and Analysis for the TJA Trace ICP

10.1.1 Perform the daily and as needed maintenance.

10.1.1.1 Refill the rinse solution.

10.1.1.2 Replace the sample and rinse pump tubing.

10.1.1.3. Inspect all other autosampler tubing for clogs and/or visible leaks.

10.1.1.4 Inspect the nebulizer for clogs and position in the spray chamber.

10.1.1.5 Inspect the spray chamber for cleanliness and clean if needed.

10.1.1.6 Set the incoming Argon pressure to 70 psi.

10.1.1.7 Clean the torch and tip if needed.

10.1.1.8 Adjust the tension on the pump tubing in the peristaltic pump.

10.1.1.9 Check the vacuum gauge. (This must read below 30 millitorr. If the pressure rises above 30, the vacuum pump oil must be changed.)

10.1.1.10 Check to be sure the water cooler is on and is filled to the proper level.

10.1.1.11 Check to be sure the hood is operating.

10.1.1.12 Empty the waste containers.

10.1.1.13 Check the reagent water level in the argon humidifier and refill if necessary.

Method: 03-6010B

Revision: 12

Date: February 13, 2007

Page 29 of 47

- 10.1.2 Turn the computer on and using the arrow keys, move to Plasma Control Panel under Set-up. Press Enter.
- 10.1.3 Press F1, Start-up and then press F9, continue. After purging for 90 sec. the torch will automatically be ignited. After the torch lights, press F2 from the menu, and scroll down to the pump speed / rate, and type in 130. Press Enter and then F9.
- 10.1.4 Press Esc. Use the arrow keys to move to Operation. Press Enter. After the Enter Method Name prompt, type in OPTIMIZE. The instrument will load and set the parameters, such as nebulizer pressure and pump speed, specified in this method. Put the probe of the autosampler in the rinse solution.
- 10.1.5 Allow the instrument to become thermally stable before beginning. This requires at least 30 minutes of running while aspirating rinse solution.
- 10.1.6 Profile the instrument using the Working Profile Solution as described in Section 7.1. When an acceptable profile is reached, print the screen by pressing Control F2, Print screen.
- 10.1.7 Leaving the probe in the profile solution, press F1, Analyze and F1, Run. The method will run 10 replicates of the Profile solution and print the %RSD. When an acceptable %RSD of less than 0.5 is achieved, print by pressing Control F2, Print Screen. Press F9, Done/Keep. You will now need to escape to the main menu and go to Analysis under the Operation tab. Next to the method command prompt type in SCICPHSB. This is the existing method set up for 6010B analysis.

*(Note: If samples from the Dept. of Defense (DoD) are being analyzed, you will need to type **DoD** method name.)*

- 10.1.8 The following parameters shall also be checked while still in Methods. When everything has been set correctly, save any changes made to the method by pressing F9, Done/Keep. After checking the method settings, hit F3 (Method Info). Scroll down to the Analysis File Date, and change it to the current days date. (NOTE: a zero cannot be entered as the first digit.) If more than analysis run is performed on the same day, the sequence date must be followed by a sequential lettering system. (ex. 11306, 11306A, 11306B.....) After entering the analysis file date hit F9 (Keep/Done), then F8 (OPTIONS), then F2 (PrintMTD), then F9 (Done/Print), and finally F9 2x (Done/Keep).

Sample Introduction Device: Normal

Calibration Mode: Concentration

Method: 03-6010B
Revision: 12
Date: February 13, 2007
Page 30 of 47

Number of Repeats: 2 (3 Replicates are required for Dept. of Defense (DoD))
Flush Time: 65.0 sec
Auto-Store Analysis Data? Yes
Auto-Store Stdzn Data? Yes
Store Individual Repeats? No
Autoprint Analysis Data? Yes
Autoprint Stdzn Report: None
Condensed Print Format? No

Output Mode: Concentration
Override Print Limits? Yes
Override Sig. Figures? No
Apply Background Correction? Yes
Apply Blank Subtraction? No

Torch gas: High Flow
Auxiliary Gas Flow: Low (0.5 L/min)
Nebulizer Pressure: 25

Approximate RF Power: 950

Analysis Pump Rate: 150
Flush Pump Rate: 150
Relaxation Time (sec): 0
Pump Tubing Type: Tygon-Orange

- 10.1.9 After selecting, saving, and printing the required method, you will need to choose an autosampler table. Select OPERATION / Autosampler Setup from the main menu. Hit F8 (Filer), and select Autosampler Tables as a file type. You will need to scroll through the existing files to find SC6010B. Place the cursor on SC6010B and hit F8 (COPY). Type in an autosampler name which matches the analysis file date you entered in (Section 10.1.8). Press Enter.

(NOTE: If samples from the Dept. of Defense (DoD) are being analyzed, you will need to use the DoD10 autosampler table.)

Method: 03-6010B
Revision: 12
Date: February 13, 2007
Page 31 of 47

10.1.10 Use the arrow keys to move to Operation, Autosampler set-up to create a sequence table for the Autosampler. Press Enter, then F1Edit Set., then press F1, Edit Samples, and ALT F2. The autosampler table will already have default QC's and sample ID's. The default sample ID's will need to be changed to match the sample ID's on your horizon analysis batch. Immediately following the sample ID, the analysts' initials in parentheses need to be entered. (If the sample requires a dilution analysis, this dilution factor must be typed inside the parentheses after the analysts' initials.) Hit Enter 2X after entering the above information so that the cursor is in the third available field from the left. The horizon batch # off your analysis batch needs to be entered in this field. Immediately following this batch #, a T or D must be typed in. The T represents samples requiring a total metals analysis, and the D represents samples requiring a dissolved metals analysis. If this T or D is not entered properly, the automatic download of data in to horizon **will not** function. Build the table and save by pressing F9, Done/Keep three times. The computer will dictate the positions for the calibration blank and standards as well as the QCs that the analyst has put into the sequence table. Rinse and refill the calibration standards, blanks, and QC sample wells in the Autosampler and insert them in the correct positions. (NOTE: The correct autosampler positions can be viewed while in the autosampler edit mode)

10.1.11 Go into Operation off the main menu screen, and select Analysis. Enter the method name. Press Enter. Press F9, Autosampler. Enter the name of the Autosampler table to be used (Section 10.1.10). Press F1, Run. The instrument will, with the use of the Autosampler and peristaltic pump, start to standardize the instrument.

10.2 Analyze by the method of standard addition (Section 8.4) any samples containing matrix interferences which cannot be eliminated by dilution.

10.3 For the inter-element spectral interference correction factors to remain valid, the interferant concentration must not exceed its limit of linearity. Sample dilution is necessary in these cases, and the reporting limit must be raised to reflect the dilution performed.

11 Calculations

11.1 Sample results are reported directly from the readout of the instrument (from the calibration curve), and input into the LIMS. Appropriate prep factors are applied to the result at the time of supervisor approval.

Method: 03-6010B
Revision: 12
Date: February 13, 2007
Page 32 of 47

- 11.2 Any sample result requiring dilution to bring the sample into the linear range, is multiplied by the dilution factor before being entered into the LIMS using the following equation:

$$A = \frac{Z(B)}{C}$$

where A = concentration of element in sample
 Z = concentration of element in diluted sample
 B = final volume of dilution (mL)
 C = volume of sample aliquot used in dilution (mL)

12 Reporting Results

- 12.1 Report results in the Horizon LIMS system: The TJA Trace 61E ICP is set up to automatically download analysis results in to a system called NuGenesis. The NuGenesis system reads the raw data and compiles it into a form that the Horizon LIMS system will understand. The NuGenesis files can be accessed by logging onto horizon and selecting "Autopost pipe" under OPERATIONS. A box labeled Advanced Find will appear. You must enter the Queue "META", and enter the analysis batch # off your batch sheet. The sample IDs that will then appear in NuGenesis, will match the sample IDs that you typed in the autosampler table (Section 10.1.10) Therefore, if the sample ID was incorrectly entered in the autosampler table, it will be wrong in NuGenesis as well. Scroll down through the samples in NuGenesis, and make sure the results match your raw data. Once you have checked for errors, hit the AUTOPOST tab and select all.
- 12.2 Checking results in Horizon LIMS system: Once sample results have been autoposted from NuGenesis, you must enter the OPERATIONS tab and select POSTING-by-Worklist. Enter the HBN (Horizon Batch Number) from the analysis batch sheet. The analyst must go through this work list and double check to make sure that all results autoposted correctly.
- 12.3 Choosing sample Condition Codes in Horizon LIMS system: The column to the right of the sample name is CC, which stands for Condition Code. The CC will read OK if Horizon received data from NuGenesis. If any sample in the analysis run required an additional dilution, or some kind of rerun while analyzing, it will appear more than once in your analysis batch. If the results were left as is, the lab report would have duplicate results. Therefore, you must change the condition code on the sample so that you can determine which analytes are taken from which run. There are three CC's that can be used in this case. They are **LR**, **LX**, and **RP**. The **LR** condition code is to be used only if an analyte could not be completed in the current analysis run. (ex.: Ag did not pass in your calibration QC). The **LX** condition code is to be used if a sample was analyzed more than once in the same run, and all reruns have been completed. (ex.: A sample had a high RSD for Pb, and it was successfully reanalyzed later in the same run.) The **RP** condition code is to be used if the digestion QC associated with the sample did not pass

Method: 03-6010B
Revision: 12
Date: February 13, 2007
Page 33 of 47

method criteria, and needs to be sent back up to the prep Dept. to be redigested. (ex.: The LCS failed high for cadmium, and the sample in question required a cadmium analysis).

- 12.4 Choosing analyte Condition Codes in Horizon LIMS system: After one of the condition codes listed in Section 12.3 has been entered next to the sample, another condition code needs to be placed after the individual analytes in order for the analyst to be able to determine which analytes are used or not used. The three available condition codes are: **MO**, **NO**, and **RP**. The **MO** condition code is used for analyte results that you want to be taken from the current sample. The **NO** condition code is used for analytes that you do not want to be taken from the current sample. The **RP** condition code is used for analytes that need to be re-digested by the prep dept. (ex.: A sample is analyzed for cadmium, silver, and zinc. The cadmium result was fine, the silver result was no good because the digestion method blank failed for silver, and zinc was over your linear range and requires an additional dilution. In this case cadmium would be MO, silver would be RP, and zinc would be NO.)
- 12.5 All raw data used for reporting results must dated and initialed by the qualified laboratory personnel performing the first and second reviews. Use the Trace Run Case Narrative sheets located next to the PC to record this information.
- 12.6 If you need to hand enter results into Horizon due to problems with the automatic download, be sure not to round off results. Horizon will automatically perform rounding appropriate to the method. The actual result needs to be typed into horizon, even if it is less than the reporting limit. Any sample with a result less than the reporting limit will automatically be reported as ND (non-detect) by Horizon.

13 Waste Disposal

- 13.1 Refer to ALSI SOP 19-Waste Disposal.

14 Pollution Prevention

- 14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. Management shall consider pollution prevention a high priority. Extended storage of unused chemicals increases the risk of accidents. The laboratory shall consider smaller quantity purchases which will result in fewer unused chemicals being stored and reduce the potential for exposure by employees.

Method: 03-6010B

Revision: 12

Date: February 13, 2007

Page 34 of 47

ALSI tracks chemicals when received by recording their receipt in a traceable logbook. Each chemical is then labeled according to required procedures and stored in assigned locations for proper laboratory use.

15 Definitions

15.1 Refer to ALSI QA Plan under Laboratory Quality Control Checks for general definitions.

16 Troubleshooting

16.1 The ICP will often have carryover issues with silver that are confusing and difficult to remedy. Due to the chemical properties of silver, the analyte will build up inside the sample lines in the form of silver chloride. When a higher acid matrix is introduced to the system, the silver will sometimes fall back into solution, and show up as false positives. Usually the problem can be mediated by running some 20% HCL solution through the system for several minutes. Other times, the analyst will need to shut down, and clean the entire system. (NOTE: If the 20% HCL solution is run through the system for an extended period of time, the sample lines will develop a white haze. It is best to change out the lines when this occurs.)

16.2 Communication Errors or Power Outage. Often after power outages or periods of time when the computer has not been restarted for awhile, the Trace software will develop communication errors. If this occurs, the instrument and PC will need to be completely shut down and restarted. To shut down: extinguish the torch and exit out of TJA software, turn off the computer, turn off the one inch wide power strip located on the back of the instrument, turn off the red one inch wide switch on the front of the voltage regulator (small floor unit next to the wall.) Wait two to three minutes and then check to see if the vacuum pump is on by pressing the black start button located on the vacuum control system panel. Turn on the PC, voltage regulator, and instrument power switches. On the front of the ICP press the black RESET button located in the controller panel. Press the red RESET button on the back of the ICP. Then once again press the black RESET button on the controller panel. If the problems are still present, repeat this process. If this process is ineffective, technical support may need to be requested from Thermo Elemental.

16.3 Monitoring instrument trends for requesting service. By monitoring the peak intensities obtained from your As Optimization, the Optimization average recoveries, and the Hg profile throughout your analytical runs, you can often tell when service will soon be needed. The instruments RF tuning is checked annually during the PM Service; however, it is not uncommon for the tuning to require further adjustment throughout the year. By recording the information listed above in your analysis logbook, a trend will be more easily monitored. If a definite trend is detected, a service call to Thermo Elemental shall be placed. The reason for the service call must be documented in the maintenance

Method: 03-6010B

Revision: 12

Date: February 13, 2007

Page 35 of 47

logbook. Following the field service, an additional note shall be made that shows that the problem was corrected, and what was done to correct it.

- 16.4 Recording instrumentation problems in the maintenance logbook. Any daily service or issue involving the instrument must be noted in the maintenance logbook. The logbook is then a good source to refer to if problems develop. Usually the current issue with the instrument has occurred sometime in the past. The analyst can then look to see what if any corrective actions were performed.

Method: 03-6010B
Revision: 12
Date: February 13, 2007
Page 36 of 47

APPENDIX A

Instrument Method: _____

Sequence #: _____

Date Started: _____

Analyst: _____

* All Standards are prepared daily prior to analysis of samples in acid matrix listed.

PDS: 3.5 ml sample + 3.5 ml High Std

Trace ICP Analytical Worksheet

Acid Matrix: ____% HNO₃ + ____% HCl

*High Std: ____ ml each of _____ + _____ into ____ ml Pipette ID # _____

*Mid Std: ____ ml High Std into ____ ml ICS: _____

*Low Std: ____ ml High Std into ____ ml RPL: ____ ml into ____ ml

*CCV/IPC: ____ ml each of _____ + _____ into ____ ml

*Second Source: ____ ml of _____ + _____ into ____ ml Pipette ID # _____

____ ml each of _____ + _____ into ____ ml

*Additional Second Source: ____ ml of _____ + _____ into ____ ml

*Cal Blank: ____ ml HNO₃ Lot #: _____ HCl Lot #: _____ Pipette ID # _____

BLANK		
LOW STD		
MID STD		71
HIGH STD		72
SECOND SOURCE	31	73
	32	74
	33	75
	34	76
	35	77
	36	78
	37	79
	38	80
	39	
	40	
		81
		82
	41	83
	42	84
1	43	85
2	44	86
3	45	87
4	46	88
5	47	89
6	48	90
7	49	
8	50	
9		
10		
		91
		92
	51	93
	52	94
11	53	95
12	54	96
13	55	97
14	56	98
15	57	99
16	58	100
17	59	
18	60	
19		
20		
		101
		102
	61	103
	62	104
21	63	105
22	64	106
23	65	107

Method: 03-6010B
Revision: 12
Date: February 13, 2007
Page 37 of 47

TABLE 1
(Element Wavelengths)

<u>Element</u>	<u>Wavelength</u>
Ag	328.068
Al	308.215
As	189.042
B	249.678
Ba	493.409
Be	313.042
Bi	223.061
Ca	317.933
Cd	226.502
Co	228.616
Cr	267.716
Cu	324.753
Fe	271.441
Mg	202.030
Mo	202.030
Mn	257.610
Ni	231.604
Pb	220.353
Sb	206.838
Se	361.384
Sn	189.989
Sr	421.552
Ti	334.941
Tl	190.864
V	292.402
Zn	213.856

Method: 03-6010B
Revision: 12
Date: February 13, 2007
Page 38 of 47

TABLE 2

(Reporting Limit Concentrations)

<u>Element</u>	<u>Concentration</u>
Ag	0.004
Al	0.10
As	0.010
B	0.10
Ba	0.010
Be	0.004
Bi	0.05
Ca	0.10
Cd	0.002
Co	0.005
Cr	0.005
Cu	0.01
Fe	0.06
Mg	0.10
Mo	0.02
Mn	0.005
Ni	0.02
Pb	0.005
Sb	0.02
Se	0.02
Sn	0.02
Sr	0.005
Ti	0.02
Tl	0.02
V	0.005
Zn	0.02

Method: 03-6010B
Revision: 12
Date: February 13, 2007
Page 39 of 47

TABLE 3
(Laboratory Fortified Blank and Matrix Spike Concentrations)

<u>Element</u>	<u>Concentration (mg/L)</u>
Ag	0.100
Al	1.00
As	0.100
B	1.00
Ba	1.00
Be	0.200
Bi	1.00
Ca	1.00
Cd	0.100
Co	1.00
Cr	0.100
Cu	1.00
Fe	1.00
Mg	0.100
Mo	1.00
Mn	0.100
Ni	1.00
Pb	0.100
Sb	0.20
Se	1.00
Sn	0.10
Sr	0.10
Ti	0.10
Tl	0.10
V	0.050
Zn	0.50

Method: 03-6010B
Revision: 12
Date: February 13, 2007
Page 40 of 47

TABLE 4
(Interference Check Solution Concentrations)

<u>Element</u>	<u>Concentration</u>
Ag	0.75
Al	30.0
As	2.50
Ba	0.75
Be	0.25
Ca	150
Cd	0.75
Co	0.75
Cr	0.75
Cu	0.75
Fe	125
Mg	75.0
Mn	0.50
Ni	0.75
Pb	2.50
Se	1.25
Tl	2.50
V	0.75
Zn	0.75

Method: 03-6010B
Revision: 12
Date: February 13, 2007
Page 41 of 47

TABLE 5
(Matrix Spike / Matrix Spike Duplicate Concentrations for TCLP and SPLP leachates)

<u>Element</u>	<u>Concentration</u>
Ag	0.90
As	0.90
Ba	9.00
Cd	0.90
Cr	4.50
Pb	4.50
Se	4.50

Method: 03-6010B
Revision: 12
Date: February 13, 2007
Page 42 of 47

SOP Change History Sheet

<u>Section No.</u>	<u>Section</u>	<u>Reason for Change</u>
<i>Revision 10: 02/24/2005</i>		
2	Summary of Method	U.S. Army Corp. audit response
6.1	Reagents	SOP Update
6.2	Reagents	SOP Update
6.5.1-6.5.2	Reagents	SOP Update
6.5.10	Reagents	SOP Update
6.6.4	Reagents	U.S. Army Corp. audit response
6.6.5	Reagents	SOP Update
6.6.6	Reagents	SOP Update
6.6.7	Reagents	SOP Update
6.6.8	Reagents	SOP Update
6.8-6.10	Reagents	SOP Update
6.9-6.11	Reagents	SOP Update
7.3	Instrument Calibration	SOP Update
7.4	Instrument Calibration	SOP Update
8.1.1	Quality Control	U.S. Army Corp. audit response
8.1.3	Quality Control	SOP Update
8.1.5	Quality Control	SOP Update
8.1.6	Quality Control	SOP Update
8.1.7	Quality Control	SOP Update
8.2.2	Quality Control	U.S. Army Corp. audit response
8.2.2.1	Quality Control	SOP Update

Method: 03-6010B
Revision: 12
Date: February 13, 2007
Page 43 of 47

SOP Change History Sheet (continued)

<u>Section No.</u>	<u>Section</u>	<u>Reason for Change</u>
8.3.1	Quality Control	SOP Update
8.3.4	Quality Control	SOP Update
8.7	Quality Control	SOP Update
8.12	Quality Control	Section Removed
9.3.2-9.3.4	Sample Handling	SOP Update
10.1.8	Procedure	U.S. Army Corp. audit response
12.1	Reporting Results	SOP Update
12.2 / 12.3	Reporting Results	Sections Removed
	Appendix A (Analytical Worksheet)	U.S. Army Corp. audit response
	Table 2 (RPL Concentrations)	SOP Update
	Table 3 (LCS / MS Concentrations)	SOP Update
	Table 4 (Interference Check Concentrations)	SOP Update

Revision 11: 01/26/2006

1.1	Scope and Application	Matrices update
1.7	Scope and Application	Project requirements note
2	Summary of Method	DoD audit response
3.1.3	Interferences	Frequency change instituted for correction factors
3.4	Interferences	SOP update from Method 4.2
4.2	Safety	Directive to read MSDS sheets and their availability
5.2 & 5.4	Apparatus and Materials	SOP materials update

SOP Change History Sheet (continued)

Method: 03-6010B
Revision: 12
Date: February 13, 2007
Page 44 of 47

<u>Section No.</u>	<u>Section</u>	<u>Reason for Change</u>
6.1 & 6.2	Reagents	Expiration date and labeling
6.5.8	Reagents	Addition of "Solution A"
6.5.9	Reagents	Addition of "Solution B"
6.6.1	Reagents	Reagent source change, documentation and hold time additions
6.6.2	Reagents	Documentation and hold time additions
6.6.3	Reagents	Documentation and hold time additions
6.6.4	Reagents	DoD audit response
6.6.5	Reagents	Documentation statement
6.6.6	Reagents	Documentation and hold time additions
6.6.7	Reagents	Preparation revision
6.6.8	Reagent	Preparation revision
6.9	Reagents	Preparation revision
6.10	Reagents	Volume & concentration change
6.34	Reagents	Section added
7.1.1	Instrument Calibration	Scandium replaces arsenic
7.1.2	Instrument Calibration	Profile line change
7.1.3	Instrument Calibration	Scandium replaces arsenic
7.1.4	Instrument Calibration	DoD audit response
7.1.5	Instrument Calibration	DoD audit response
7.3	Instrument Calibration	DoD audit response
8.1.1	Quality Control	DoD audit response

SOP Change History Sheet (continued)

Method: 03-6010B
Revision: 12
Date: February 13, 2007
Page 45 of 47

<u>Section No.</u>	<u>Section</u>	<u>Reason for Change</u>
8.1.2	Quality Control	DoD audit response
8.1.3	Quality Control	Reference to QA Plan
8.1.4	Quality Control	Instructions added for IECs
8.1.8	Quality Control	DoD audit response
8.2.3	Quality Control	LCS replaces LFB
8.2.3.1	Quality Control	Change in Stock Solution
8.2.3.2	Quality Control	Section Removed
8.2.2	Quality Control	DoD audit response
8.2.6	Quality Control	DoD audit response
8.3.1	Quality Control	DoD audit response
8.3.2	Quality Control	DoD audit response
	Quality Control Requirements (Calibration Blanks)	DoD audit response
	Quality Control Requirements (RPL)	DoD Audit Response
	Quality Control Requirements (Matrix Spike)	SOP Update
	Quality Control Requirements (Sample Replicates)	SOP Update
8.6	Quality Control	Scandium intensity adjusted from $\pm 20\%$ to $\pm 30\%$
10	Procedure (throughout section)	Additional instrument directions
12	Reporting Results	Major revisions throughout
16	Troubleshooting	DoD audit response

Method: 03-6010B

Revision: 12

Date: February 13, 2007

Page 46 of 47

SOP Change History Sheet (continued)

<u>Section No.</u>	<u>Section</u>	<u>Reason for Change</u>
<u>Revision 12: 02/13/2007</u>		
1.6	Scope and Application	Updated MDL file location/maintenance
4.2	Safety	Updated MSDS file locations
6.4	Reagents	Updated vendor information
6.8	Reagents	Revised Working Internal Standard Solution concentration
6.10	Reagents	Revised volumes of single element solution for some elements
8.11	Quality Control	Deleted terminology “reprofile and/or” from corrective action for High Cal Stnd, QCS, and ICP; reworded RPL corrective action and revised control limits; revised Scandium Internal Standard corrective action for greater detail; revised Internal Check Solution frequency
2	Table	Revised some of the element reporting limit concentrations

Method: 03-6010B
Revision: 12
Date: February 13, 2007
Page 47 of 47

**SOP Concurrence Form
for the Distribution and Revision of Standard Operating Procedures**

I have read, understood, and concurred with the Standard Operating Procedure (SOP) described above and will perform this procedure as it is written in the SOP.

Print Name	Signature	Date
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____

UNCONTROLLED DOCUMENT: DO NOT Transfer or Print

Method: 04-ANION2

Revision: 7

Date: March 13, 2006

Page: 1 of 30

Document Title: Determination of Inorganic Anions by Ion Chromatography

Document Control Number: _____

Organization Title: ANALYTICAL LABORATORY SERVICES, INC. (ALSI)

Address: 34 Dogwood Lane
Middletown, PA 17057

Phone: (717) 944-5541

Approved by:

Helen MacMinn,
Quality Assurance Manager

Date

Jason Badman,
Wet Chemistry Supervisor

Date

Heather McCall,
Validator

Date

UNCONTROLLED DOCUMENT: DO NOT Transfer or Print

Method: 04-ANION2

Revision: 7

Date: March 13, 2006

Page: 2 of 30

TABLE OF CONTENTS

1	Scope and Application.....	3
2	Summary of Method.....	3
3	Interferences	4
4	Safety	5
5	Apparatus and Materials.....	5
6	Reagents.....	6
7	Instrument Calibration	12
8	Quality Control.....	14
9	Sample Collection, Preservation and Handling	17
10	Procedure	18
11	Calculations	23
12	Reporting Results.....	23
13	Waste Disposal	24
14	Pollution Prevention	25
15	Definitions	25
16	Troubleshooting.....	26
	SOP Change Summary	27
	SOP Concurrence Form.....	30

1 Scope and Application

- 1.1 This document states the laboratory's policies and procedures established in order to meet the requirements of all certifications/accreditations currently held by the laboratory, including the most current NELAC standards.
- 1.2 This method is adapted from EPA Method 300.0, Rev 2.1 Aug. 1992. Determination of Inorganic Anions by Ion Chromatography and SW-846 9056, Rev. 0 Sept. 1994, Determination of Inorganic Anions by Ion Chromatography.
- 1.3 This method is restricted for use by or under the supervision of analysts trained on the use of the ion chromatograph.
- 1.4 This method covers the determination of inorganic anions including fluoride, chloride, nitrite, bromide, nitrate, and sulfate. The applicable matrices are drinking water, surface water, mixed domestic and industrial wastewaters, groundwater, reagent waters, solids (after extraction, Section 11), and leachates (when no acetic acid is used).
- 1.5 Method Detection Limits can be found in the current Wet Chemistry method detection limit book. The detection limits for a specific sample may differ from those listed due to the nature of interferences in a particular sample matrix.
- 1.6 Individual project requirements may override criteria listed in this SOP.

2 Summary of Method

- 2.1 A small volume of sample, typically 5 mL, is introduced into an ion chromatograph. The anions of interest are separated and measured, using a system comprised of a guard column, analytical column, suppressor device, and conductivity detector.
- 2.2 An extraction procedure must be performed to use this method for solids (See Section 10.4).
- 2.3 When this method is used to analyze unfamiliar samples for any of the above anions, anion identification shall be supported by the use of a fortified sample matrix covering the anions of interest. The fortification procedure is described in Section 6.23 and 6.25.
- 2.4 Limited performance-based method modifications may be acceptable provided they are fully documented and meet or exceed requirements expressed in Section

8, Quality Control.

3 Interferences

- 3.1 Interferences can be caused by substances with retention times that are similar to and overlap those of the anion of interest. Large amounts of an anion can interfere with the peak resolution of an adjacent anion. Sample dilution and/or fortification can be used to solve most interference problems associated with retention times.
- 3.2 The water dip or negative peak that elutes near, and can interfere with, the fluoride peak can usually be eliminated by the addition of an equivalent amount of concentrated eluent to each standard and sample, or by using working eluent solution (Section 6.12) to make dilutions or standards.
- 3.3 Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baseline in ion chromatograms.
- 3.4 Samples that contain particles larger than 0.45 microns and reagent solutions that contain particles larger than 0.20 microns require filtration to prevent damage to instrument columns and flow systems.
- 3.5 Any anion that is not retained by the column or only slightly retained will elute in the area of fluoride and interfere. Known coelution is caused by carbonate and other small organic anions. At concentrations of fluoride above 1.5 mg/L, this interference may not be significant. However, it is the responsibility of the user to generate precision and accuracy information in each sample matrix.
- 3.6 The acetate anion elutes early during the chromatographic run. The retention times of the anions also seem to differ when large amounts of acetate are present. Therefore, this method is not recommended for leachates of solid samples when acetic acid is used for pH adjustment.
- 3.7 The quantitation of unretained peaks shall be avoided, such as low molecular weight organic acids (formate, acetate, propionate etc.) that are conductive and coelute with or near fluoride and would bias the fluoride quantitation in some drinking and most waste waters.

4 Safety

Method: 04-ANION2

Revision: 7

Date: March 13, 2006

Page: 5 of 30

- 4.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical shall be regarded as a potential health hazard and exposure shall be as low as reasonably achievable.
- 4.2 ALSI maintains material safety data sheets (MSDSs) on all chemicals used in this procedure. MSDSs are available to all staff and are located in the QA office. Each analyst shall become familiar with the reagents used by referencing the appropriate (MSDSs). In doing so, the analyst will become familiar with the appropriate precautions for each reagent.
- 4.3 Analysts must wear a buttoned lab coat and safety glasses at all times during the analysis. PVC gloves shall be worn when handling samples and reagents.

5 Apparatus and Materials

- 5.1 Balance - Analytical, capable of accurately weighing to the nearest 0.0001 g. A Mettler AE163 is currently in use or equivalent.
- 5.2 Ion chromatograph - Dionex DX-120 and DX-500, including a guard column (AG-14), separator column (AS-14), suppressor, and conductivity detector.
- 5.3 The Dionex PeakNet Chromatography Software version 5.11.
- 5.4 Autosampler – Dionex AS40
- 5.5 Glassware - Class A volumetric flasks and pipets as required.
- 5.6 150 mL glass beakers purchased from VWR catalog #13912-502 or equivalent.
- 5.7 Automatic pipette capable of dispensing 0.200 mL to 1.00 mL. An Eppendorf variable volume 1000 purchased from VWR Catalog #53511-582 or equivalent.
- 5.8 Automatic pipette capable of dispensing 0.020 mL to 0.200 mL. An Eppendorf variable volume 200 purchased from VWR Catalog #53513-408 or equivalent.
- 5.9 Automatic pipette capable of dispensing 2.0 mL to 10.0 mL. A Finnpiquette variable volume 10 mL purchased from VWR Catalog #53515-050 or equivalent.
- 5.10 Stir plate purchased from VWR Catalog #33994-348 or equivalent.
- 5.11 Stir bars purchased from VWR Catalog #58948-230 or equivalent.

Method: 04-ANION2

Revision: 7

Date: March 13, 2006

Page: 6 of 30

- 5.12 0.45-micron nylon syringe filters purchased from VWR Catalog #28138-194 or equivalent.
- 5.13 Polyvial and Filter Caps, 5 mL purchased from Dionex VWR Catalog #38141 or equivalent.
- 5.14 Clear 40-mL vial purchased from Industrial Glassware, Catalog #2795FL-PC or equivalent.

6 Reagents

NOTE: Verification that the chemical or reagent purchased is of the correct purity and traceability before being put into use is the responsibility of the supervisor of the department in which the reagent will be used. The preparation of working reagents is recorded in bound logbooks. These logbooks document the name of the reagent, reference ID number, and the concentration of the reagent, the reference number(s) of the stock reagent(s) used as well as the dilutions performed, date of preparation, date of expiration, and initials of the preparer. The container holding the working reagent is labeled with the reference ID number, initials of the preparer, the date of the preparation, and the expiration date as determined by the method. Any health and/or safety concerns are also listed on the container.

- 6.1 Reagent water: Distilled or deionized water, free of the anions of interest. Water shall contain particles no larger than 0.20 microns. For this purpose, ALSI uses a deionizer that provides analyte-free, greater than 16 megohm-cm, DI water on demand.
- 6.2 Sodium Carbonate (Na_2CO_3) - ACS Reagent Grade. Purchase Aldrich 22, 348-4 (500gm) or equivalent. This reagent carries a five (5) year shelf life.
- 6.3 Sodium Bicarbonate (NaHCO_3) - ACS Reagent Grade. Purchase Aldrich 43, 144-3 (250gm) or equivalent. This reagent carries a five (5) year shelf life.
- 6.4 Sodium Bromide (NaBr) - ACS Reagent Grade. Purchase Aldrich 22, 988-1 (10gm) or equivalent. This reagent carries a five (5) year shelf life.
- 6.5 Sodium Chloride (NaCl) - ACS Reagent Grade. Purchase Aldrich 20, 443-9 (100gm) or equivalent. This reagent carries a five (5) year shelf life.
- 6.6 Sodium Fluoride (NaF) - ACS Reagent Grade. Purchase Aldrich 21, 530-9 (50gm)

UNCONTROLLED DOCUMENT: DO NOT Transfer or Print

Method: 04-ANION2

Revision: 7

Date: March 13, 2006

Page: 7 of 30

or equivalent. This reagent carries a five (5) year shelf life.

- 6.7 Sodium Nitrate (NaNO_3) - ACS Reagent Grade. Purchase Aldrich 46, 777-4 (250gm) or equivalent. This reagent carries a five (5) year shelf life.
- 6.8 Sodium Nitrite (NaNO_2) - ACS Reagent Grade. Purchase Aldrich 43, 160-5 (250gm) or equivalent. This reagent carries a five (5) year shelf life.
- 6.9 Potassium Sulfate (K_2SO_4) - ACS Reagent Grade. Purchase Aldrich 22, 132-5 (500gm) or equivalent. This reagent carries a five (5) year shelf life.
- 6.10 0.5 M Sodium Carbonate Eluent Concentrate Solution: Dissolve 52.98g of sodium carbonate (Na_2CO_3) (Section 6.2) in reagent water, dilute to 1000mL in a volumetric flask and label appropriately. This solution is stable for 1 month at room temperature.
- 6.11 0.5 M Sodium Bicarbonate Eluent Concentrate Solution: Dissolve 21.00g of sodium bicarbonate (NaHCO_3) (Section 6.3) in reagent water, dilute to 500mL in a volumetric flask and label appropriately. This solution is stable for 1 month at room temperature.
- 6.12 Working Eluent Solution: 3.5mM Sodium Carbonate/1.0 mM Sodium Bicarbonate. Pipet 28.0 mL of the 0.5 M (Na_2CO_3) (Section 6.2) plus 8.0 mL of the 0.5 M NaHCO_3 (Section 6.3) into a 2 liter volumetric flask. Dilute to volume with reagent water and transfer over to a 4-liter side-arm flask. Add 2 additional liters of reagent water to the side-arm flask. Add a spin bar to the 4-liter flask and place on a magnetic stirrer. Degas the solution by pulling a vacuum on the solution for 20 to 30 minutes (until all air bubbles are gone) and label appropriately. This solution expires after 1 month at room temperature.
- 6.13 100X Eluent: Measure 350mL of 0.5 M (Na_2CO_3) concentrate (Section 6.10) and 100mL of 0.5 M NaHCO_3 concentrate (Section 6.11) into a 500mL volumetric flask, dilute to volume with reagent water and label appropriately. This solution is stable for one month at room temperature.
- 6.14 Stock Calibration Standard Solutions, 1000 mg/l: Purchased from Inorganic Ventures. Calibration stock solution "A" containing bromide, fluoride, nitrate and nitrite is catalog #ALSI-ICAL 3, or equivalent Calibrating stock solution "B" containing chloride and sulfate is catalog #NHL-ICAL-2, or equivalent. These stock solutions shall be stored above the freezing point of water up to 6°C and discarded according to the manufacturer's expiration date. If purchased standards are unavailable, the stock calibration standards can be prepared from ACS reagent

Method: 04-ANION2

Revision: 7

Date: March 13, 2006

Page: 8 of 30

grade materials (dried at 104 degrees C for 60 min), except for the nitrite which shall be dried in a dessicator for 24 hours prior to use. Prepared stock standards are stable for at least 1 month when stored above the freezing point of water up to 6°C. Dissolve the following amounts of each chemical in reagent grade water and dilute to 500mL. Calibration stock solution 'A' contains bromide, fluoride, nitrate, and nitrite. Calibration stock solution 'B' contains chloride and sulfate.

- Sodium Bromide - 0.6438 grams
- Sodium Chloride - 0.8243 grams
- Sodium Fluoride - 1.105 grams
- Sodium Nitrate - 3.0340 grams
- Sodium Nitrite - 2.4629 grams
- Potassium Sulfate - 0.9071 grams

6.15 Working Calibration Standard Solutions: Working calibration standards are prepared by diluting the stock calibration standard (Section 6.14) to volume with working eluent solution (Section 6.12) and labeling appropriately. Working calibration standard solutions must be prepared fresh monthly and stored at room temperature.

Calibration Levels

Calibration Stock Solution	A (F1, NO2, Br, NO3) mL added	B (Cl, SO4) mL added	Final volume (mL)	Final [A] (mg/L)	Final [B] (mg/L)
1	0.10	1.00	1000	0.10	1.00
2	0.25	2.50	1000	0.25	2.50
3	0.50	5.00	1000	0.50	5.00
4	0.10	1.00	100	1.00	10.0
5	0.15	1.50	100	1.50	15.0
6	0.20	2.00	100	2.00	20.0
7	0.25	2.50	100	2.50	25.0
8	0.50	5.00	100	5.00	50.0
9	1.00	10.0	100	10.0	100

6.16 Fortification/Spike Solution – Purchased from Inorganic Ventures. “Method A” fortification/spike solution is catalog #ALSI-ICAL-1A, or equivalent. “Method L” fortification/spike solution is catalog #ALSI-ICAL-2A, or equivalent. These solutions shall be stored above the freezing point of water up to 6°C and discarded according to the manufacturer’s expiration date. If purchased standards are unavailable, the fortification/spike solution can be prepared from ACS reagent

Method: 04-ANION2

Revision: 7

Date: March 13, 2006

Page: 9 of 30

grade materials (dried at 104 degrees C for 60 min), except for the nitrite which shall be dried in a dessicator for 24 hours prior to use. Prepared solutions are stable for 1 month when stored above the freezing point of water up to 6°C. Dissolve the following amounts of each chemical in reagent grade water, dilute to 500mL and label appropriately. All reagents shall be dissolved in the same 500mL of water for a combined analyte solution.

6.16.1 Method "A" Fortification/Spike Solution

Analyte	Grams/500mL	Final Concentration, mg/l
Sodium bromide	0.0644	100
Sodium chloride	3.297	4000
Sodium fluoride	0.1105	100
Sodium nitrate	1.5170	500
Sodium nitrite	0.2463	100
Potassium sulfate	3.6284	4000

6.16.2 Method "L" Fortification/Spike Solution

Analyte	Grams/500mL	Final Concentration, mg/l
Sodium bromide	0.0644	100
Sodium chloride	0.4121	500
Sodium fluoride	0.1105	100
Sodium nitrate	0.3034	100
Sodium nitrite	0.2463	100
Potassium sulfate	0.4536	500

6.17 Continuing Calibration Verification Standard Method A Level 1 (CCV1A):
Dilute 0.500 mL of Method A Fortification/Spike Solution (Section 6.16.1) to volume with Working Eluent (Section 6.12) in a 100 mL class A volumetric flask and label appropriately. This solution is stable for 24 hours and stored at room temperature. This solution contains anions at the following concentrations: F- 0.50 mg/L; Cl – 20.0 mg/L; NO₂ 0.50 mg/L; Br – 0.50 mg/L; NO₃ – 2.50 mg/L; and SO₄ – 20.0 mg/L. (This solution satisfies the Instrument Performance Check Solution (IPC) requirement in Method 300.0 Section 9.3.4.)

6.18 Continuing Calibration Verification Standard Method A Level 2 (CCV2A):
Dilute 1.00 mL of Method A Fortification/Spike Solution (Section 6.16.1) to volume with Working Eluent (Section 6.12) in a 100 mL class A volumetric

UNCONTROLLED DOCUMENT: DO NOT Transfer or Print

Method: 04-ANION2

Revision: 7

Date: March 13, 2006

Page: 10 of 30

flask and label appropriately. This solution is stable for 24 hours and stored at room temperature. This solution contains anions at the following concentrations: F- 1.00 mg/L; Cl – 40.0 mg/L; NO₂ – 1.00 mg/L; Br – 1.00 mg/L; NO₃ – 5.00 mg/L; and SO₄ – 40.0 mg/L. (This solution satisfies the Instrument Performance Check Solution (IPC) requirement in Method 300.0 Section 9.3.4.)

6.19 Continuing Calibration Verification Standard Method L Level 1 (CCV1L): Dilute 0.500 mL of Method L Fortification/Spike Solution (Section 6.16.2) to volume with Working Eluent (Section 6.12) in a 100 mL class A volumetric flask and label appropriately. This solution is stable for 24 hours and stored at room temperature. This solution contains anions at the following concentration: F-0.50 mg/L; Cl – 2.50 mg/L; NO₂ 0.50 mg/L; Br – 0.50 mg/L; NO₃ – 0.50 mg/L; and SO₄ – 2.50 mg/L. (This solution satisfies the Instrument Performance Check Solution (IPC) requirement in Method 300.0 Section 9.3.4.)

6.20 Continuing Calibration Verification Standard Method L Level 2 (CCV2L): Dilute 1.00 mL of Method L Fortification/Spike Solution (Section 6.16.2) to volume with Working Eluent (Section 6.12) in a 100 mL class A volumetric flask and label appropriately. This solution is stable for 24 hours and stored at room temperature. This solution contains anions at the following concentrations: F – 1.00 mg/L; Cl – 5.00 mg/L; NO₂ – 1.00 mg/L; Br – 1.00 mg/L; NO₃ – 1.00 mg/L; and SO₄ – 5.00 mg/L. (This solution satisfies the Instrument Performance Check Solution (IPC) requirement in Method 300.0 Section 9.3.4.)

6.21 Second Source Standard Method A (SSA): This standard must be prepared from an NIST traceable material other than the calibration standards. A suitable second source standard is prepared from AccuStandard Stock Solutions by diluting the stock solutions in Working Eluent (Section 6.12) as follows. All anions shall be diluted in a single solution and be labeled with a seven (7) day expiration time and stored at room temperature. (This solution satisfies the Laboratory Fortified Blank (LFB) requirement in Method 300.0 Section 9.3.2.)

(See Second Source Standard Method A Table for Section 6.21 on page 11)

Standard	Catalog No.	Concentration (mg/L)	Volume (mL) Diluted to 100 mL	Concentration (mg/L) In Final Solution
Fluoride	IC-F-10X-1	1000	0.050	0.50

UNCONTROLLED DOCUMENT: DO NOT Transfer or Print

Method: 04-ANION2

Revision: 7

Date: March 13, 2006

Page: 11 of 30

Chloride	IC-CL-10X-1	1000	2.00	20.0
Nitrite	IC-NO ₂ -N-10X-1	1000	0.050	0.50
Bromide	IC-BR-10X-1	1000	0.050	0.50
Nitrate	IC-NO ₃ -N-10X-1	1000	0.25	2.50
Sulfate	IC-SO ₄ -10X-1	1000	2.00	20.0

- 6.22 Second Source Standard Method L (SSL): This standard must be prepared from an NIST traceable material other than the calibration standards. A suitable second source standard is prepared from AccuStandard stock solutions by diluting the stock solutions in Working Eluent (Section 6.12) as follows. All anions shall be diluted in a single solution and be labeled with a seven (7) day expiration time and stored at room temperature. (This satisfies the Laboratory Fortified Blank requirement in Method 300.0 Section 9.3.2.)
- and (LFB)

Standard	Catalog No.	Concentration (mg/L)	Volume (mL) Diluted to 100 mL	Concentration (mg/L) In Final Solution
Fluoride	IC-F-10X-1	1000	0.050	0.50
Chloride	IC-CL-10X-1	1000	0.250	2.50
Nitrite	IC-NO ₂ -N-10X-1	1000	0.050	0.50
Bromide	IC-BR-10X-1	1000	0.050	0.50
Nitrate	IC-NO ₃ -N-10X-1	1000	0.050	0.50
Sulfate	IC-SO ₄ -10X-1	1000	0.250	2.50

- 6.23 Matrix Spike: Pipette 0.025 mL of Method A Fortification/Spike Solution (Section 6.16.1) into 5.0 mL of sample. The spike concentrations are as follows: F - 0.50 mg/L; Cl - 20.0 mg/L; NO₂ - 0.50 mg/L; Br - 0.50 mg/L; NO₃ - 2.50 mg/L; and SO₄ - 20.0 mg/L.
- 6.24 300S Laboratory Control Sample (LCS): Pipet 1.00 mL of Method A. Fortification/Spike Solution (Section 6.16.1) into 10.0 grams of laboratory sand. The LCS concentrations are as follows: F - 10 mg/kg; Cl - 400 mg/kg; NO₂ - 10 mg/kg; Br - 10 mg/kg; NO₃ - 50 mg/kg; and SO₄ - 400 mg/kg.
- 6.25 300S Matrix Spike: Pipette 1.00 mL of Method A Fortification/Spike Solution (Section 6.16.1) into 10.0 grams of sample. The spike concentrations are as follows: F - 10 mg/kg; Cl - 400 mg/kg; NO₂ - 10 mg/kg; BR - 10 mg/kg; NO₃ - 50 mg/kg; and SO₄ - 400 mg/kg.
- 6.26 Laboratory Sand - Purchased from VWR Catalog #20118-003 or equivalent and labeled with a five (5) year shelf life and stored at room temperature.

7 Instrument Calibration

- 7.1 Prepare the series of calibration standards listed in Section 6.15. All standard dilutions must be recorded in the standards logbook located in the Wet Chemistry area.
- 7.2 Two ranges of calibration curves shall be prepared from these calibration standards. The first range shall be identified as Method A XXYY.met (XX-Month; YY-Year). This calibration covers the range where analytes of interest are most commonly quantitated and is used for a majority of samples.

The second range shall be identified as Method L XXYY.met. This calibration has lower reporting limits for some analytes and is commonly used for samples that require dilutions because of a matrix problem or for clients who require a lower reporting limit than is associated with the Method A calibration.

Either calibration can in fact be used for a sample as long as the analyte concentration falls within the calibration range and all appropriate quality control samples have been analyzed. The calibration ranges are listed below in mg/L.

	Method L	Method A
Fluoride	0.10 – 2.00	0.10 – 2.00
Chloride	1.00 – 10.0	10.0 – 100
Nitrite	0.10 – 2.00	0.10 – 2.00
Bromide	0.10 - 2.00	0.10 – 2.00
Nitrate	0.10 – 1.50	1.50 – 10.0
Sulfate	1.00 – 10.00	10.0 - 100

- 7.3 In the PeakNet schedule, identify the standards as Level 1 thru Level 9 calibration standards and analyze them using Method A XXYY.
- 7.4 Following analysis, access Method A XXYY in the PeakNet Method Editor section. Access the component table. Detail each analyte and select the standards representing the calibration range for that analyte. (For example: Fluoride would use Standard Level 1 (0.10) thru Level 6 (2.00). All standards not contained in this range must be flagged with “U” for underrange or “O” for Overrange.
- 7.5 Once this is complete, the appropriate calibration techniques must be selected for the analytes. The linear regression technique shall be used for all analytes. Detail

UNCONTROLLED DOCUMENT: DO NOT Transfer or Print

Method: 04-ANION2

Revision: 7

Date: March 13, 2006

Page: 13 of 30

each analyte and select the curve type linear. The correlation coefficient for each analyte shall be ≥ 0.995 . The origin (blank) shall be included for all Method L curves and for Method A curved for fluoride, nitrate, and bromide. The origin (blank) shall be ignored for Method A curves for chloride, nitrate, and sulfate. If the correlation coefficient does not meet criteria, the calibration must be reanalyzed.

- 7.6 Rebatch the calibration standards using Method L XXYY, and repeat Sections 7.4 and 7.5 for Method L XXYY.
- 7.7 Print a copy of the calibration curves to include with the raw data from the calibration standards.
- 7.8 All initial calibrations must be verified with a second source standard SSA (Section 6.21) or SSL (Section 6.22), prior to the analysis of samples. All standard recoveries must be 90-110% of their actual value for the calibration to be acceptable. If the second source standard recoveries do not meet this criteria, remake the standard once, then the calibration shall be reanalyzed.
- 7.9 If the calibration blank concentration is greater than or equal to the reporting limit AND is greater than 1/10 the sample concentration, the source of contamination must be investigated and measures taken to minimize or eliminate the problem and affected samples reanalyzed. If reanalysis is not possible, data shall be reported with a qualifying statement.

For DoD Samples: If the calibration blank concentration is greater than or equal to $\frac{1}{2}$ the reporting limit AND is greater than 1/10 the sample concentration, the source of contamination must be investigated and measures taken to minimize or eliminate the problem and affected samples reanalyzed. If reanalysis is not possible, data shall be reported with a qualifying statement.

- 7.10 When an initial calibration is not performed on the day of analysis, the validity of the initial calibration shall be verified prior to sample analysis by the analysis of continuing calibration verification standard (Section 6.17, 6.18, 6.19, or 6.20).
 - 7.10.1 The continuing calibration verification standard is used to confirm the validity of the initial calibration. It is not used for sample quantitation. Only the initial calibration is used to quantitate samples.
 - 7.10.2 A continuing calibration verification standard must be analyzed at the beginning of the run, after every ten samples, and at the conclusion of the run. Refer to Section 8 for specifics.

Method: 04-ANION2

Revision: 7

Date: March 13, 2006

Page: 14 of 30

7.10.3 If the response or retention time for any calibration verification standard analyte varies from its expected values by more than 10%, the standard must be reanalyzed using a fresh calibration standard. If the result is still unacceptable, a new calibration curve shall be prepared and all associated samples be reanalyzed. Refer to Section 8 for specifics.

7.10.3.1 The retention time window for an analyte will be established during a new calibration. The retention time window will be based on the retention times of calibration standards used for that current calibration.

8 Quality Control

8.1 All policies and procedures in the most current revision of the ALSI QA Plan shall be followed when performing this procedure. All analyses and quality-related activities shall be conducted by personnel who have been trained and qualified based on education, experience, external/internal training and evaluation as per ALSI SOP 99-Train. Ongoing proficiency must be established annually as specified in the QA Plan, Technical Training.

8.2 Aqueous Samples

Quality Control Requirements

(Specific Project Requirements may override these requirements)

Parameter	Concentration	Frequency	Control Limits	Corrective Action
Method Blank	---	Beginning of batch, every ten samples, and end of run.	<Reporting Limit. For DoD QSM samples: <1/2 the reporting limit.	Identify and correct the problem. Rerun the blank and all detectable samples since the last acceptable blank. If reanalysis is not possible, report with a qualifying statement.
Calibration Verification Standards (CCV1A) and CCV2A)	Refer to Sections 6.17 and 6.18	Beginning of run, every ten samples, and end of run. Alternate levels.	Method 300: 90 – 110% Method 9056: 95 - 105%	Rerun once. If still unacceptable rerun all associated samples. If reanalysis is not possible report with a qualifying statement.
Calibration Verification Standards (CCV1L and CCV2L)	Refer to Sections 6.19 and 6.20	Only if Method L is used. Beginning of run, every 10 samples, and	Method 300: 90 – 110% Method 9056:	Rerun once. If still unacceptable rerun all associated samples. If

UNCONTROLLED DOCUMENT: DO NOT Transfer or Print

Method: 04-ANION2

Revision: 7

Date: March 13, 2006

Page: 15 of 30

		end of run. Alternate levels.	95 - 105%	reanalysis is not possible, report with a qualifying statement.
Second Source Standard (SSA)	Refer to Section 6.21	Beginning of run, every 20 samples.	90 – 110%	Rerun once. If still unacceptable rerun all associated samples. If reanalysis is not possible report with a qualifying statement.
Second Source Standard (SSL)	Refer to Section 6.22	Only if Method L is used. Beginning of run, every 20 samples.	90 – 110%	Rerun once. If still unacceptable rerun all associated samples. If reanalysis is not possible report with a qualifying statement.
Matrix Spike*	Refer to Section 6.23	1 per 10 samples, with at least 1 per run.	80 – 120%	If all CCV standards associated with the sample are acceptable, report with a qualifying statement.
Matrix Spike Duplicate*	---	Method 300: 1 per 20 samples Method 9056: 1 per 10 samples At least 1 per run.	RPD \leq 20%	Report with a qualifying statement.
Sample Duplicate for DoD sample	---	1 per 10 samples, with at least 1 per run.	\leq 10%	Rerun and if reanalysis is not possible, report with a qualifying statement.

*Samples selected for matrix spike and matrix spike duplicate analyses shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a matrix spike or duplicate may indicate a problem with the sample composition and shall be reported to the client whose sample produced the poor recovery.

*If the method blank concentration is greater than or equal to the reporting limit AND is greater than 1/10 the sample concentration, the source of contamination must be investigated and measures taken to minimize or eliminate the problem and affected samples reanalyzed. If reanalysis is not possible, data shall be reported with a qualifying statement.

8.3 Linear Calibration Range (LCR) – The LCR must be verified every 6 months or whenever a significant change in instrument response is observed or expected. The verification of linearity must use a minimum of a blank and three standards. If any verification data exceeds the initial values by $\pm 10\%$, linearity must be reestablished. If any portion of the range is shown to be non-linear, sufficient

UNCONTROLLED DOCUMENT: DO NOT Transfer or Print

Method: 04-ANION2

Revision: 7

Date: March 13, 2006

Page: 16 of 30

standards must be used to clearly define the nonlinear portion.

- 8.4 Method Detection Limit (MDL) - MDLs shall be determined every 6 months, when a new operator begins work or whenever there is a significant change in the background or instrument response. MDLs must be established for all analytes, using reagent water (blank) fortified at a concentration of two to three times the estimated instrument detection limit. Refer to previous MDL studies for suggested concentrations to be used. To determine MDL values, take seven replicate aliquots of the fortified reagent water and process through the entire analytical method. Perform all calculations defined in the method and report the concentration values in the appropriate units. Calculate the MDL as follows:

$$\text{MDL} = (t) \times (s)$$

where $t = 3.14$ for seven replicates and $s =$ standard deviation of the replicate analyses

An MDL verification check shall always be performed immediately following an MDL study. The MDL check sample must be spiked at approximately 2 times the current reported MDL, and must have a recovery of 50-150%. MDL studies must be performed according to SOP 99-MDL or the reference method, whichever is more frequent.

- 8.5 At least quarterly, a replicate of SSA (Section 6.21) must be analyzed and evaluated for precision.
- 8.6 Solid/Soil Samples: Refer to Section 8.2 Quality Control Requirements for Aqueous Samples. With the exception of the matrix spike and matrix spike duplicate, all requirements listed in this table also apply to solid/soil samples. Additional requirements are as follows.

Quality Control Requirements

(Specific Project Requirements may override these requirements)

Parameter	Concentration	Frequency	Control Limits	Corrective Action
300S Method Blank	---	1 extracted per 20 samples. At least 1 per batch.	<Reporting Limit. For DoD QSM samples: <1/2 the reporting limit.	Re-extract and reanalyze all associated detectable samples. If reanalysis is not possible, report with a qualifying statement.
300S Laboratory Control Sample (6.24)	See Section 6.24	1 extracted per 20 samples. At least 1 per	90 – 110%	Re-extract and reanalyze all associated detectable

UNCONTROLLED DOCUMENT: DO NOT Transfer or Print

Method: 04-ANION2

Revision: 7

Date: March 13, 2006

Page: 17 of 30

		batch.		samples. If reanalysis is not possible, report with a qualifying statement.
300S Matrix Spike* (6.25)	See Section 6.25	1 per 10 samples with at least 1 per batch.	80 – 120%	If 300S LCS is acceptable report with a qualifying statement.
300S Matrix Spike Duplicate*	---	1 per 20 samples with at least 1 per batch.	RPD \leq 20%	Report with a qualifying statement.
Sample Duplicate for DoD sample	---	1 per 10 samples, with at least 1 per run.	\leq 10%	Rerun and if reanalysis is not possible, report with a qualifying statement.

*Samples selected for matrix spike and matrix spike duplicate analyses shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a matrix spike or duplicate may indicate a problem with the sample composition and shall be reported to the client whose sample produced the poor recovery.

*If the LCS is acceptable and the specific matrix interference is identified, report with a qualifying statement. If the specific matrix interference is unknown, reanalyze the sample and matrix spike to determine matrix effect or analytical error.

8.7 Initial Demonstration of Capability

8.7.1 Select and analyze four (4) replicate LCS standards (Section 6.24). All results shall be within 10% of the true value.

8.7.2 Compare the results obtained to the results obtained for the same samples by and analyst previously deemed proficient in the procedure. To be acceptable, the % RPD must be less than 10% for all sample analyzed.

$$\% \text{ RPD} = \frac{\text{Result 1} - \text{Result 2}}{\text{Average Result}} \times 100$$

If the samples analyzed do not meet this requirement, the DOC shall be repeated before independent analysis of samples is begun.

9 Sample Collection, Preservation and Handling

9.1 Samples shall be collected in plastic or glass bottles. All bottles must be thoroughly cleaned and rinsed with reagent water. Volume collected shall be sufficient to insure a representative sample, allow for replicate analysis, if required, and minimize waste disposal.

Method: 04-ANION2

Revision: 7

Date: March 13, 2006

Page: 18 of 30

- 9.2 Sample preservation and holding times for the anions that can be determined by this method are as follows:

<u>Analyte</u>	<u>Preservation</u>	<u>Holding Time</u>
Bromide	None	28 Days
Chloride	None	28 Days
Fluoride	None	28 Days
Nitrate-N	Cool to 4 C	48 Hours
Nitrite-N	Cool to 4 C	48 Hours
Sulfate	Cool to 4 C	28 Days

- 9.3 The method of preservation and the holding time for samples analyzed by this method are determined by the anions of interest. In a given sample, the anion that requires the most preservation treatment and the shortest holding time will determine the preservation treatment.

10 Procedure

- 10.1 Starting the Dionex Ion Chromatograph.

10.1.1 Fill the eluent reservoir with eluent (Section 6.12).

10.1.2 From the Windows desktop, click on the PeakNet icon.

10.1.3 From the PeakNet main Menu, click on Run.

10.1.4 Click the "Load Method" button.

10.1.5 Double click on "Method A XXYY", then OK.

10.1.6 The lights on the Dionex shall light up for the Eluent Pressure, Pump and SRS.

10.1.7 Click on the "Baseline" button and wait for the baseline to stabilize. The pressure shall come up to about 1000 psi or more. The flow shall read approximately 1.2 mL/minute.

10.1.8 Record the pressure, flow, total conductivity and offset conductivity in the appropriate maintenance notebook. Also complete maintenance checklist daily (located in the beginning of the same book).

10.2 Create the Sample Analysis Schedule

10.2.1 From the Dionex Main Menu, click on Schedule.

10.2.2 An example of the sequence of samples and QC checks, is as follows:

- 1) Blank
- 2) CCV Level 1A
- 3) CCV Level 1L
- 4) SSA
- 5) SSL
- 6) CCV Level 2A
- 7) CCV Level 2L
- 8) Shutdown
- 9–16) Samples
- 17) MS
- 18) MSD
- 19) CCV Level 1A
- 20) CCV Level 1L
- 21) Blank
- 22–30) Samples
- 31) MS
- 32) CCV Level 2A
- 33) CCV Level 2L
- 34) Blank
- 35) Shutdown

* Note: Second source standards are not required if no further samples are followed for analysis.

10.2.3 Under Sample, the sample number or the type of QC check shall be entered. For samples that are diluted, follow the sample number with the dilution factor, so that it will print out on the PeakNet report.

10.2.4 Under Sample Type, enter “Sample” for all.

10.2.5 Under Level, enter nothing.

10.2.6 Under Method, enter Method A XXYY.met or Method L XXYY.met depending on which calibration will be used for that sample.

10.2.7 Under Data File, change the file name to be “the date” followed by run

UNCONTROLLED DOCUMENT: DO NOT Transfer or Print

Method: 04-ANION2

Revision: 7

Date: March 13, 2006

Page: 20 of 30

number, followed by “_A001.DXD”. For example, the file name for the first sample analysis run on February 8, 1999 would be 020899RUN1_A001.DXD. Copy this same file name down for all samples and QC checks.

10.2.8 Under comment, place the batch number.

10.2.9 For groundwater samples that are known or suspected to be high in anion concentrations, check the history of sample analyses in the LIMS to determine what sample dilutions are required. A reference sheet shall be maintained for the routinely received samples that require dilutions.

10.2.10 PeakNet will multiply the dilution factor times any positive hits in Horizon LIMS.

10.2.11 If the instrument shuts down after the analysis run, the last line of the schedule shall have a name of “Shutdown”, sample type shall be “Sample”, and the shutdown method (SHUTDOWN.MET) shall be under the method column.

10.2.12 Click the “File/Save As”.

10.2.13 Give the schedule a file name similar to the data file naming procedure. The file name shall be “the date” followed by run number. For example, the file name for the second schedule run on February 8, 1999 would be 020899RUN2.SCH.

10.2.14 Print a hard copy of the schedule and save it for the data review process.

10.2.15 Close the Schedule Editor Window.

10.2.16 Place a copy of the schedule into the IC Log Runbook and record on the schedule the analysis date, analyst, calibration date, standards, pipettes and reagents used.

10.3 Sample Analysis

10.3.1 Back at the Run Window, click on the “Load Schedule” button.

10.3.2 Double click on the schedule name as set above, in Section 10.2.12.

10.3.3 Samples that are relatively free of suspended solids do not require pre-

UNCONTROLLED DOCUMENT: DO NOT Transfer or Print

Method: 04-ANION2

Revision: 7

Date: March 13, 2006

Page: 21 of 30

filtering. The autosampler vial caps contain a filtering frit in them.

10.3.4 Samples containing finely divided particles require pre-filtering through a 0.45-micron nylon filter. The use of a 0.45-micron filter syringe is an acceptable procedure for filtering. If the sample contains fine particles after filtration, make a 1:10 dilution on the sample and record the dilution factor in the schedule.

10.3.5 DO NOT RUN PRESERVED SAMPLES. Ask customer service to change to a more suitable method.

10.3.6 Pour 5 mL of the appropriate sample (or 5 mL of a diluted sample) and the QC checks into the autosampler vials. Add 0.035 mL of 100x eluent concentrate (Section 6.13) to each sample that requires a dilution at full strength. (Do not add 100x eluent to blanks and standards since they are made up in working eluent.) Refer to the sequence in the schedule and position them into the appropriate autosampler tray position.

10.3.7 Push the "Hold/Run" button on the AS40 Autosampler so that the light is in the Run position.

10.3.8 Once the ready light comes on, push the "Load" button on the AS40 Autosampler.

10.3.9 Wait for the Load button to start blinking (1 to 2 minutes).

10.3.10 On the PeakNet Run window, click on the Run Menu, then Start.

10.4 Preparation of Solid Samples

10.4.1 Weigh 10 ± 0.5 grams of solid material into a glass or plastic beaker. Record the weight in the IC solids prep logbook.

10.4.2 Add 100 mL of reagent water to the beaker.

10.4.3 This slurry is mixed for ten minutes using a magnetic stirrer.

10.4.4 Filter the resulting slurry using a 0.45-micron membrane type filter. This can be done by using a 0.45-micron filter syringe, or through the use of a vacuum pump.

10.4.5 Schedule the sample with a dilution of 10 to compensate for the 1:10 ratio

UNCONTROLLED DOCUMENT: DO NOT Transfer or Print

Method: 04-ANION2

Revision: 7

Date: March 13, 2006

Page: 22 of 30

of sample to reagent water.

10.4.6 Analyze the filtrate according to Section 10.1-10.3.

10.4.7 Report the result in mg/kg. The LIMS will make the adjustment for the dry weight conversion.

10.5 Data Review

10.5.1 Review the PeakNet schedule to make certain that all samples were identified properly, all dilution factors were correct and the correct method calibration was used to process the samples.

10.5.2 Review each sample report and its chromatogram to make certain that the retention time windows have properly identified each analyte. The width of the retention time window shall be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time can be used to calculate a suggested window size. However, due to possible shifts in retention times for an individual sample due to the sample's ionic strength, the experience of the analyst shall weigh heavily in chromatogram interpretation. If the identity of a peak is in question, the sample shall be spiked and reanalyzed for confirmed.

If peaks have not been identified properly, they can be renamed through the Optimize/Name Peaks menu item. If this option is used, both the original and modified chromatograms must be included with the raw data.

10.5.3 Review each sample chromatogram to make certain that baselines are drawn correctly. If modifications are required they can be made through the Optimize/Adjust Baseline menu item. Whenever manual integrations are performed both the original and modified chromatogram must be included with the raw data. Refer to SOP 99-Integration as a guideline for manual integration procedures. The date, initials of the analyst, and a comment shall also be included as to the reason for the manual integration.

10.5.4 Review each sample report to make certain that the reported concentrations are within the lowest and highest calibration standard. If the results are above the highest standard, the sample must be rerun at a different dilution.

Method: 04-ANION2

Revision: 7

Date: March 13, 2006

Page: 23 of 30

10.5.5 Review all quality control samples for comparison to the acceptance criteria in Sections 8.2 and 8.6.

10.5.6 Review each sample to make certain that all analytes were analyzed within the appropriate hold times.

11 Calculations

11.1 No sample calculations are required. PeakNet performs all calculations.

11.2 Standard Recovery is calculated as:

$$\% \text{ Recovery} = \frac{(\text{Result, mg/L})}{(\text{True Value, mg/L})} \times 100$$

11.3 Spike recovery is calculated as:

$$\% \text{ Recovery} = \frac{(\text{Spike Result} - \text{Unspiked Sample, mg/L})}{\text{Spike Added, mg/L}} \times 100$$

11.4 Precision (Relative Percent Difference, RPD) is calculated as:

$$\% \text{ RPD} = \frac{(\text{Result 1} - \text{Result 2})}{\text{Average Result}} \times 100$$

11.5 LCS Recovery is calculated as:

$$\% \text{ Recovery} = (\text{Cm/Cn}) \times 100$$

where Cm = measured concentration of LCS

Cn = spiking concentration

12 Reporting Results

12.1 Report only those values that are greater than the reporting limits and fall between the lowest and highest calibration standards. For samples diluted due to matrix interferences, the reporting limits are also increased by the same sample dilution factor. If a sample is diluted because of an over-range analyte, the reporting limit shall not be raised. Report the actual result, even if it is less than the reporting limit. Any sample with a result less than the reporting limit is reported as ND (non-detectable); LIMS will automatically report the appropriate

detection limit.

- 12.2 In cases where samples are run at full strength and at a dilution, use the reporting limit for the undiluted sample, except for when matrix interferences prevent the analyst from accurately reporting the lower reporting limit. The reporting limits for undiluted samples are as follows:

	Method A(mg/L)	Method L (mg/L)
Fluoride	0.10	0.10
Chloride	10.0	1.00
Nitrite	0.10	0.10
Bromide	0.10	0.10
Nitrate	1.50	0.10
Sulfate	10.0	1.00

- 12.3 Horizon LIMS results are reported to three significant figures but limited to the number of decimal places in the reporting limit for the individual compound or analyte.
- 12.4 When entering data into the Horizon LIMS do not round off results: Horizon will automatically round off to 3 significant figures after all internal calculations are completed.
- 12.5 Samples exceeding the Maximum Contaminant Level (MCL) must be reported to the Customer Service Representative immediately following determination in order to comply with the Pennsylvania Code: Title 25, Chapter 109, Section 109.810 for Reporting and Notification Requirements.
- 12.6 All raw data used for reporting results must be dated and initialed by the qualified laboratory personnel performing first and second review.

13 Waste Disposal

- 13.1 Refer to ALSI SOP 19-Waste Disposal.

14 Pollution Prevention

- 14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities

UNCONTROLLED DOCUMENT: DO NOT Transfer or Print

Method: 04-ANION2

Revision: 7

Date: March 13, 2006

Page: 25 of 30

for pollution prevention exist in laboratory operations. Management shall consider pollution prevention a high priority. Extended storage of unused chemicals increases the risk of accidents. The laboratory shall consider smaller quantity purchases which will result in fewer unused chemicals being stored and reduce the potential for exposure by employees. ALSI tracks chemicals when received by recording their receipt in a traceable logbook. Each chemical is then labeled according to required procedures and stored in assigned locations for proper laboratory use.

15 Definitions

- 15.1 Refer to ALSI QA Plan under Laboratory Quality Control Checks for general definitions.
- 15.2 Calibration Standard Solution: A solution prepared from the primary dilution standard solution or stock standard solutions and the internal standards and surrogate analytes. These solutions are used to calibrate the instrument response with respect to analyte concentration.
- 15.3 Eluent Solution: A specialized solution run through an analytical instrument to maintain operational quality.
- 15.4 Laboratory Control Standard (LCS): A standard usually certified by an outside agency, used to measure the bias in a procedure. For certain constituents and matrices, use the National Institute of Standards and Technology (NIST) Standard Reference Materials when available.
- 15.5 Linear Calibration Range (LCR): The concentration range over which the instrument response is linear.
- 15.6 Material Safety Data Sheet (MSDS): Written information provided by vendors concerning a chemical's toxicity, health hazards, physical properties, fire and reactivity data including storage, spill and handling precautions.
- 15.7 Method Detection Limit (MDL): The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero.
- 15.8 Second Source Standard: A Laboratory Control Standard that represents a completely different origin than the LCS.

16 Troubleshooting

UNCONTROLLED DOCUMENT: DO NOT Transfer or Print

Method: 04-ANION2

Revision: 7

Date: March 13, 2006

Page: 26 of 30

- 16.1 Refer to maintenance logs and instrument manuals for guidance in troubleshooting specific problems related to the instrumentation used in this method.

SOP Change History Summary

<u>Section No.</u>	<u>Section</u>	<u>Reason for Change</u>
1.2	Scope and Application	Removed redundant MDL statement explained

UNCONTROLLED DOCUMENT: DO NOT Transfer or Print

Method: 04-ANION2

Revision: 7

Date: March 13, 2006

Page: 27 of 30

		in Section 1.5.
3.5	Interferences	Added clarifying statement in last sentence.
6	Reagents	Added "Note"
6.14, 6.16	Reagents	PA DEP audit response
7.3	Sample Collection...	Added full section for further direction.
10.2.9	Procedure	Removed "AMS LIMS"; no longer applies
17.2 – 17.8	Definitions	Added definitions for term clarification

Revision 7: 03/13/2006

Throughout SOP: Grammatical, spelling and format revisions were made and references added for expiration dates.

1.6	Scope and Application	Added project requirement verbiage
4.2	Safety	Added MSDS availability
5.1	Apparatus and Materials	Revised balance identification
5.7	Apparatus and Materials	Revised pipette volume
5.13, 5.14	Apparatus and Materials	Added materials
Throughout 6	Reagents	Added chemical names and respective section References
6.12	Reagents	Added descriptive degassing details
6.15	Reagents	Added preparation frequency

SOP Change History Summary (continued)

<u>Section No.</u>	<u>Section</u>	<u>Reason for Change</u>
6.26	Reagents	Added sand details

UNCONTROLLED DOCUMENT: DO NOT Transfer or Print

Method: 04-ANION2

Revision: 7

Date: March 13, 2006

Page: 28 of 30

7.9 and	Instrument Calibration	Added corrective action for calibration blank DoD sample requirements
7.10.3.1	Instrument Calibration	Retention time window verbiage added
8.1	Quality Control	Added verbiage to address ongoing proficiency
8.2	Quality Control	Added DoD Sample Duplicate reference; added corrective action for method blank
8.4	Quality Control	Added MDL verification check verbiage
8.6	Quality Control	Added DoD Sample Duplicate reference; added corrective action for matrix spike
8.7	Quality Control	Added Initial Demonstration of Capability
10.2.2	Procedure	Revised sequence 31 to read “MS” instead of “MSD”; added Note
10.2.8	Procedure	Added section for batch # placement
10.3.4	Procedure	Added “Nylon” to filter description
10.3.6	Procedure	Added dilution strength
10.4.4	Procedure	Added alternate filtering method
10.5.3	Procedure	Added reference to SOP 99-Integration
11.5	Calculations	Added LCS Recovery calculation
12.1	Reporting Results	Added sentence addressing Non-detects

SOP Change History Summary (continued)

<u>Section No.</u>	<u>Section</u>	<u>Reason for Change</u>
12.5, 12.6	Reporting Results	Added sections concerning CSR notification and raw data reporting

UNCONTROLLED DOCUMENT: DO NOT Transfer or Print

Method: 04-ANION2

Revision: 7

Date: March 13, 2006

Page: 29 of 30

16

Troubleshooting

Added section

Method: 04-OP
Revision: 4
Date: July 20, 2006
Page 1 of 16

Document Title: Orthophosphate

Document Control Number: _____

Organization Title: ANALYTICAL LABORATORY SERVICE, INC. (ALSI)

Address: 34 Dogwood Lane
Middletown, PA 17057

Phone: (717) 944-5541

Approved by:

_____	_____
Helen MacMinn, Quality Assurance Manager	Date
_____	_____
Jason Badman, Wet Chemistry Supervisor	Date
_____	_____
Megan Rebuck, Validator	Date

Annual Review:

_____	_____
Reviewed By	Date Reviewed
_____	_____
Approved By	Date Approved

_____	_____
Reviewed By	Date Reviewed
_____	_____
Approved By	Date Approved

Method: 04-OP
Revision: 4
Date: July 20, 2006
Page 3 of 16

TABLE OF CONTENTS

1 Scope and Application 4
2 Summary of Method 4
3 Interferences 4
4 Safety 5
5 Apparatus and Materials 5
6 Reagents and Standards 6
7 Instrument Calibration 8
8 Quality Control 9
9 Sample Collection, Preservation and Handling 11
10 Procedure 11
11 Calculations 11
12 Reporting Results 12
13 Waste Disposal 12
14 Pollution Prevention 12
15 Definitions 12
16 Troubleshooting 12
Appendix A 13
SOP Change Summary 14
SOP Concurrence Form 16

Method: 04-OP
Revision: 4
Date: July 20, 2006
Page 4 of 16

1 Scope and Application

- 1.1 This document states the laboratory's policies and procedures established in order to meet the requirements of all certifications/accreditations currently held by the laboratory, including the most current NELAC standards.
- 1.2 This standard operating procedure is adapted from Standard Methods for the Examination of Water and Wastewater, 20th ed. Method 4500-PE, "Phosphorus, Ascorbic Acid Method." This SOP covers the determination of orthophosphate in drinking, surface and saline waters, domestic and industrial wastes.
- 1.3 This method is restricted for use by or under the supervision of analysts experienced in the determination of orthophosphate.
- 1.4 This method is applicable in the approximate range from 0.02 to 1.0 mg/L.
- 1.5 Method Detection Limits can be found in the current Wet Chemistry method detection limit book. The detection limits for a specific sample may differ from those listed due to the nature of interferences in a particular sample matrix.
- 1.6 Individual project requirements may override criteria listed in SOP.

2 Summary of Method

- 2.1 Ammonium molybdate and antimony potassium tartrate react in an acid medium with ortho-phosphate to form an antimony-phospho-molybdate complex. This complex is reduced to an intensely blue-colored complex by ascorbic acid. The color is proportional to the orthophosphate concentration.
- 2.2 Only orthophosphate forms a blue color in this test. Polyphosphates (and some organic phosphorus compounds) may be converted to the orthophosphate form by sulfuric-acid-hydrolysis. Organic phosphorus compounds may be converted to the orthophosphate form by persulfate digestion.

3 Interferences

- 3.1 Arsenate is determined similarly to phosphorus and shall be considered when present. This interference may be eliminated by reducing the arsenic acid to arsenious acid with sodium bisulfite.
- 3.2 When high concentrations of iron are present low recovery of phosphorus will be obtained because it will use some of the reducing agent. The bisulfite treatment will also eliminate this interference.

Method: 04-OP
Revision: 4
Date: July 20, 2006
Page 5 of 16

- 3.3 Turbidity and color in the sample will interfere with the absorbance readings on the spectrophotometer. Preliminary filtration may be necessary for turbid samples. For colored samples, sample blanks or serial dilutions may be necessary to compensate for the color.
- 3.4 Hexavalent Chromium and NO_2^- interfere to give results 3% low at concentrations of 1 mg/L and 10% to 15% low at concentrations of 10 mg/L.

4 Safety

- 4.1 The toxicity or carcinogenicity of each reagent used in this method may not have been fully established. Each chemical shall be regarded as a potential health hazard and exposure shall be as low as reasonably achievable.
- 4.2 ALSI maintains material safety data sheets (MSDS) on all chemicals used in this procedure. MSDSs are available to all staff and are located in the QA office.
- 4.3 The following chemicals have the potential to be highly toxic or hazardous.
 - 4.3.1 Antimony potassium tartrate.
 - 4.3.2 Sulfuric acid.
- 4.4 The minimum personal protective equipment (PPE) needed to run this analysis is a fully-button lab coat, safety glasses, and vinyl or latex gloves.

5 Apparatus

- 5.1 Spectrophotometer – For use at 880 nm, with a light path of 1 cm or longer - Shimadzu UV-1201, or equivalent.
- 5.2 Volumetric flasks - Class A, various sizes purchase from VWR.
- 5.3 Class A pipets, various sizes, purchased from VWR.
- 5.4 Graduated cylinders – Class A, various sizes, purchased from VWR.
- 5.5 Pipet bulbs - purchased from VWR catalog #53947-202 or equivalent.
- 5.6 Cuvettes - 10 mm, purchased from HACH, catalog #2095100, or equivalent.
- 5.7 150 mL plastic or glass beakers – purchased from VWR.
- 5.8 0.45 micron filter paper - Purchased from VWR catalog #28148-584, or equivalent.

Method: 04-OP
Revision: 4
Date: July 20, 2006
Page 6 of 16

- 5.9 Filtering flask – purchased from VWR, catalog #29410-993, or equivalent.
- 5.10 Filter funnel – purchased VWR, catalog #28143-550, or equivalent.
- 5.11 Vacuum Pump, Model SA55JXGTD-4144, purchased from VWR Scientific, catalog #54906-001 or equivalent.

6 Reagents

- 6.1 Reagent Water – Reagent water is water in which an interferant is not observed at the analyte of interest. For this purpose ALSI uses a Filson Water Purification System that provides analyte-free, greater than 16.0 megohm-cm deionized water on demand.
- 6.2 Sulfuric Acid (concentrated) - purchased from VWR catalog #JT9681-33, or equivalent. If the reagent is unopened, assign an expiration as per manufacturer's assigned dating or 5 years post receipt, whichever is less. Once the reagent has been opened, assign an expiration of one year. Store at room temperature in an acid cabinet.
- 6.3 Sulfuric Acid Solution (5 N) - Using a graduated cylinder, slowly add 70 mL concentrated H₂SO₄ to approximately 300 mL reagent water in a 500 mL volumetric flask. Allow to cool and dilute to the mark with reagent water. Store at room temperature for up to 6 months.
- 6.4 Ammonium molybdate - purchased from VWR catalog #JT0716-1, or equivalent; expiration as per manufacturer's assigned dating or 5 years post receipt, whichever is less; hold at room temperature on a dry shelf.
- 6.5 Ammonium Molybdate Solution – Dissolve 20 g (NH₄)₆ MO₇O₂₄·4H₂O in reagent water and dilute to 500 mL in a volumetric flask. Reagent is stable for 1 month; store at room temperature.
- 6.6 Antimony Potassium Tartrate – Purchased from Fisher Scientific, catalog #A-865, or equivalent; expiration as per manufacturer's assigned dating or 5 years post receipt, whichever is less; hold at room temperature on a dry shelf.
- 6.7 Antimony Potassium Tartrate Solution – Dissolve 1.3715 g K(SbO) C₄H₄O₆ · ½ H₂O in reagent water and dilute to 500 mL in a volumetric flask. Reagent is stable for 1 month; store at room temperature.
- 6.8 Ascorbic Acid – purchased from VWR, catalog #JTB581-7, or equivalent; expiration as per manufacturer's assigned dating or 5 years post receipt, whichever is less; hold at room temperature on a dry shelf.

Method: 04-OP
Revision: 4
Date: July 20, 2006
Page 7 of 16

- 6.9 Ascorbic Acid Solution – Dissolve 1.76 g ascorbic acid in reagent water and dilute to 100 mL in a volumetric flask. This solution is stable for 1 week held above the freezing point of water up to 6°C.
- 6.10 Combined Reagent – Mix the above reagents in the following proportions for 100 mL of combined reagent: 50 mL 5 N H₂SO₄, 5 mL antimony potassium tartrate, 15 mL ammonium molybdate solution, and 30 mL ascorbic acid solution. Mix after addition of each reagent. Let all reagents reach room temperature before they are mixed and mix in the order given. If turbidity forms, shake and let stand for a few minutes until turbidity disappears before proceeding. The reagent is stable for 4 hours.
- 6.11 Potassium dihydrogen phosphate (KH₂PO₄) – purchased from VWR catalog #EM-5108-1, or equivalent; expiration as per manufacturer's assigned dating or 5 years post receipt, whichever is less; hold at room temperature on a dry shelf. Dry in an oven at 104°C for one hour before using. Following drying, this reagent may be stored in a desiccator for up to 3 months.
- 6.12 Stock Phosphorus Solution (1000 mg/L) - Dissolve 4.3936 g of KH₂PO₄, which has been dried, in approximately 800 mL reagent water in a 1000 mL volumetric flask. Dilute to the mark. Hold for 3 months at room temperature.
- 6.13 Standard Phosphorus Solution (1 mg/L) - Dilute 2 mL of the Stock Phosphorus Solution (Section 6.12) to 2000 mL or a series of serial dilutions can be made for a total of a 1/1000 dilution of the stock. Discard after 24 hours.
- 6.14 Second Source Stock Solution (1000 mg/L) - purchased from HACH catalog #23211-42. Hold for 3 months at room temperature.
- 6.15 Second Source Standard Solution (1 mg/L). Dilute 1 mL of the second source stock solution (Section 6.14) to 1000 mL in a 1 liter volumetric or a series of serial dilutions can be made for a total of a 1/1000 dilution of the stock. Discard after 24 hours.
- 6.16 Calibration Verification Standard Level 1 (0.20 mg/L) – Dilute 10 mL of second source standard solution (Section 6.15) to volume in a 50 mL volumetric. Discard after 24 hours.
- 6.17 Calibration Verification Standard Level 2 (0.80 mg/L) – Dilute 40 mL of second source standard solution (Section 6.15) to volume in a 50 mL volumetric. Discard after 24 hours.
- 6.18 Phenolphthalein powder – available from VWR, catalog #JT2870-4 or equivalent. Store at room temperature until manufacturer's expiration date.
- 6.19 90% Ethyl Alcohol Reagent grade – available from VWR, catalog #VW0470-3 or equivalent. Store at room temperature in a flammables cabinet for up to five years

Method: 04-OP
Revision: 4
Date: July 20, 2006
Page 8 of 16

post receipt.

6.20 Phenolphthalein indicator - Add 5 g of phenolphthalein powder (Section 6.18) to 500 mL Ethyl Alcohol (Section 6.19) in a 1 L volumetric. Dilute to volume. Store at room temperature for 6 months.

7 Instrument Calibration

7.1 Using the standard phosphorus solution (Section 6.13), prepare the following standards in 50 mL volumetric flasks: If 50 mL volumetric flasks are not available, larger sizes can be used. Make sure to adjust the mL of standard phosphorus solution used accordingly.

<u>mL of Standard Phosphorus Solution</u>	<u>Concentration, mg/L</u>
0	0.00
5.0	0.10
10.0	0.20
20.0	0.40
30.0	0.60
40.0	0.80
50.0	1.00

7.2 Measure 50 mL of each standard into an appropriately labeled beaker.

7.3 Add 1 drop phenolphthalein in indicator.

7.4 If solution turns pink add 5 N H₂SO₄ drop wise until color disappears.

7.5 Add 8 mL combined reagent and mix.

7.6 Zero the spectrophotometer at 880 nm using reagent water.

7.7 Let stand at least 10 but no more than 30 minutes and measure the absorbance of each standard.

7.8 Place the standards to be read into the same cuvette used to zero the spectrophotometer. Read and record absorbance.

7.9 Access the curve generation program that uses linear regression analysis. Access this program in the current Network server of the Network Neighborhood. Open folder "WET CHEM DEPT." Open folder "CURVES." Open folder "Ortho Phosphate."

7.10 Enter the concentrations of the calibration standards in mg/L in the "x" column starting

Method: 04-OP
 Revision: 4
 Date: July 20, 2006
 Page 9 of 16

with the 0.10 mg/L standard. Enter the corresponding absorbances in the “y” column.

- 7.11 The correlation coefficient of the calibration curve must be 0.995 or greater for the curve to be acceptable. Re-analyze the calibration curve if this criteria is not met.
- 7.12 After a calibration curve has been established, a separate source QC sample must be analyzed to verify the curve. If this fails, it shall be remade and reanalyzed. If the QC sample continues to fail, a new curve shall be prepared.
- 7.13 The calibration curve must be prepared at a minimum of every three months or whenever the QC sample fails, whichever comes first.

8 Quality Control

- 8.1 All policies and procedures in the most current revision of the ALSI QA Plan shall be followed when performing this procedure.

8.2 Quality Control Requirements

(Specific Project Requirements may override these requirements)

Parameter	Concentration	Frequency	Control Limits	Corrective Action
Method Blank	--	Beginning of run, every 10 samples, and end of run.	<Reporting Limit (0.02 mg/L) or <1/10 sample concentration	Investigate and correct the source of contamination, reanalyze the blank and any associated samples. If reanalysis is not possible report with a qualifying statement.
Calibration Verification Standard	Level 1 – 0.20 mg/L Level 2 – 0.80 mg/L	Beginning of run, every 10 samples, and end of run. Alternate levels.	90 – 110%	Investigate the source of the problem and reanalyze all samples since the last acceptance standard. If reanalysis is not possible report with a qualifying statement.
Matrix Spike **	0.50 mg/L*	One per 10 samples with at least 1 per batch.	90 – 110%	If Calibration Verification Std. meets criteria, reanalyze the spike once. If the spike still fails or if reanalysis is

Method: 04-OP
 Revision: 4
 Date: July 20, 2006
 Page: 10 of 16

				not possible report with a qualifying statement.
Matrix Spike Duplicate**	--	One per 10 samples with at least 1 per batch	± 10%	Reanalyze the duplicate once. If the duplicate still fails or if reanalysis is not possible report with a qualifying statement.

* To prepare a 0.50 mg/L spike pipet 0.025 mL of stock phosphorus solution (Section 6.12) into 50 mL of sample.

**Samples selected for duplicate and matrix spike analysis shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a duplicate or spike analysis may indicate a problem with the sample composition and shall be reported to the client whose sample produced the poor recovery.

8.3 Initial Demonstration of Capability

8.3.1 Prepare and analyze four replicate Level 1 calibration verification standards (Section 6.16). All four results shall be within 10% of their true value. If the standards analyzed do not meet this requirement, the DOC shall be repeated before independent analysis of samples begins.

8.3.2 Using the data generated in Section 8.3.1, calculate the percent relative standard deviation (%RSD) of the replicate analysis as indicated below. To be acceptable, the % RSD must be less than 10%. If the standards analyzed do not meet this requirement, the DOC shall be repeated before independent analysis of samples is begun.

$$\%RSD = \frac{\text{Sample Standard Deviation (S)}}{\text{Mean Recovered Concentration}} \times 100$$

8.3.3 Ongoing proficiency must be established annually as specified in the QA Plan, Technical Training.

8.4 MDL studies must be performed according to SOP 99-MDL or the reference method, whichever is more frequent.

9 Sample Collection, Preservation and Handling

9.1 Refer to SOP 20 – Field Services Sampling Plan.

9.2 Sample containers may be plastic or glass. Samples shall be cooled at the time of collection and stored above the freezing point of water up to 6°C.

Method: 04-OP
Revision: 4
Date: July 20, 2006
Page 11 of 16

9.3 The maximum holding time for this analysis is 48 hours.

10 Procedure

10.1 Prepare a reagent blank and the appropriate check standards (Section 6.16 and 6.17) for the analytical batch.

10.2 Using a graduated cylinder, measure 50 mL of each sample into an appropriately labeled beaker. If samples are colored or turbid, filtration may be required.

10.2.0 Filter samples through 0.45 µm membrane filters. A glass fiber filter may be used to pre-filter hard-to-filter samples. Filters shall be pre-rinsed with 1:1 HCl and deionized water prior to use.

10.3 Prepare any spikes that are required.

10.4 Process samples following Sections 7.3 through 7.5.

10.5 Zero the spectrophotometer at 880 nm using reagent water.

10.6 After 10-30 minutes measure the absorbance of each sample.

10.7 Any absorbances that fall above the highest calibration standard's absorbance shall be diluted until it falls within the range of the curve.

10.8 Correct absorbance readings for sample blanks. Sample blanks shall contain everything except the ascorbic acid solution.

10.9 Plug absorbance values into the linear regression equation to determine the orthophosphate concentration - making corrections to this value for any dilutions that may have occurred.

11 Calculations

11.1 Total mg P/L = $\frac{\text{mg P (in approximately 58 mL final volume)} \times 1000}{\text{mL sample}}$

11.2 RPD = $\frac{(\text{Difference between results})}{(\text{Average of results})} \times 100$

11.3 Spike recovery = $\frac{(\text{Spiked sample result} - \text{Sample results})}{(\text{Spike amount})} \times 100$

12 Reporting Results

Method: 04-OP
Revision: 4
Date: July 20, 2006
Page 12 of 16

- 12.1 All raw data used for reporting results must be dated and initialed by the qualified laboratory personnel performing first and second review.
- 12.8 When entering data into Horizon LIMS, do not round off results: Horizon will automatically perform rounding appropriate to the method. Horizon LIMS results are reported to three significant figures but limited to the number of decimal places in the reporting limit for the individual compound or analyte.
- 12.3 Report the actual result, even if it is less than the reporting limit. Any sample with a result less than the reporting limit is reported as ND (non-detectable); LIMS will automatically report the appropriate detection limit.

13 Waste Disposal

- 13.1 Refer to ALSI SOP 19-Waste Disposal.

14 Pollution Prevention

- 14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. Management shall consider pollution prevention a high priority. Extended storage of unused chemicals increases the risk of accidents. The laboratory shall consider smaller quantity purchases which will result in fewer unused chemicals being stored and reduce the potential for exposure by employees. ALSI tracks chemicals when received by recording their receipt in a traceable logbook. Each chemical is then labeled according to required procedures and stored in assigned locations for proper laboratory use.

15 Definitions

- 15.1 Refer to ALSI QA Plan under Laboratory Control Checks for general definitions.

16 Troubleshooting

- 16.1 Refer to maintenance logs and instrument manuals for guidance in troubleshooting specific problems related to the instrumentation used in this method.

Method: 04-OP
Revision: 4
Date: July 20, 2006
Page 14 of 16

SOP Change History Summary

<u>Section #</u>	<u>Section</u>	<u>Reason for Change</u>
<u>Revision 4: 07/20/2006</u>		
Refrigeration temperature range revised throughout SOP to reflect NELAC parameters.		
1.2	Scope and Application	Revised Standard Method to 20 th Edition
1.6	Scope and Application	Added statement of project requirement criteria
3.4	Interferences	Added interference of hexavalent chromium and NO ₂
4.2	Safety	Added MSDS availability
5.11	Apparatus and Materials	Added vacuum pump
6.18-9.20	Reagents	Added reagents
7.7	Instrument Calibration	Added more detailed statement of limits
7.12	Instrument Calibration	Added corrective action for separate source QC sample curve verification failure
8.3	Quality Control	Added section on DOC and ongoing proficiency
8.4	Quality Control	Added section about MDL and SOP reference
9.1	Sample Collection...	Added SOP 20-Field Services Sampling Plan reference
11	Calculations	Added calculation

Method: 04-OP
Revision: 4
Date: July 20, 2006
Page 15 of 16

SOP Change History Summary (continued)

<u>Section #</u>	<u>Section</u>	<u>Reason for Change</u>
12	Reporting Results	Revised/added sections to reflect current practices
15	Definitions	Added section
16	Troubleshooting	Added section
A	Appendix	Added section

UNCONTROLLED DOCUMENT: DO NOT Print or Transfer

Method: 04-S
Revision: 4
Date: November 22, 2006
Page 1 of 16

Document Title: The Determination of Sulfide in Water and Wastewater

Document Control Number: _____

Organization Title: ANALYTICAL LABORATORY SERVICE, INC. (ALSI)

Address: 34 Dogwood Lane
Middletown, PA 17057

Phone: (717) 944-5541

Approved by: _____
Helen MacMinn, Date
Quality Assurance Manager

Jason Badman, Date
Wet Chemistry Supervisor

Adam Hough, Date
Validator

Annual Review:

Reviewed By Date Reviewed

Approved By Date Approved

Reviewed By Date Reviewed

Approved By Date Approved

Method: 04-S
Revision: 4
Date: November 22, 2006
Page 2 of 16

Annual Review (continued):

Reviewed By Date Reviewed

Approved By Date Approved

Reviewed By Date Reviewed

Approved By Date Approved

Reviewed By Date Reviewed

Approved By Date Approved

Reviewed By Date Reviewed

Approved By Date Approved

Reviewed By Date Reviewed

Approved By Date Approved

Method: 04-S
Revision: 4
Date: November 22, 2006
Page 3 of 16

TABLE OF CONTENTS

1	Scope and Application	4
2	Summary of Method	4
3	Interferences.....	4
4	Safety	5
5	Apparatus and Materials	5
6	Reagents	6
7	Instrument Calibration	7
8	Quality Control	7
9	Sample Collection, Preservation and Handling.....	9
10	Procedure	10
11	Calculations.....	11
12	Reporting Results.....	11
13	Waste Disposal.....	12
14	Pollution Prevention.....	12
15	Definitions.....	12
16	Troubleshooting	13
	Appendix A	14
	SOP Change History Summary	15
	SOP Concurrence Form	16

Method: 04-S
Revision: 4
Date: November 22, 2006
Page 4 of 16

1 Scope and Application

- 1.1 This method is adapted from the U.S. EPA Method 376.1, "Sulfide, Titrimetric, Iodine" and "Standard Methods for the Examination of Water and Wastewater", 20th Edition, Method 4500F, "Iodometric Method".
- 1.2 This method is applicable to the measurement of total sulfides in drinking, surface, and saline waters, domestic and industrial wastes.
- 1.3 This document states the laboratory's policies and procedures established in order to meet the requirements of all certifications/accreditations currently held by the laboratory, including the most current NELAC standards.
- 1.4 Method detection limits (MDL) can be found on the ALSI network in the Wet Chemistry Department MDL tab, which is maintained and updated by the QA department. The detection limits for a specific sample may differ from those listed due to the nature of interferences in a particular sample matrix.
- 1.5 This method is suitable for the measurement of sulfide in concentrations above 1 mg/L.
- 1.6 Acid insoluble sulfides are not measured by use of this test. (Copper sulfide is the only common sulfide in this class).
- 1.7 Individual project requirements may override criteria listed in this SOP.
- 1.8 This method is restricted to use by or under the supervision of analysts trained to run the sulfide procedure.

2 Summary of Method

- 2.1 Excess iodine is added to a sample which has been treated with zinc acetate to produce zinc sulfide. The iodine oxidizes the sulfide to sulfur under acidic conditions. The excess iodine is back titrated with sodium thiosulfate.

3 Interferences

- 3.1 The iodometric method suffers interference from reducing substances that react with iodine, including thiosulfate, sulfite, and various organic compounds, both solid and dissolved.
- 3.2 Thiosulfates at concentrations about 10 mg/L retard color formation or completely prevent it. Sulfide itself prevents the reaction if its concentration is very high, in the

Method: 04-S
Revision: 4
Date: November 22, 2006
Page 5 of 16

range of several hundred mg/L. Iodine that is likely to be present in oil-field wastewaters may diminish color formation if its concentration exceeds 2 mg/L. Ferrocyanide produces a blue color.

- 3.3 Eliminate interferences due to sulfite, thiosulfate, iodide, and many other soluble substances, but not ferrocyanide, by first precipitating ZnS, removing the supernatant, and replacing it with distilled water. Use the same procedure, even when not needed for removal of interferences, to concentrate sulfide.

4 Safety

- 4.1 MSDSs are available to all staff and are located in hard copy format in the lower level conference room and electronically on the ALSI public network, F:\MSDS - Material Safety Data Sheets. All laboratory personnel must read, understand and comply with the ALSI Chemical Hygiene Plan..
- 4.2 This method utilizes chemical compounds that are significant health hazards. All possible steps shall be taken to limit analyst contact with these chemicals.
- 4.3 The minimum personal protective equipment (PPE) requirements for performing this test are PVC gloves, safety glasses, and a fully buttoned lab coat. Donning the PPE shall reduce the possibility of contact to a safe level, but the analysts shall not limit themselves to these PPE minimums.

5 Apparatus and Materials

- 5.1 Analytical Balance – Any analytical balance capable of weighing to 0.0001 g. A Mettler AE100 is in use.
- 5.2 Glass Fiber Filters - Gelman A/E VWR catalog #38150-190, or equivalent.
- 5.3 Filter Flask - 1000 mL VWR catalog #29410-993 or 500 mL filter flask VWR catalog #29410-990, or equivalent.
- 5.4 Filter Funnel - magnetic filter funnel from Gelman VWR catalog #78144-812, or equivalent.
- 5.5 Vacuum Pump – VWR catalog #54906-001, or equivalent.
- 5.6 10 mL Class A Microburet – VWR catalog #17491-122, or equivalent.
- 5.7 Buret Stand – VWR catalog #60110-244, or equivalent.

Method: 04-S
Revision: 4
Date: November 22, 2006
Page 6 of 16

- 5.8 Stir Plate – VWR catalog #58941-015, or equivalent.
- 5.9 Stir Bar – 1 in. VWR catalog #58948-230, or other available stir bar that fits the container correctly.
- 5.10 150 mL Glass Beakers – VWR catalog #13910-325, or equivalent.
- 5.11 5 mL Volumetric Pipet – Class A VWR catalog #52967-107, or equivalent.
- 5.12 2 mL Volumetric Pipet – Class A VWR catalog #52967-049, or equivalent.
- 5.13 25 mL Volumetric Pipet – Class A VWR catalog #52967-264, or equivalent.
- 5.14 50 mL Volumetric Pipet – Class A VWR catalog #52967-322, or equivalent.
- 5.15 Transfer Pipet – Polyethylene 7 mL, VWR catalog #14670-103, or equivalent.
- 5.16 Mortar and Pestle – ceramic VWR catalog #50420-289/50420-449, or equivalent.
- 5.17 100 mL Class A Vol. Flask VWR catalog #29619-610, or equivalent.
- 5.18 200 mL Class A Volumetric Flask – VWR catalog #29620-164, or equivalent.

6 Reagents

- 6.1 Reagent water - Reagent water is water in which an interferant is not observed at the analyte of interest. For this purpose, ALSI uses a Filson Water Purification System that provides analyte-free, greater than 16.0 megohm-cm DI water on demand.
- 6.2 Sodium Thiosulfate (0.025 N) - NIST Traceable VWR catalog #RC790032, or equivalent. Store at room temperature and discard at five years or according to the manufacturer's expiration date, if the manufacturer's expiration date is less than five years. A certificate of analysis shall be kept on file.
- 6.3 Iodine Solution (0.025 N) – VWR catalog #VW3206-1, or equivalent. Store at room temperature and discard at five years or according to the manufacturer's expiration date, if the manufacturer's expiration date is less than five years. A certificate of analysis shall be kept on file.
- 6.4 Starch Indicator - VWR catalog #VW3262-1, or equivalent. Store at room temperature and discard at five years or according to the manufacturer's expiration date, if the

Method: 04-S
Revision: 4
Date: November 22, 2006
Page 7 of 16

manufacturer's expiration date is less than five years.

- 6.5 Hydrochloric Acid – VWR catalog #JT9535-33, or equivalent. Store in an appropriate acid storage cabinet. Discard at five years or according to the manufacturer's expiration date, if the manufacturer's expiration date is less than five years.
- 6.6 6 N Hydrochloric Acid Solution – Place approximately 400 mL reagent water into a 1000 mL volumetric flask. Add 500 mL hydrochloric acid, cool, and dilute to volume with reagent water. Store at room temperature for a maximum of 6 months.
- 6.7 Sodium Sulfide – 9-Hydrate – VWR catalog #EM-SX0770-1, or equivalent. Store above the freezing point of water up to 6°C. Discard at five years or according to the manufacturer's expiration date, if the manufacturer's expiration date is less than five years.
- 6.8 1000 mg/L Sulfide Stock Solution – Place a piece of sodium sulfide (Section 6.7) into a mortar and grind it until fine. Weigh out 0.75 grams, place in 100 mL volumetric flask and dilute to volume with reagent water. Prepare fresh daily.
- 6.9 25 mg/L Sulfide Laboratory Control Sample – Dilute 5 mL of the sulfide stock solution (Section 6.8) to volume with reagent water in a 200 mL volumetric flask. Prepare fresh daily.

7 Instrument Calibration

Not applicable.

8 Quality Control

- 8.1 All policies and procedures in the most current revision of the ALSI QA Plan shall be followed when performing this procedure.

8.2 Quality Control Requirements

Quality Control Requirements

(Specific Project Requirements may override these requirements)

Method * Blank	--	Beginning of run, every 10 samples, and end of run.	Less than the reporting limit of 1.0 mg/L	Reanalyze all detectable samples if sufficient volume is available. If not report with a qualifying statement.
Laboratory Control Sample (LCS)	25 mg/L	Beginning of run, every 10 samples, and end of run.	(90-110%)	Do not analyze samples until an acceptable beginning LCS has been obtained. If ending LCS fails, reanalyze samples. If reanalysis is not possible report with a qualifying statement.
Duplicate**	--	1 per 10 samples with a minimum of 1 per batch	RPD \leq 20% or calculated control limits whichever is tighter	Since reanalysis will not be possible, report with a qualifying statement.

* If the method blank concentration is greater than or equal to the reporting limit AND is greater than 1/10 the sample concentration, the source of contamination must be investigated and measures taken to minimize or eliminate the problem and affected samples reanalyzed. If reanalysis is not possible, data shall be reported with a qualifying statement.

** Samples selected for duplicate analysis shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a duplicate analysis may indicate a problem with the sample composition and shall be reported to the client whose sample produced the poor precision.

8.3 Initial Demonstration of Capability

8.3.1 Prepare and analyze four replicate Laboratory Control Samples (Section 6.9). All four results shall be within 10% of the true value or between 22.5 and 27.5 mg/L. If the standards analyzed do not meet this requirement, the DOC shall be repeated before independent analysis of samples begins.

Method: 04-S
Revision: 4
Date: November 22, 2006
Page 9 of 16

8.3.2 Until sufficient data becomes available from within the laboratory, usually a minimum of results from 20 to 30 analyses, the laboratory shall assess performance against the control limits in Section 8.4. When sufficient internal performance data becomes available, develop control limits from the mean percent recovery (R) and standard deviation (S) of the percent recovery. The data is used to establish upper and lower control limits as follows:

Upper Control Limit = $R + 3SR$

Lower Control Limit = $R - 3SR$

On an annual basis control limits will be calculated using the most recent 20 – 30 data points. Ongoing proficiency must be established annually as specified in the QA plan, Technical Training.

8.3.3 Using the data generated in Section 8.3.1, calculate the percent relative standard deviation (%RSD) of the replicate analysis as indicated below. To be acceptable, the %RSD must be less than 10%.

$$\%RSD = \frac{\text{Sample Standard Deviation (S)} \times 100}{\text{Mean Recovered Concentration}}$$

8.3.4 If the standards analyzed do not meet this requirement the DOC shall be repeated before independent analysis of samples is begun.

8.4 Method Detection Limit (MDL) studies must be performed annually. MDLs will be performed over a minimum of three days as specified in 99-MDL, which is derived from the procedures outlined in 40 CFR. MDLs will be processed in the same manner as a field sample. The calculated MDL must be between 10% and 100% of the spike concentration and the recovery of the MDL standards must be between 50 and 150% of their true value, and %RSD values $\leq 20\%$, or repeat the MDL determination.

9 Sample Collection, Preservation and Handling

9.1 Samples shall be collected in clean, chemically resistant, zero headspace glass containers. A minimum of 250 mL is required.

9.2 Sample shall be preserved with 2 mL zinc acetate plus NaOH to pH>9.

9.3 Samples shall be cooled and stored above the freezing point of water up to 6°C. Samples must be analyzed within 7 days of the time of collection.

- 9.4 A minimum of 10% of the samples shall be collected in duplicate to provide adequate volume for QC requirements.

10 Procedure

10.1 Preparation of blank.

- 10.1.1 Add 5.0 mL iodine solution (Section 6.3) to each flask.
- 10.1.2 Add 250 mL reagent water to the flask while keeping the pipet tip below the surface of the iodine.
- 10.1.3 Add 2.0 mL hydrochloric acid solution (Section 6.6) using a Class A pipet or a repeating pipet.
- 10.1.4 If the acid addition does not turn the solution to orange, add another 5.0 mL iodine. The total amount of iodine used must be recorded in the laboratory notebook.
- 10.1.5 Using a transfer pipet, add a few drops of starch indicator (Section 6.4). The solution shall turn dark blue/black.
- 10.1.6 Add a stir bar to the beaker, place on stir plate, and titrate with sodium thiosulfate from black through blue to clear.

10.2 Preparation of standard.

- 10.2.1 Pipet 5.0 mL of iodine into the flask.
- 10.2.2 Place 50 mL prepared 25 mg/L Sulfide Laboratory Control Sample (Section 6.9) into the flask making sure to keep the pipet tip below the surface of the iodine and repeat above procedure (Sections 10.1.3 through 10.1.6).

10.3 Preparation of sample.

NOTE: The zinc acetate preservative creates a precipitate that must be filtered.

- 10.3.1 Place a glass fiber filter onto the filter funnel and slowly filter sample. For easier filtering, pour a majority of the sample through the filter prior to shaking the bottle and disturbing the settled precipitate. Then, shake the bottle and pour the remaining liquid with precipitate through the filter.
- 10.3.2 When all of the sample has been filtered, rinse the sample bottle with reagent

water and add to the filter. Make sure all the precipitate has been removed from the bottle and the bottle has been thoroughly rinsed.

10.3.3 Rinse the sides of filter funnel. The sample on the pad must be very dry.

10.3.4 Add the filter(s) to a beaker that contains 5 mL of iodine solution (Section 6.3) and 50 mL of reagent water.

10.3.5 Repeat above procedure (Sections 10.1.3 through 10.1.6).

11 Calculations

11.1 Blank correction: (Calculated using the initial blank analyzed.)

$$(N \text{ Iodine} \times V \text{ Iodine}) - (N \text{ Sodium Thiosulfate} \times V \text{ Sodium Thiosulfate})$$

11.2 Calculation of samples:

$$\frac{((N \text{ Iodine} \times V \text{ Iodine}) - (N \text{ Sodium Thiosulfate} \times V \text{ Sodium Thiosulfate}) - \text{Blank}) \times 16,000}{\text{mL sample}}$$

The calculation shall be based on the volume of original sample put through the filter.

11.3 Standard recovery is calculated as:

$$\% \text{ Recovery} = C_m / C_n \times 100$$

where: C_m = measured concentration of LCS
 C_n = spiking concentration

11.4 Precision (RPD) is calculated as:

$$\% \text{ RPD} = \frac{|(R_1 - R_2)|}{(R_1 + R_2) / 2} \times 100$$

where: R_1 = sample or spike result
 R_2 = duplicate or spike duplicate result

12 Reporting Results

12.1 All raw data used for reporting results must be dated and initialed by the qualified laboratory personnel performing first and second review.

Method: 04-S
Revision: 4
Date: November 22, 2006
Page 12 of 16

- 12.2 When entering data into Horizon LIMS do not round off results: Horizon will automatically perform rounding appropriate to the method. Horizon LIMS results are reported to three significant figures but limited to the number of decimal places in the reporting limit for the individual compound or analyte.
- 12.3 Report the actual result, even if it is less than the reporting limit. Any sample with a result less than the reporting limit is reported as ND (non-detectable); LIMS will automatically report the appropriate detection limit.
- 12.4 Drinking water samples exceeding the Maximum Contaminant Level (MCL) must be reported to the Customer Service Representative immediately following determination in order to comply with the Pennsylvania Code: Title 25, Chapter 109, Section 109.810 for Reporting and Notification Requirements.
- 12.5 Horizon results entered will be reported on the analytical report exactly as entered by the analyst. No rounding will occur for these entries.

13 Waste Disposal

- 13.1 No specific instructions apply to waste generated using this procedure. For general guidelines, refer to ALSI SOP 19-Waste Disposal.

14 Pollution Prevention

- 14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. Management shall consider pollution prevention a high priority. Extended storage of unused chemicals increases the risk of accidents. The laboratory shall consider smaller quantity purchases which will result in fewer unused chemicals being stored and reduce the potential for exposure by employees. ALSI tracks chemicals when received by recording their receipt in a traceable logbook. Each chemical is then labeled according to required procedures and stored in assigned locations for proper laboratory use.

15 Definitions

- 15.1 Refer to ALSI QA Plan under Laboratory Quality Control Checks for general definitions.

Method: 04-S
Revision: 4
Date: November 22, 2006
Page 13 of 16

16 Troubleshooting

- 16.1 As applicable, refer to maintenance logs and instrument manuals for guidance in troubleshooting specific problems related to the instrumentation used in this method.

Method: 04-S
Revision: 4
Date: November 22, 2006
Page 15 of 16

SOP Change History Summary

<u>Section No.</u>	<u>Section</u>	<u>Reason for Change</u>
1	Scope and Application	NELAC additions
5	Apparatus and Materials	Update to SOP
6	Reagents	NELAC additions
8	Quality Control	Update to SOP
9	Sample Collection...	Update to SOP
10	Procedure	Update to SOP
12	Reporting Results	Update to new LIMS
15	Definitions	NELAC additions
	Appendix A	Added

Revision 4: 11/22/2006

1.4	Scope and Application	Update made to indicate MDL study location
1.7	Scope and Application	Addressed the use of project specific criteria
4.1	Safety	Added availability of MSDS
8.2	Quality Control	Added corrective action for the positive method blank
8.3.2	Quality Control	Added statement of ongoing proficiency
12	Reporting Results	Multiple revisions/additions made throughout section
16	Troubleshooting	Added section
A	Appendix	Updated bench worksheet

Method: 19-COC
Revision: 4
Date: November 22, 2006
Page: 1 of 19

Document Title: Standard Operating Procedure for Chain of Custody Entry

Document Control Number: _____

Organization Title: ANALYTICAL LABORATORY SERVICE, INC. (ALSI)

Address: 34 Dogwood Lane
Middletown, PA 17057

Phone: (717) 944-5541

Approved by:

Helen MacMinn, Date
Quality Assurance Manager

Steve Smith, Date
Sample Receiving

Deborah J. Wood, Date
Validator

Annual Review:

Reviewed By Date Reviewed

Approved By Date Approved

Reviewed By Date Reviewed

Approved By Date Approved

Method: 19-COC
Revision: 4
Date: November 22, 2006
Page: 3 of 19

TABLE OF CONTENTS

1 Scope and Application..... 4
2 Summary of Method..... 4
3 Interferences 4
4 Safety 4
5 Apparatus and Materials 5
6 Reagents 5
7 Instrument Calibration..... 5
8 Quality Control..... 5
9 Sample Collection, Preservation and Handling 5
10 Procedure..... 5
11 Calculations 10
12 Reporting Results 10
13 Waste Disposal 10
14 Pollution Prevention 10
15 Definitions 11
16 Troubleshooting..... 11
Appendix A 12
Appendix B..... 16
SOP Change History Summary 18
SOP Concurrence Form..... 19

Method: 19-COC
Revision: 4
Date: November 22, 2006
Page: 4 of 19

1 Scope and Application

- 1.1 This standard operating procedure addresses the entry of chain-of-custodies for all customers of ALSI. This document shall serve as a guideline to entering the chain-of-custody when the sample(s) are received in the Sample Receiving Department.
- 1.2 This document states the laboratory's policies and procedures established in order to meet requirements of all certifications/accreditations currently held by the laboratory, including the most current NELAC standards.
- 1.3 Individual projects may override criteria listed in this SOP.

2 Summary of Method

- 2.1 Enter your user name and password to access the Horizon LIMS.
- 2.2 From the Samples Menu select Login, then select By Container.
- 2.3 Access the "sample login" section and enter all data pertaining to the individual sample such as date/time sampled and analyses required.

3 Interferences

- 3.1 Not Applicable.

4 Safety

- 4.1 Samples may contain compounds and preservatives that are significant health hazards. All possible steps should be taken to limit analyst contact with these samples. The minimum personal protective equipment requirements when handling samples are PVC gloves, safety glasses, and a fully-buttoned lab coat. This PPE should reduce the possibility of contact to a safe level, but the analyst should not limit themselves to these PPE requirements.
- 4.2 ALSI maintains material safety data sheets (MSDS) on all chemicals used in this procedure. MSDS are available in hard copy in the QA office and electronically on the ALSI network.

Method: 19-COC
Revision: 4
Date: November 22, 2006
Page: 5 of 19

5 Apparatus and Materials

5.1 Laboratory computer station having access to the current version of Horizon; Horizon 9.0 is in use at the time of SOP 19-COC, revision 4.

6 Reagents

6.1 Not Applicable

7 Instrument Calibration

7.1 Not Applicable

8 Quality Control

8.1 All policies and procedures in the most current revision of the ALSI QA Plan shall be followed when performing this procedure.

8.2 An Initial Demonstration of Capability shall be documented as follows:

8.2.1 Completion of the “New Employee Orientation: Sample Receiving Training Checklist: Sample Login.” See Appendix C.

8.2.2 Corrective action for DOC failure:

8.2.2.1 Repeat training shall be performed until all forms are successfully completed.

8.2.2.2 It shall be at the discretion of supervisory staff and management to determine at what point repeat training is no longer applicable and reassignment or company termination is appropriate.

8.3 Ongoing proficiency on an annual basis, as specified in the QA Plan, Technical Training, does not apply to this procedure.

9 Sample Collection, Preservation, and Handling

9.1 Not Applicable

10 Procedure

10.1 Accessing Horizon

10.1.1 Double-click on the Horizon icon on the desktop menu. The Horizon login screen will appear.

Method: 19-COC
Revision: 4
Date: November 22, 2006
Page: 6 of 19

10.1.2 Enter your user name (first initial and last name) and password (password provided to the IT department.) The Horizon main menu will appear.

10.2 Creating Workorders

10.2.1 From the main menu select “Samples”.

10.2.2 Select “Login”.

10.2.2.1 Select “By Container”

10.2.3 From the Client field, click on the LOV (List Of Values) and select the client from the list.

10.2.4 Select the “Profile” field and click on the LOV (List of Values) icon.

10.2.5 Select the “Profile” field and click on the LOV (List of Values) icon.

10.2.6 The list shown will be only the profiles available for the client you have selected. Scroll down the list and pick the profile that best matches the COC you are logging in.

10.2.6.1 Profiles for DEP reportable drinking water samples will be identified by the 7 digit DEP identification number.

10.2.7 Selecting the profile will fill in all client information on the “Customer Login” screen. This information includes the client number and name, the type of deliverable, the report format, the sequence, the status, charges, the earliest due date, and the date created.

10.2.7.1 The type of deliverable can be changed if the COC requests a type different from the listed in the profile. Options under the LOV menu include: CM (commercial w/o deliverable), CT (commercial timeframe invoice), IL (invoicing at large), CQ (commercial QA/QC package), and SD (EPA sample deliverable group).

10.2.7.2 The report format can be changed if the COC requests a type different from that listed in the profile. Options under the LOV menu include: 40CFR (40 CFR report deliverable), SDWA (SDWA deliverable), Standard (standard

Method: 19-COC
Revision: 4
Date: November 22, 2006
Page: 7 of 19

deliverable), and UCMR (UCMR EDD deliverable).

10.2.8 Under “Work ID” field, type in a keyword(s) that will identify that project. These keywords can be identifiers such as “monthly DEP”, weekly wastewater, etc. or they can be project or job numbers provided by a specific client. You can also press enter from this field and the computer will fill in the default workorder ID associated with the profile you have chosen. The workorder ID field must be blank for the default ID to work.

10.2.9 The PO field will be empty at this point. If a purchase order number is available enter it her. Unless the client has a PO that is used consistently, the PO in the “case” can be saved in the “Client Info”. If the PO is stored in the client profile, the PO field will be filled in by the LIMS.

10.2.10 From the Collector field click on the LOV (List Of Values) Icon. A list will appear. Begin typing the last name of the collector, this will begin reducing the list of names making it easier to find the collector you are looking for.

10.3 Sample Login

10.3.1 From the Sample ID field, click on the LOV (List Of Values) Icon. If any valid ID’s are stored in the client profile, they will appear in a list. If no valid ID’s are available, the user can manually type in a Sample ID. Refrain from using abbreviations, all small cases or all upper case.

10.3.2 The “Phone”, “Report to”, “PO”, “Location”, “Description”, “Rec Codes”, “Chain”, “Original”, “Keywords”, and “Paired” fields are empty fields.

10.3.3 Enter the date and time collected.

10.3.4 The “Matrix” field is filled in according to the profile, but can be changed if necessary. A list of matrix options can be seen by accessing the LOV menu for the “Matrix” field.

10.3.5 Enter the date and time received. The date and time received is the date and time the last signature on the COC was completed upon receipt by the sample receiving department.

10.3.6 The “Type” field and “Mgr” fields are filled in according to the profile.

Method: 19-COC
Revision: 4
Date: November 22, 2006
Page: 8 of 19

- 10.3.7 The “Turn” field is filled in according to the profile, but can be changed if necessary. A list of options can be seen by accessing the LOV menu for the “Turn” field. When selecting an option, always pick one expressed as “Workdays from Receipt”.
- 10.3.8 The “Priority” field is filled in according to the profile, but can be changed if necessary. A list of options can be seen by accessing the LOV menu for the “Priority” field.
- 10.3.9 Click on the “Load Line” button from the top of the Horizon Screen. A list of available testing will be displayed associated with the client and profile chosen.
- 10.3.9.1 Line Item 1 for the profile will initially appear on the first line of the “Line Item” section. Click on the “Line Item”
- 10.3.9.2 All the line items available under that profile will appear. Line items can be a single test or a group of several different tests.
- 10.3.9.3 Select a line item that includes tests codes necessary for this sample. Click on “Set Line Item” Unchecking any tests will cause that test or tests not to load in the workorder.
- 10.3.9.4 You can continue to add testing from the list by “appending line items. Appending will add to what has already been loaded into the workorder. Setting A Line will cause everything loaded in the workorder to be replaced. Select the Line Item you wish to append then, click on “Append Line Item” to add these codes to the test codes already selected. Again unchecking any test will cause that test not to load.
- 10.3.9.5 Continue in this manner until all test codes needed are selected.
- 10.3.9.6 Once all test codes are loaded that are needed, click on OK on the Line Items window.
- 10.3.9.7 If a needed test code is not included as a line item, it can be entered manually. In the next unused Type field key in the type of container, or click on the LOV Icon to select from a list.

Method: 19-COC
Revision: 4
Date: November 22, 2006
Page: 9 of 19

- 10.3.9.8 Next key in the preservative or click on the LOV Icon and select the preservative from the list.
- 10.3.9.9 In the CC field key in “OK”
- 10.3.9.10 In the Count field key in the number of containers received.
- 10.3.9.11 Click in the ACODE 1 Field and then click on the LOV Icon and select the test code you are trying to add. You can continue to add additional testing to that bottle by going to the next ACODE to the Right. There are 16 available ACODES on a single line. You more is needed for a single bottle continue on the next line making sure The “Type” field remains blank. When the LIMS sees a blank type field, it automatically associates the testing for that line with the bottle listed above that line.
- 10.3.9.12 Once all test codes are entered, and all bottles are correct, click on “Save”.
- 10.3.9.13 If auxiliary data is required for the sample, such as for DEP reportable samples, the auxiliary data field will appear.
- 10.3.9.14 Auxiliary data information will include: 1) Raw or Finished Water; Level of Compositing (Usually 0); Source (Surface (1), GUDI (2), or Groundwater (3); Start of Sampling Period; and End of Sampling Period.
- 10.3.9.15 Once the Auxiliary Data is entered, click “Save”.
- 10.3.9.16 A “Lab ID” number will be generated, and unique bottle IDs will also be generated for each container, and the sample will be saved, and labels will print.

NOTE: If a second sample is included on the workorder, the LIMS will advance to the next sample. Proceed starting with Section 10.3.1. Information from the first sample will be carried over to the second and can be saved as is or changed. DO NOT changed the Work ID Field. If this field is altered in any way, the LIMS will begin a new workorder.

- 10.3.9.17 To begin a new workorder simply start from Section 10.2.3 and remember that you must changed the Work ID field to clear the

Method: 19-COC
Revision: 4
Date: November 22, 2006
Page: 10 of 19

WO number that the LIMS last assigned. You can also hold down the shift key while pressing F7 to clear the enter Container Login Screen the begin again from section 10.2.3. This is probably the “safest” way until you are comfortable with changing workorders.

10.4 Creating DEP Sample Identifier

- 10.4.1 From the Horizon main menu select the “Clients” icon.
- 10.4.2 Select “Setup/Edit”
- 10.4.3 Select the profile the DEP identified is to be added to.
- 10.4.4 Select “Valid ID” (Double check)
- 10.4.5 Add the new DEP identifier.

11 Calculations

- 11.1 Not Applicable

12 Reporting Results

- 12.1 Not Applicable

13 Waste Disposal

- 13.1 Refer to ALSI SOP 19-Waste Disposal

14 Pollution Prevention

- 14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. Management shall consider pollution prevention a high priority. Extended storage of unused chemicals increases the risk of accidents. The laboratory shall consider smaller quantity purchases which will result in fewer unused chemicals being stored and reduce the potential for exposure by employees. ALSI tracks chemicals when received by recording their receipt in a traceable logbook. Each chemical is then labeled according to required procedures and stored in assigned locations for proper laboratory use.

Method: 19-COC
Revision: 4
Date: November 22, 2006
Page: 11 of 19

15 Definitions

15.1 Refer to ALSI QA Plan under Laboratory Quality Control Checks for general definitions.

16 Troubleshooting

16.1 Refer to the maintenance logs and instrument manuals for guidance in troubleshooting specific problems related to the apparatus used in this method.

Method: 19-COC
Revision: 4
Date: November 22, 2006
Page: 12 of 19

APPENDIX A

From the Sample Menu, select Login, then By Container. This screen will be displayed:

Horizon LIMS - Mr. Steven L Smith

File Clients Samples Workorders Batching Operations Systems Help Window

Load Line Billable Acodes Clear Containers

Container Login

Client Work ID WD
Profile Type Report QA Created 10/06/06 03:06

Collector Phone PO
Report To Location
Chain Delivered From
Released To Smith, Mr. Steven L Released By

Sample ID Collected Matrix Type SAMPLE
Turn Received 10/06/06 03:06 Comments Lab
Lab ID Desc Rec Code Aux Keywords
Patient Priority Mgr Original Paired Haz
SSN Folder Carrier Airbill Reason

Container ID	Type	Preservative	CC	Count	Original Volume	pH	Temp	Acode 1	Acode 2	Acode 3

Record: 1/1 List of Values <OSC <DBG

Method: 19-COC
Revision: 4
Date: November 22, 2006
Page: 13 of 19

In the Client Field, Click on the List Of Values button. The client for the workorder will be displayed here. The login screen will look similar to this and the following fields will be required for the user to fill in:

Horizon LIMS - Mr. Steven L. Smith

File Clients Samples Workorders Batching Operations Systems Help Window

Load Line Billable Acodes Clear Containers

Container Login

Client: TEST1 Test 1 Customer Work ID: WD

Profile: 7653 TESTSV ACODE FOR TESTING Type: CM Report: STANDARD QA: Created:

Collector: Phone: PO:

Report To: Location:

Chain: Delivered From:

Released To: Smith, Mr. Steven L. Released By:

Sample ID: Collected: Matrix: GW Type: SAMPLE

Turn: W010 10 Work days from receipt Received: 10/06/06 03:06 Comments: Lab:

Lab ID: Desc: Rec Code: Aux: Keywords:

Patient: Priority: 5 Mgr: JER Original: Paired: Haz: N

SSN: Folder: Carrier: Airbill: Reason:

Container ID	Type	Preservative	CC	Count	Original	Volume	pH	Temp	Acode 1	Acode 2	Acode 3
	A	UNP	OK	2					TESTSV		

Record: 1/1 List of Values <OSC> <DBG>

Method: 19-COC
Revision: 4
Date: November 22, 2006
Page: 14 of 19

When the user is ready to add testing, simply click on the Load Line button. All available testing for the client and profile will be displayed. A window similar to this will be displayed:

The screenshot shows the Horizon LIMS software interface. The main window is titled 'Container Login' and contains several input fields for client information, including Client (TEST1), Profile (7653), Collector (8975), and Work ID (SOP Test). A 'Load Line' button is visible in the top right of the main window. Overlaid on this is a 'Profile Line Items' dialog box with a table of test items.

Item Description	Mx	Turn	Aux	Crit		
1 TESTSV		GwW010		1	<input checked="" type="checkbox"/>	TESTSV Test Acode for SV
2 GRO	S	W010		1	<input type="checkbox"/>	
3 FLASH POINT	0	W010		1	<input type="checkbox"/>	
4 SPECIFIC GRAVITY	0	W010		1	<input type="checkbox"/>	
5 FP AND SP GR	0	W010		1	<input type="checkbox"/>	
6 additional GW matrix tests		GwW010		1	<input type="checkbox"/>	

From the Profile Line Items window, select the line item that best matches the COC being worked on. If a line item contains more testing than what is requested, simply uncheck what isn't to be added to the workorder. Click on the Set Line Item button. If additional line items are to be appended, simply click on the line item to be appended, uncheck any testing not needed to be added and click Append to Line Item.

NOTE: Clicking on the Set Line Item Button replaces everything.

Method: 19-COC
Revision: 4
Date: November 22, 2006
Page: 16 of 19

APPENDIX B

**New Employee Orientation
Sample Receiving Training Checklist**

Procedure: Sample Log-in

SOP: _____

Revision: _____

	Analyst	Trainer
1) Analyst has read and understands SOP 19-COC and signed the concurrence form.	_____	_____
2) Analyst can access the Horizon LIMS.	_____	_____
3) Analyst can create a new workorder.	_____	_____
3) Analyst knows how to select a client profile.	_____	_____
4) Analyst knows how to change deliverable type and report format if necessary.	_____	_____
5) Analyst knows how to access the sample log-in screen.	_____	_____
6) Analyst knows how to select a sample collector.	_____	_____
7) Analyst knows how to select a sample ID, including an ID for a DEP reportable sample.	_____	_____
8) Analyst knows how to enter the date/time collected and received.	_____	_____
9) Analyst knows how to change the sample matrix if necessary.	_____	_____
10) Analyst knows how to modify the priority and turn-around fields if necessary.	_____	_____
12) Analyst knows how to add test codes using the "set line item" function.	_____	_____
13) Analyst knows how to append line items.	_____	_____

This document is the property of Analytical Laboratory Services, Inc. It may be used by the recipient only for the purpose for which it was transmitted. It is submitted in confidence and its disclosure to you is not intended to constitute public disclosure or authorization for disclosure to other parties. It may not be copied or communicated without the written consent of Analytical Laboratory Services, Inc.

Rev. 5/05

Method: 19-COC
Revision: 4
Date: November 22, 2006
Page: 17 of 19

	Analyst	Trainer
14) Analyst knows how to add test codes manually that are not included in line items.	_____	_____
15) Analyst knows how to enter auxiliary data.	_____	_____
16) Analyst knows how to create DEP sample identifiers.	_____	_____
17) Analyst demonstrated this ability on at least five separate projects/workorders, one of which is a DEP sample.	_____	_____

The workorders used for demonstration are:

Analyst Name Analyst Signature Date

Supervisor Name Supervisor Signature Date

This document is the property of Analytical Laboratory Services, Inc. It may be used by the recipient only for the purpose for which it was transmitted. It is submitted in confidence and its disclosure to you is not intended to constitute public disclosure or authorization for disclosure to other parties. It may not be copied or communicated without the written consent of Analytical Laboratory Services, Inc.

Rev. 5/05

Method: 19-COC
Revision: 4
Date: November 22, 2006
Page: 18 of 19

SOP Change Summary

<u>Section No.</u>	<u>Section</u>	<u>Reason for Change</u>
<u>Revision 4: 11/22/2006:</u>		
1.2	Scope and Application	Added references to the most current NELAC standards
1.3	Scope and Application	Added use of project specific criteria
4.1	Safety	Added statement about maintaining MSDS
5.1	Apparatus and Materials	Revised reference to a specific Horizon version to read "current"
8.1	Quality Control	Added standard verbiage concerning ALSI QA Plan
8.2	Quality Control	Added DOC requirements
8.2.2	Quality Control	Added DOC failure corrective action
8.3	Quality Control	Added comment concerning ongoing proficiency
16	Troubleshooting	Added section
A	Appendix	Updated appendix
B	Appendix	Deleted screenshots; added training documentation

Method: 19-Disp
Revision: 0
Date: July 19, 2000
Page: 1 of 6

Document Title: Standard Operating Procedure for Disposal of Samples, Extracts, Digestates, and Leachates

Document Control Number: _____

Organization Title: ANALYTICAL LABORATORY SERVICES, INC. (ALSI)

Address: 34 Dogwood Lane
Middletown, PA 17057

Phone: (717) 944-5541

Approved by:

Susan Magness,
Quality Assurance Manager

Date

Ray Martrano,
Laboratory Manager

Date

Method: 19-Disp
Revision: 0
Date: July 19, 2000
Page: 2 of 6

TABLE OF CONTENTS

1 Scope and Application 3

2 Summary of Method..... 3

3 Interferences 3

4 Safety 3

5 Apparatus and Materials..... 3

6 Reagents 4

7 Instrument Calibration..... 4

8 Quality Control..... 4

9 Sample Collection, Preservation and Handling 4

10 Procedure..... 4

SOP Concurrence Form 16

1 Scope and Application

Method: 19-Disp
Revision: 0
Date: July 19, 2000
Page: 3 of 6

1.1 The purpose of this Standard Operating Procedure (SOP) is to document the procedures for disposing of all samples, extracts, leachates, and digestates.

2 Summary of Method

2.1 Samples are mainly disposed of by the Sample Receiving Department, but all departments dispose of waste in some form or another.

2.2 There are four categories of waste for disposal

2.2.1 Non-hazardous aqueous waste (which can be samples, extracts, leachates, or digestates)

2.2.2 Hazardous aqueous waste (which can be samples, extracts, leachates, or digestates)

2.2.3 Non-hazardous non-aqueous waste (which can be samples, extracts, leachates, or digestates)

2.2.4 Hazardous non-aqueous waste (which can be samples, extracts, leachates, or digestates)

3 Interferences

3.1 Not applicable.

4 Safety

4.1 The minimum personal protective equipment requirements are safety glasses, a fully buttoned lab coat, and PVC gloves. Tyvek suits, dust masks, or respirators may be required depending on the waste being handled (hazardous waste).

5 Apparatus and Materials

5.1 pH monitoring system

5.2 55 gallon drums

Method: 19-Disp
Revision: 0
Date: July 19, 2000
Page: 4 of 6

6 Reagents

6.1 Not applicable.

7 Instrument Calibration

7.1 Not applicable.

8 Quality Control

8.1 Not applicable.

9 Sample Collection, Preservation and Handling

9.1 Not applicable.

10 Procedure

10.1 As noted in the ALSI SOP 19-Rec/Han, Section 10.8, samples are stored at least two weeks following completion of the last analysis on the lab report, with the exception of VOA vials and microbiology samples. Samples requiring special storage considerations, such as USACE samples requiring 6 month storage, are handled project by project. This also applies to extracts, leachates, and digestates (from this point on to be noted as waste in this SOP).

10.2 Prior to any disposal, a Sample Discard Report is printed from the ALSI LIM system. The date used for disposal is two weeks after the disposal date. Data from the LIMS (laboratory reports) are also used to determine if the waste is hazardous or non-hazardous.

10.3 The non-hazardous aqueous waste is disposed of using pre-approved drains. The waste created by this disposal goes into a pH monitoring system. The system adjusts the pH level to within the limits allowable by our local POTW. After this adjustment, the waste is disposed into the local septic system.

10.4 The non-hazardous non-aqueous waste is disposed of by utilizing the 55-gallon drums. This waste is dumped into these containers. A pre-determined disposal company disposes the 55-gallon drums.

Method: 19-Disp
Revision: 0
Date: July 19, 2000
Page: 5 of 6

- 10.5 The hazardous aqueous waste is disposed of by utilizing the 55-gallon drums. This waste is dumped into these containers. A predetermined disposal company disposes of the 55-gallon drums. The individual departments dispose of the majority of this waste as it is created.
- 10.6 The hazardous non-aqueous waste is disposed of by utilizing the 55-gallon drums. This waste is dumped into these containers. A predetermined disposal company then disposes of the 55-gallon drums.
- 10.7 All waste is disposed of in accordance with all federal, state, and local regulations.

Method: 19-Disp
Revision: 0
Date: July 19, 2000
Page: 6 of 6

SOP Concurrence Form
for the Distribution and Revision of Standard Operating Procedures

I have read, understood, and concurred with the Standard Operating Procedure (SOP) described above and will perform this procedure as it is written in the SOP.

Print Name	Signature	Date
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____
_____	_____	_____

Method: 19-Rec/Han

Revision: 11

Date: February 6, 2007

Page: 1 of 36

Document Title: Standard Operating Procedure for Sample Receipt/Sample Handling

Document Control Number: _____

Organization Title: ANALYTICAL LABORATORY SERVICE, INC. (ALSI)
Address: 34 Dogwood Lane
Middletown, PA 17057

Phone: (717) 944-5541

Approved by: _____
Helen MacMinn, Date
Quality Assurance Manager

Anna Milliken, Date
Laboratory Manager

Deborah J. Wood, Date
Validator

Annual Review:

Reviewed By Date Reviewed

Approved By Date Approved

Reviewed By Date Reviewed

Approved By Date Approved

Method: 19-Rec/Han

Revision: 11

Date: February 6, 2007

Page: 2 of 36

Annual Review (continued):

Reviewed By

Date Reviewed

Approved By

Date Approved

Reviewed By

Date Reviewed

Approved By

Date Approved

Reviewed By

Date Reviewed

Approved By

Date Approved

Reviewed By

Date Reviewed

Approved By

Date Approved

Reviewed By

Date Reviewed

Approved By

Date Approved

TABLE OF CONTENTS

1 Scope and Application4

2 Summary of Method4

3 Interferences.....4

4 Safety...4

5 Apparatus and Materials5

6 Reagents.....6

7 Instrument Calibration9

8 Quality Control9

9 Sample Collection, Preservation and Handling11

10 Procedure11

11 Calculations18

12 Reporting Results.....18

13 Waste Disposal.....18

14 Pollution Prevention19

15 Definitions19

16 Troubleshooting19

Appendix A.....20

Appendix B21

Appendix C22

Appendix D23

Appendix E25

Appendix F.....31

Appendix G.....32

SOP Change Summary33

SOP Concurrence Form36

1 Scope and Application

- 1.1 The purpose of this Standard Operating Procedure (SOP) is to demonstrate the procedures for receiving and handling of all samples coming into the laboratory.
- 1.2 This document states the laboratory's policies and procedures established in order to meet the requirements of all certifications/accreditations currently held by the laboratory, including the most current NELAC standards.
- 1.3 Individual project requirements may override criteria listed in this SOP.

2 Summary of Method

- 2.1 The sample receiving area is staffed Monday through Friday from 08:00 to 22:00. Samples are received from a variety of sources including ALSI field services personnel, walk-in clients, client's field personnel, and shipping carriers.
- 2.2 Upon receipt of samples, sample receiving personnel complete the appropriate documentation which may include, but not be limited to, the signing of Chain of Custody forms and the completion of DC-2 Forms (EPA projects only.)
- 2.3 Sample receiving personnel also check for any non-conformances regarding the condition of the samples upon receipt. These non-conformances may include but not be limited to, incorrect containers, preservatives, or packaging, unacceptable sample temperatures, expired holding times, and incomplete or inaccurate documentation.
- 2.4 Weekend receipt of samples is discussed in Section 10.15.

3 Interferences

- 3.1 Not applicable to this method.

4 Safety

- 4.1 ALSI maintains material safety data sheets (MSDS) on all chemicals used in this procedure. MSDS are available to all staff and are located in the QA office.
- 4.2 Incoming samples may contain a variety of unknown and potentially serious safety hazards associated with both the samples themselves and the preservatives added to them and as such, shall be treated with the utmost caution.

- 4.3 The minimum personal protective equipment requirements are safety glasses, a fully buttoned lab coat, and PVC gloves.
- 4.4 Incoming coolers will be opened in or next to a ventilation hood when sample conditions or accompanying information dictate.

5 Apparatus and Materials

- 5.1 pH paper (1-6 range), purchased from VWR catalog #66777-027, or equivalent
- 5.2 pH paper (9-14 range), purchased from VWR catalog #34175-499, or equivalent.
- 5.3 5-3/4" Pasteur pipettes, purchased from VWR catalog #14672-200, or equivalent.
- 5.4 Potassium iodide starch paper purchased from VWR catalog #60799-008, or equivalent.
- 5.5 Total chlorine paper, purchased from Fisher Scientific catalog #3108T34, or equivalent.
- 5.6 Thermometers, capable of measuring to 1°C in the range which includes -10°C to 40°C, purchased from VWR catalog #61222-504, or equivalent. Thermometers are calibrated annually by a representative of the QA department. Thermometer information, including correction factors, is maintained in the temperature logbook.
- 5.7 Calibrated infrared temperature gun, capable of measuring to 1°C; HB Infrared Thermometer 900LS, purchased from VWR, or equivalent. The infrared thermometer is calibrated quarterly by a representative of the QA department. Thermometer information, including correction factors, is maintained in the temperature logbook.
- 5.8 Refrigerators. Each refrigerator used for storage of samples is maintained above the freezing point of water up to 6°C. Temperatures are monitored 7 days a week and are recorded in the temperature logbook after the correction factor is applied. The refrigerators include:
 - 5.8.1 Walk-in Refrigerator by AmeriKooler, Inc. with a Winland Electronics, Inc. temperature alarm located on upper level.
 - 5.8.2 Walk-in Refrigerator by Bally, Serial #DX9101996-01 located on lower level.

- 5.8.3 Walk-in Refrigerator by Harford, Model DL3676W487-V located on upper level
- 5.8.4 Refrigerator #7 - Gibson Market Master located in the GC lab.
- 5.8.5 Refrigerator #10 - Gibson Market Master located in the GCMS lab.
- 5.8.6 Refrigerator #18 – Admiral. (Used for microbiology samples only.)
- 5.8.7 Refrigerator #26 – Kenmore located in the GC/MS lab.
- 5.8.8 Refrigerator #27 – General Electric. (Used for holding volatile samples in sample Receiving before distribution to departments.)

6 Reagents

- 6.1 Reagent water provided by a Filson water purification system, which generates analyte-free greater than 16.0 megohm water on demand, used for the preparation of reagents.
- 6.2 Distilled reagent water as provided by the Corning Mega-Pure system, MP-3A and Barnstead, Fistream II, in the Wet Chemistry department, used in non-volatile trip blank preparation.
- 6.3 GC/MS reagent water used in volatile trip blank preparation.
- 6.4 Preservatives are prepared as follows:

Note: Sample matrix and buffering capacity determine, to a large extent, the effectiveness of the added preservatives. Therefore, each appropriate bottle is pH checked upon receipt at the laboratory to insure that the preservative was adequate. All preservatives are kept at room temperature.

- 6.4.1 Concentrated Sulfuric acid - Sulfuric acid is purchased from VWR item #JT9681-33 or equivalent. Sulfuric acid has an expiration date of five years after receipt, and is stored in an acid chemical storage cabinet. The dispenser top is set to dispense 2 mL. For a 250 mL bottle, 1 squirt is used. For a 500 mL bottle, 2 squirts are used. For a 1000 mL bottle, 4 squirts are used.

- 6.4.2 Nitric Acid (1:1) – Nitric acid is prepared by the Prep department as a 1:1 dilution and recorded in the reagent preparation logbook. The concentrated nitric acid is purchased from VWR, item #JT9598-34 or equivalent. The concentrated nitric acid has an expiration date of five years after receipt, and is stored in an acid storage cabinet. The 1:1 nitric acid is prepared by adding 500 mL of concentrated nitric acid to 500 mL of reagent water in a fume hood. The 1:1 nitric acid has an expiration date of six months after preparation and is stored in the Sample Receiving department. The dispenser top is set to dispense 2 mL. For a 250 mL bottle, 1 squirt is used. For a 500 mL bottle, 2 squirts are used. For a 1000 mL bottle, 4 squirts are used.
- 6.4.3 Hydrochloric acid (1:1) – Hydrochloric acid (1:1) can be purchased directly from the manufacturer or prepared by the Prep department from concentrated hydrochloric acid.
- 6.4.3.1 The manufacturer prepared hydrochloric acid (1:1) is purchased from VWR, item #MKH16807 or equivalent. This hydrochloric acid has an expiration date of five years after receipt, and is purchased on an as needed basis.
- 6.4.3.2 The hydrochloric acid prepared by the Prep department is also a 1:1 dilution. Concentrated hydrochloric acid is purchased from VWR, item #MK261246 or equivalent. The concentrated hydrochloric acid has an expiration date of five years after receipt, and is stored in an acid storage cabinet. The 1:1 dilution hydrochloric acid is prepared by adding 500 mL of concentrated hydrochloric acid to 500 mL of DI water in a fume hood. The 1:1 hydrochloric acid has an expiration date of six months after preparation and is stored in the Sample Receiving department.
- 6.4.3.3 For EPA method 525.2, 3 mL is used for a 1000 mL bottle. For TOCs (125 mL bottle), 1 drop is used.
- 6.4.4 Zinc Acetate Reagent – The zinc acetate reagent is prepared by the Wet Chemistry department, and recorded in the reagent preparation logbook. Zinc acetate is purchased from VWR, item #JT4296-1 or equivalent. The zinc acetate has an expiration date of five years after receipt, and is stored in the wet chemistry department. The zinc acetate reagent used in sample receiving is prepared by diluting 55 grams of zinc acetate in 250 mL of reagent water. The prepared zinc acetate has an expiration date of six months after preparation, and is stored in the Sample Receiving department. For a 250 mL zero headspace bottle, 4 mL of zinc acetate is

added.

- 6.4.5 10 N Sodium hydroxide – 10 N sodium hydroxide is prepared by the prep department, and recorded in the reagent preparation logbook. Sodium hydroxide pellets are purchased from VWR, item #JT3722-7 or equivalent. The sodium hydroxide has an expiration date of five years after receipt, and is stored in the prep department. The 10 N sodium hydroxide used in sample receiving is prepared by diluting 400 grams of sodium hydroxide in 1000 mL of reagent water. The prepared sodium hydroxide has an expiration date of six months after preparation, and is stored in the Sample Receiving department. For a 500 mL bottle, 4 mL of sodium hydroxide is added.
- 6.4.6 EDTA Reagent – EDTA reagent is prepared by the Wet Chemistry department, and recorded in the reagent preparation logbook. Solid disodium EDTA is purchased from VWR, item #JT8993-1 or equivalent. The disodium EDTA has an expiration date of five years after receipt, and is stored in the Wet Chemistry department. The EDTA used in sample receiving is prepared by diluting 5 grams of disodium EDTA in 200 mL of DI water. The prepared EDTA has an expiration date of six months after preparation, and is stored in the Sample Receiving department. For a 250 mL bottle, 2.5 mL of EDTA is added.
- 6.4.7 Sodium thiosulfate – Sodium thiosulfate is used directly from the manufacturer. Sodium thiosulfate is purchased from VWR, item #JT3954-1 or equivalent. Sodium thiosulfate has an expiration date of five years after receipt, and is stored in the Sample Receiving department. Refer to the current revision of the QA Plan, Appendix B-Container, Preservation, Storage, and Holding Time, for guidance in using sodium thiosulfate as a dechlorinating agent for organic samples.
- 6.4.8 Sodium sulfite – Sodium sulfite is used directly from the manufacturer. Sodium sulfite is purchased from VWR item #JT3722-1 or equivalent. Sodium sulfite has an expiration date of five years after receipt, and is stored in the Sample Receiving department. Refer to the Refer to the current revision of the QA Plan, Appendix B-Container, Preservation, Storage, and Holding Time, for guidance in using sodium thiosulfate as a dechlorinating agent for organic samples.
- 6.4.9 Ascorbic acid – Ascorbic acid is used directly from the manufacturer. Ascorbic acid is purchased from VWR, item #JT0938-7 or equivalent. Ascorbic acid has an expiration date of five years after receipt, and is stored in the Sample Receiving department. For a 40 mL vial, 25 mg of

ascorbic is used for organic samples containing residual chlorine.

6.4.10 Ethylenediamine (EDA) Solution – EDA solution is prepared by the wet chemistry department. EDA is purchased from Aldrich, catalog # 24,072-9 or equivalent. The EDA has an expiration date of five years after receipt, or sooner if specified by the manufacturer. The EDA solution used in sample receiving is prepared by diluting 5.6 mL of EDA to 50 mL with reagent water. The prepared EDA has an expiration period of 30 days and is stored in the wet chemistry department.

7 Instrument Calibration

- 7.1 IR Thermometer Gun. The IR thermometer will be calibrated on a quarterly basis by the QA/QC department. Records of calibrations will be retained in the QA/QC department and the correction factors will be listed on the IR thermometer for use by the sample receiving department. The IR thermometer shall be calibrated at 0°C, 4°C, and 10°C. The 0°C correction factor shall be applied to all measurements less than 2°C. The 4°C correction factor shall be applied to all measurements between 2°C and 6°C. The 10°C correction factor shall be applied to all measurements above 6°C. The recorded measurement shall have the correction factor applied.
- 7.2 Thermometers. All other thermometers used for sample temperature measurement will be calibrated on an annual basis by the QA/QC department. Records of calibrations will be retained in the QA/QC department and the correction factors will be listed on a label affixed to the thermometer for use by the Sample Receiving department. The thermometer shall be calibrated at 0°C, 4°C, and 10 °C. The 0°C correction factor shall be applied to all measurements less than 2°C. The 4°C correction factor shall be applied to all measurements between 2°C and 6°C. The 10°C correction factor shall be applied to all measurements above 6°C. The recorded measurement shall have the correction factor applied.

8 Quality Control

- 8.1 All policies and procedures in the most current revision of the ALSI QA Plan shall be followed when performing this procedure.
- 8.2 It is the responsibility of Sample Receiving personnel to check for and document any non-conformances regarding the condition of the samples upon receipt. These non-conformances may include but not be limited to, incorrect containers, preservatives, or packaging, unacceptable sample temperatures, expired holding times, and incomplete or inaccurate documentation. These non-conformances shall be recorded on the COC and communicated to the appropriate customer

Service Representative so that the client can be notified. Any non-conformances associated with sample preservation shall also be recorded in the sample preservation logbook (Appendix A).

8.3 ALSI reserves the right to reject a sample upon receipt in the laboratory if any of the following conditions occur:

8.3.1 The sample is not properly identified on the sample label and/or the Chain-of-custody form.

8.3.2 The sample has exceeded the holding time for the requested analysis.

8.3.3 The incorrect preservative was used during sample collection.

8.3.4 Incorrect sampling protocols were used during sampling.

8.3.5 Improper sample container was used.

8.3.6 Insufficient sample is present to perform the requested analysis.

8.3.7 Improper storage or transport of sample has occurred prior to receipt.

8.3.8 Excessive amount of sample has been collected or other conditions exist which would make disposal difficult.

8.3.9 Excessive air bubbles are present in samples requiring zero headspace.

8.4 An Initial Demonstration of Capability shall be documented as follows:

8.4.1 Completion of the “New Employee Orientation: Sample Receiving Training Checklist: Checking Sample Preservatives.” See Appendix E.

8.4.2 Completion of the “New Employee Orientation: Sample Receiving Training Checklist: Measuring Cooler Temperatures.” See Appendix E.

8.4.3 Completion of the “New Employee Orientation: Sample Receiving Training Checklist: Receiving Samples.” See Appendix E.

8.4.4 Reading, understanding and acceptance of the contents of this SOP shall be documented through the signing of the 19-Rec/Han Concurrence form.

8.4.5 Corrective action for DOC failure:

8.4.5.1 Repeat training shall be performed until all forms are successfully completed.

8.4.5.2 It shall be at the discretion of supervisory staff and management to determine at what point repeat training is no longer applicable and reassignment or company termination is appropriate.

8.5 Ongoing proficiency on an annual basis, as specified in the QA Plan, Technical Training, does not apply to this procedure.

9 Sample Collection, Preservation and Handling

9.1 Not applicable.

10 Procedure

10.1 The Sample Receiving area is staffed Monday through Friday from 08:00 to 22:00. Samples are received from a variety of sources including ALSI field services personnel, walk-in clients, client's field personnel, and shipping carriers.

10.1.1 Samples received from a carrier (Fed-Ex, UPS, etc.) shall be accepted through a signature by a laboratory employee upon receipt at the facility and taken to the Sample Receiving area.

10.1.2 ALSI field personnel shall deliver all samples to the Sample Receiving area.

10.1.3 Walk-in clients and other client's field personnel shall be directed to the Sample Receiving area to deliver their samples.

10.2 All samples shall be accompanied by a Chain of Custody. An example of ALSI's Chain of Custody is included in Appendix C. All information required in the gray shaded area of the ALSI Chain shall be filled in by the client/sampler. If the sample is accompanied by a Chain of Custody from another source, the following information shall be included: client information (name, address, contact person, and phone number), sample(s) description, date/time sampled, analyses requested, date/time results required, PWSID information (if applicable), and any special deliverable or handling instructions (if applicable). The Chain of Custody shall also be signed by each individual having possession of the samples since collection (both when they receive the sample and when they relinquish it). A representative of the sample receiving department shall sign the Chain of Custody when the samples are received at the laboratory. If a walk-in customer does not have a Chain of Custody, one must be completed before samples will be accepted.

- 10.2.1 If a Chain of Custody references microbiological samples that require immediate attention due to holding time restrictions, the Chain of Custody may also be signed by a representative of the microbiology department. In signing the Chain of Custody, the representative from the microbiology department shall follow the same procedures as sample receiving personnel.
- 10.3 The condition of the samples at the time of receipt must be documented.
- 10.3.1 If the sample is from an EPA project, a DC-2 form (Appendix D) must be completed to document the condition of the samples at the time of receipt.
- 10.3.2 Any other checklist or cooler receipt form provided by a client must be completed in its entirety.
- 10.3.3 The condition of all other samples at the time of receipt shall be documented in the receipt information section of the ALSI Chain of Custody. If the Chain of Custody is from another source other than ALSI, a receipt information label shall be affixed to the Chain to record this information.
- 10.3.4 If the project requires an internal Chain of Custody, this procedure must be initiated by the Sample Receiving Department at login. Refer to the Legal Chain of Custody SOP (99-LCOC) for details.
- 10.3.5 The appropriate customer service representative shall be notified of non-conformances so that the client can be contacted and a decision made whether to run the sample as received or to resample.
- 10.4 The temperature of the cooler/container must be taken at the time of sample receipt.
- 10.4.1 If a temperature blank is provided with the cooler, a calibrated mercury thermometer shall be used to determine the cooler temperature. The correction factor as discussed in Section 7.2 shall be applied to the measurement and the corrected temperature along with the thermometer ID shall be recorded on the Chain of Custody in the receipt information section.
- 10.4.2 If a temperature blank is not provided, the temperature of the sample is taken with a calibrated IR gun held no more than one inch away from the sample bottle or temperature blank. The reading must be taken directly

through the side of the bottle. It shall not be taken through the lid or through any label, etc., adhered to the bottle. The correction factor as discussed in Section 7.1 shall be applied to the measurement and the corrected temperature along with the thermometer ID shall be recorded on the Chain of Custody in the receipt information section.

10.4.3 If this protocol is not feasible, a small amount of sample shall be decanted from a non-volatile bottle and a calibrated thermometer shall be used to read the temperature of the sample as discussed in Section 10.4.1.

10.4.4 If the temperature reading does not fall within the acceptable range (above the freezing point of water up to 6°C), multiple readings shall be taken using at least two other bottles contained in the same cooler to insure that a representative reading has been obtained. All readings shall then be recorded along with the identification of the bottles from which they were taken.

10.4.5 Samples that are hand delivered to the laboratory on the same day that they are collected may not meet these criteria. In these cases, the samples shall be considered acceptable if there is evidence that the chilling process has begun such as arrival on ice.

10.4.6 All samples shall be observed for receipt on ice. The observation shall be documented by checking the appropriate Y/N box in the receipt information section of the Chain of Custody. The individual noting this fact shall also initial the corresponding line.

10.4.7 Samples shall remain on ice or be transferred to an appropriate refrigerator until processing. Microbiological samples shall be stored in Refrigerator 18 and volatile samples shall be stored in Refrigerator 27 to prevent contamination until transport to the appropriate departments.

10.5 All samples shall be checked for custody seals. The presence of custody seals shall be noted on the Chain of Custody by checking the appropriate Y/N box in the receipt information section of the Chain and initialing the corresponding line. If custody seals are present, the presence or absence of tampering shall be documented by checking the appropriate Y/N box in the receipt information section of the Chain and initialing the corresponding line. Details involving any tampering with custody seals shall be noted in the sample/COC comments section of the Chain.

Method: 19-Rec/Han

Revision: 11

Date: February 6, 2007

Page: 14 of 36

- 10.6 All samples shall be checked for breakage/leakage. The presence or absence of damage shall be noted by checking the appropriate Y/N box under "Cont. in Good Cond." in the receipt information section of the Chain and initialing the corresponding line. Details involving any breakage shall be noted in the sample/COC comments section of the Chain.
- 10.7 All samples shall be checked to insure that the information on the Chain of Custody agrees with the information on the container labels. This information would include sample descriptions, date/time sampled, sampler ID, and analyses requested. Whether or not the Chain of Custody and labels agree shall be documented by checking the appropriate Y/N box in the receipt information section of the Chain and initialing the corresponding line. Any discrepancies between the Chain and labels shall be specifically mentioned in the sample/COC comments section of the Chain.
- 10.8 All samples shall be checked to insure that the containers received are appropriate for the analyses requested and that a sufficient sample volume has been provided. This information shall be documented by checking the appropriate Y/N boxes under "Correct Containers" and "Correct Sample Volumes" in the receipt information section of the Chain and initialing the corresponding line. Any non-conformances shall be specifically mentioned in the sample/COC comments section of the Chain.
- 10.9 Samples contained in a septa-sealed container to be analyzed for volatile analytes, shall be checked for the appearance of headspace. Headspace is the appearance of air in a septum-lined container. A pea sized amount of air or smaller is allowable. Whether or not headspace is present shall be noted by checking the appropriate Y/N box in the receipt information section of the Chain and initialing the corresponding line. Any non-conformances shall be specifically mentioned in the sample/COC comments section of the Chain.

All volatile samples shall be held in the Sample Receiving refrigerator designated for VOC only (Refrigerator 27) and protected from contamination until the COC is entered into the LIMS system and the identifying labels are attached to each vial. The samples will then be transported to the VOC refrigerators in the organics lab for analysis.

- 10.10 The pH of all acid or caustic preserved bottles (with the exception of volatile containers) shall be taken using the pH paper listed in Section 5. Bottles shall be preserved according to the SOP for Bottle Preservation (19-BOTTLE). (See the most recent revision of the QA Manual for a list of analyses that require pH testing.)

- 10.10.1 The pH of the samples is measured by inserting a 5-3/4" Pasteur pipette into the sample. After the pipette is drawn out, a drop of the sample is placed on the pH paper. The pH is read using the color charts on each package of the pH strips. If the pH falls within the correct range, it is recorded on the lid of the sample container using a permanent waterproof marker and in the sample preservation logbook. (See Appendix A).
- 10.10.2 If the pH is not within the acceptable range, the pH is adjusted by adding the appropriate preservative (see SOP for bottle preservation). The initial pH, the amount of preservative added, the lot number of the preservative used, and the final pH are recorded on the lid of the sample container using a permanent waterproof marker and in the sample preservation logbook.
- 10.10.3 For samples requiring **DoD** certification, a small amount of sample shall be poured out of each bottle (excluding volatile samples) to check for the appropriate pH.
- 10.10.4 The following information must be recorded in the sample preservation logbook: the sample number, date received, preservative, initial pH, and the initials of the person taking the pH. In cases when the pH was adjusted, the amount of preservative added and the final pH are also recorded along with the lot of the preservative added. The logbook is generated in a Microsoft Excel spreadsheet that is protected against making any changes to the posted results. Each bottle is scanned using a Symbol Pocket PC with Pocket Excel installed.
- 10.10.5 Whether or not samples are preserved properly shall be documented by checking the appropriate Y/N box in the receipt information section of the Chain and initialing the corresponding line. Any non-conformances shall be specifically mentioned in the sample/COC comments section of the Chain.
- 10.10.6 If a metals bottle was acidified upon receipt at the laboratory, a copy of the preservation log shall be attached to the bottle with a rubber band when it is delivered to the department. This will notify the Metals Department of the date and time of preservation.
- 10.11 All samples received must be checked for holding times.
- 10.11.1 Particular attention shall be given to any microbiological samples

received, especially fecal coliforms which have a holding time of 6 hours for wastewater samples and frequently arrive at the laboratory with little time remaining prior to expiration. A representative of the microbiology department shall be notified immediately upon the arrival of these samples.

10.11.2 The presence of any other short holding time tests (less than 48 hours) that require immediate attention shall also be noted. If any short holding time tests require attention, the appropriate analytical department shall be notified. A list of holding times is maintained in the most recent revision of the QA Manual.

10.11.3 Particular attention shall also be given to soil samples that require any type of volatile analysis (8260, 8021, GRO, etc.). If these samples are received as encores or in soil jars they must be extracted within 48 hours of collection. Pre-weigh vials do not need any special treatment. The procedure for processing encores and soil jars is detailed in Appendix F.

10.11.4 Any samples received more than five days after the time of collection shall be evaluated for all holding times. Some extractions and wet chemistry procedure have 7 day holding times and while these would not normally be considered "short holds", they may be in jeopardy due to the excessive time elapsed between sampling and receipt. Analytical departments shall be notified of any such samples. Customer service personnel or a laboratory manager shall be consulted if a holding time is in question.

10.11.5 For any samples received at the laboratory with analyses that are already past hold at the time of receipt, the Chain of Custody shall be stamped with the red "EXPIRED" stamp and the expired tests noted in the stamp's box area.

10.12 The chlorine content shall be checked for samples requiring sodium thiosulfate or sodium sulfite as a preservative or samples requesting cyanide. See the most recent revision of the ALSI QA Manual for a list of analyses that require sodium thiosulfate or sodium sulfite as a preservative.

10.12.1 In order to verify that the preservative has been added, the samples are tested for the absence of chlorine by inserting a 5-3/4" Pasteur pipette into the sample and placing a drop on KI paper for non drinking water samples. For drinking water samples, the residual chlorine is verified by using the total chlorine paper. If a color

change to blue or violet is indicated, sodium thiosulfate must be added to the sample until the chlorine has been neutralized.

- 10.12.2 The presence/absence of chlorine must be documented in the sample preservation logbook file. Whether or not samples are preserved properly shall be documented on the Chain of Custody by checking the appropriate Y/N box in the receipt information section of the Chain and initialing the corresponding line. Any non-conformances shall be specifically mentioned in the sample/COC comments section of the Chain.
- 10.13 The turbidity of drinking water samples for metals analysis shall be determined by the Metals or Wet Chemistry department.
- 10.14 At this time, the samples are entered into the Horizon LIMS. See SOP for COC Entry (19-COC).
- 10.15 Labels generated by the Horizon LIMS are then affixed to the bottles and the bottles are stored as follows:
 - 10.15.1 Volatile aqueous samples for GC are stored in Refrigerator #26 and 29 located in the GC lab. Samples known to contain a high concentration of volatiles shall not be stored in this area. (Depending upon the matrix, high-concentration volatiles are often stored in the oil cabinet in Sample Receiving, or if requiring refrigeration, may be stored in the GCMS Soil Refrigerator.)
 - 10.15.2 Volatile aqueous samples for GC/MS are stored in Refrigerators #26 and 29 located in the GC/MS lab. Samples known to contain a high concentration of volatiles shall not be stored in this area. (Depending upon the matrix, high-concentration volatiles are often stored in the oil cabinet in Sample Receiving, or if requiring refrigeration, may be stored in the GCMS Soil Refrigerator #10.)
 - 10.15.3 Metals samples are stored in numerical order on shelves in the Inorganic Prep Lab excluding drinking water metals samples. Drinking water metals samples that do not require a digestion are stored in the Metals lab. Dissolved metals are stored in the Metals Department.
 - 10.15.4 Total Organic Carbon and Total Organic Halogen samples are stored in the walk-in refrigerator on the lower level.
 - 10.15.5 Samples requiring any sort of organic extractions are stored on the right

side of the Harford walk-in refrigerator located on the upper level.

10.15.6 All other liquid samples not mentioned are stored in the remaining two walk-in refrigerators in numerical order.

10.15.7 Solid samples are stored on the left side of the Harford walk-in refrigerator on the upper level.

10.15.8 Solid volatile samples and petroleum samples are stored in either GC or GCMS, wherever the testing is done.

10.15.9 A very limited number of samples do not require refrigeration. These are often associated with atypical analysis. Approval from a department supervisor is required to store samples without refrigeration.

10.15.10 Samples with special storage considerations like solvents or free product are stored in the prep hoods.

10.16 All samples are stored at least two (2) weeks following the completion of the last analysis on the lab report with the exception of VOA vials and microbiology samples. Samples requiring special storage considerations are handled project by project by the appropriate customer service representative. Samples requiring **DoD** certification must be stored for 60 days.

10.17 Samples received over the weekend and during off-peak hours shall be handled in the manner outlined in Appendix B.

11 Calculations

11.1 Not applicable

12 Reporting Results

12.1 Not applicable

13 Waste Disposal

13.1 Refer to ALSI SOP 19-Waste Disposal.

14 Pollution Prevention

14.1 Pollution prevention encompasses any technique that reduces or eliminates the

quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. Management shall consider pollution prevention a high priority. Extended storage of unused chemicals increases the risk of accidents. The laboratory shall consider smaller quantity purchases which will result in fewer unused chemicals being stored and reduce the potential for exposure by employees. ALSI tracks chemicals when received by recording their receipt in a traceable logbook. Each chemical is then labeled according to required procedures and stored in assigned locations for proper laboratory use.

15 Definitions

- 15.1 Refer to ALSI QA Plan under Laboratory Quality Control Checks for general definitions.

16 Troubleshooting

- 16.1 Refer to maintenance logs and instrument manuals for guidance in troubleshooting specific problems related to the instrumentation used in this method.

APPENDIX B

The following steps can be followed for samples received after hours and on weekends:

1. Sign the Chain of Custody (COC) in the “received by” section on the bottom of the COC form only after inspecting samples to ensure that all information on the COC matches the samples being submitted for client submission.
2. Tear off the gold copy of the COC (last page), and give it to the client submitting the sample.
3. Check the analyses required for the samples received on the COC for any analyses on the following list which require short holding times (HT). If any of these tests are required and there are no trained personnel available to complete the analysis within the holding time, contact the supervisor of the department that performs the analysis. If the supervisor cannot be reached, contact the Laboratory Manager or appropriate Customer Service Representative.

Analyze Immediately

Chlorine Demand

Turbidity

Residual Chlorine

Total Chlorine

Odor

Dissolved Oxygen

pH

Sulfite

Fecal Coliform (6hr)

24 Hour HT

Hexavalent Chromium

Total Coliform (30 hr)

48 Hour HT

BOD/CBOD

Nitrate

Nitrite

Orthophosphate

MBAS

Osmotic Pressure

Color

Settleable Solids

UV254

Encore Extraction

4. Check the temperature of the samples as specified in Section 10.4
5. Place samples and COC form together in the lower level walk-in.
6. Leave an E-mail message for the appropriate Customer Service Representative letting them know what was received, and what actions were taken.

This document is the property of Analytical Laboratory Services, Inc. It may be used by the recipient only for the purpose for which it was transmitted. It is submitted in confidence and its disclosure to you is not intended to constitute public disclosure or authorization for disclosure to other parties. It may not be copied or communicated without the written consent of Analytical Laboratory Services, Inc.



Analytical Laboratory Services, Inc.

Environmental • Industrial Hygiene • Field Services

34 Dogwood Lane • Middletown, PA 17057 • 717.944.5541 • Fax: 717.944.1430

**CHAIN OF CUSTODY/
REQUEST FOR ANALYSIS**

**ALL SHADED AREAS MUST BE COMPLETED BY THE CLIENT /
SAMPLER. INSTRUCTIONS ON THE BACK.**

COC #: _____ of _____
 ALSI Quote #: _____

Client Name: _____			**Container Type			Receipt Information (completed by Receiving Lab)		
Address: _____			**Container Size			Cooler Temp: _____ Therm. ID: _____		
Contact: _____			***			No. of Coolers: _____ Y N Initial		
Phone#: _____			Preservative			Custody Seals Present? <input type="checkbox"/>		
Project Name/#: _____			ANALYSES/METHOD REQUESTED			(If present) Seals Intact? <input type="checkbox"/>		
Bill To: _____						Received on Ice? <input type="checkbox"/>		
TAT <input type="checkbox"/> Normal-Standard TAT is 10-12 business days.						COC/Labels Complete/Accurate? <input type="checkbox"/>		
<input type="checkbox"/> Rush-Subject to ALSI approval and surcharges.						Cont. in Good Cond.? <input type="checkbox"/>		
Date Required: _____ Approved By: _____						Correct Containers? <input type="checkbox"/>		
Email? <input type="checkbox"/> -Y			Correct Sample Volumes? <input type="checkbox"/>			Correct Preservation? <input type="checkbox"/>		
Fax? <input type="checkbox"/> -Y No.:			Headspace/Volatiles? <input type="checkbox"/>			Courier/Tracking #: _____		
Sample Description/Location <small>(as it will appear on the lab report)</small>		Sample Date	Military Time	*G or C	**Matrix	Enter Number of Containers Per Analysis Requested or Field Results Below.		
						Sample/COC Comments		
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								
SAMPLED BY (Please Print): _____			LOGGED BY (signature): _____ DATE: _____			Data Deliverables: <input type="checkbox"/> Standard <input type="checkbox"/> CLP-like <input type="checkbox"/> NJ-Reduced <input type="checkbox"/> NJ-Full <input type="checkbox"/> if yes, format type: _____ EDD's Required? _____ DOD Criteria Required? _____		
REVIEWED BY (signature): _____			DATE: _____					
Relinquished By / Company Name		Date	Time	Received By / Company Name		Date	Time	State Samples Collected In: NC <input type="checkbox"/> SDWA Forms Required? <input type="checkbox"/> NY <input type="checkbox"/> PA <input type="checkbox"/> Other: _____ PWSID: _____ ALSI Field Services: <input type="checkbox"/> Pickup <input type="checkbox"/> Labor <input type="checkbox"/> Composite <input type="checkbox"/> Sampling <input type="checkbox"/> Rental <input type="checkbox"/> Enrollment <input type="checkbox"/> Other: _____
1 SAMPLED BY				2				
3				4				
5				6				
7				8				
9				10				

* G=Grab; C=Composite **Matrix: AF=Air; DW=Drinking Water; GW=Groundwater; OL=Oil; OL=Other Liquid; SL=Sludge; SO=Soil; WP=Wipe; WW=Wastewater
 Copies: WHITE - ORIGINAL. CANARY - CUSTOMER COPY ***Container Type: AG-Amber Glass; CG-Clear Glass, PL-Plastic. Container Size: 250ml, 500ml, 1L, 8oz., etc. Preservative: HCl, HNO3, NaOH, etc. Rev 5/06

APPENDIX C

Method: 19-Rec/Han
 Revision: 11
 Date: February 6, 2007
 Page: 22 of 36

UNCONTROLLED DOCUMENT! DO NOT Transfer or Print

Method: 19-Rec/Han
 Revision: 11
 Date: February 6, 2007
 Page: 23 of 36

**APPENDIX D
 DC-2 Forms**

Lab Name: Analytical Laboratory Services, Inc.	DAS Number:	SDG Number:
City: Middletown	State: Pennsylvania	Zip Code: 17057
Order Number:	Parameter:	

	Page Numbers From	To	Check Lab	EPA
Inventory Sheet	XXXXXXXX	XXXXXXXX		
SDG Narrative				
SDG Cover Sheet/Traffic Report				
QC Data				
Sample Data				
Standard Data				
Blank Data				
Raw Data				
Preparation Logs				
Clean-up Logs				
Analysis Logs				
Internal Chain of Custody Logs				
Shipping / Receiving Documents				
Telephone / e-mail Logs				
Other Records				

Organization	Lab Inventory	Region 3 Auditor	EPA Verifier
Print Name			
Title			
Date			
Signature			

Method: 19-Rec/Han

Revision: 11

Date: February 6, 2007

Page: 24 of 36

** Reference: Critical Elements for Certification of Drinking Water Laboratories for Chemistry, October 1, 1997.*

¹ In addition cool to 4°C.

² The holding time for Heptachlor under this method is 7 days.

³ Samples that have been preserved with mercuric chloride may be disposed of in at least two ways: as a hazardous waste, or by passing over an absorbent column (i.e., alumina, activated with carbon, etc.) for mercury absorption, with the effluent analyzed periodically for breakthrough. The absorbent would then be disposed of as a hazardous waste. Other techniques may be applicable.

⁴Mercuric chloride is only required in drinking water samples in which biological degradation of the target pesticides and herbicides are exhibited.

⁵No extract.

Method: 19-Rec/Han
Revision: 11
Date: February 6, 2007
Page: 25 of 36

APPENDIX E

New Employee Orientation

Sample Receiving Training Checklist

Procedure: Checking Sample Preservatives

SOP: _____

Revision: _____

	Analyst	Trainer
1) Analyst has read and understands SOP 19-Rec/Han and signed the concurrence form.	_____	_____
2) Analyst knows where to find the list of sample types that require preservation.	_____	_____
3) Analyst knows which pH paper to use for checking the pH of acidified samples and where to find it.	_____	_____
4) Analyst knows how to apply the sample to the pH paper and how to interpret the reading for acidified samples.	_____	_____
5) Analyst knows which pH paper to use for checking the pH of basic samples and where to find it.	_____	_____
6) Analyst knows how to apply the sample to the pH paper and how to interpret the reading for basic samples.	_____	_____
7) Analyst knows how to adjust the pH of any sample if the measured value is not acceptable.	_____	_____
8) Analyst knows which test papers to use for checking chlorine content and where to find them.	_____	_____
9) Analyst knows how to apply the sample to the test papers and how to interpret the reading for chlorine content.	_____	_____

This document is the property of Analytical Laboratory Services, Inc. It may be used by the recipient only for the purpose for which it was transmitted. It is submitted in confidence and its disclosure to you is not intended to constitute public disclosure or authorization for disclosure to other parties. It may not be copied or communicated without the written consent of Analytical Laboratory Services, Inc.

Rev. 5/05

Method: 19-Rec/Han

Revision: 11

Date: February 6, 2007

Page: 26 of 36

	Analyst	Trainer
10) Analyst knows how to properly complete the preservation logbook.	_____	_____
11) Analyst knows that USACE samples require a small amount of Sample to be poured out for sampling pH.	_____	_____
12) Analyst knows how to complete all information on the chain of custody regarding preservation including where to record non-conformances.	_____	_____
13) Analyst is aware that when metals samples are acidified upon Receipt a copy of the preservation log must accompany the sample to the metals prep department.	_____	_____
14) Analyst has demonstrated his/her ability on at least five separate projects/workorders.	_____	_____

The workorders used for demonstration are:

_____ Analyst Name	_____ Analyst Signature	_____ Date
_____ Supervisor Name	_____ Supervisor Signature	_____ Date

This document is the property of Analytical Laboratory Services, Inc. It may be used by the recipient only for the purpose for which it was transmitted. It is submitted in confidence and its disclosure to you is not intended to constitute public disclosure or authorization for disclosure to other parties. It may not be copied or communicated without the written consent of Analytical Laboratory Services, Inc.

Rev. 5/05

Method: 19-Rec/Han

Revision: 11

Date: February 6, 2007

Page: 27 of 36

New Employee Orientation

Sample Receiving Training Checklist

Procedure: Measuring Cooler Temperatures

SOP: _____

Revision: _____

	Analyst	Trainer
1) Analyst read and understands SOP 19-Rec/Han and signed the concurrence form.	_____	_____
2) Analyst knows the location of the mercury thermometer.	_____	_____
3) Analyst knows where to find and how to apply the correction factor for the mercury thermometer.	_____	_____
4) Analyst can properly take and record a cooler temperature using a provided temperature blank.	_____	_____
5) Analyst knows the acceptable limits for cooler temperatures are above the freezing point of water to 6 degrees C.	_____	_____
6) Analyst knows the location of the IR temperature gun.	_____	_____
7) Analyst knows how to properly use the IR gun to take a sample temperature (i.e. where to shoot the gun through the bottle).	_____	_____
8) Analyst knows where to find and how to apply the correction factors for the IR temperature gun.	_____	_____
9) Analyst knows to take multiple temperatures if the initial bottle used is outside the acceptable limits.	_____	_____
10) Analyst is aware that samples received same day as sampling are acceptable if evidence exists that the chilling process was begun.	_____	_____

This document is the property of Analytical Laboratory Services, Inc. It may be used by the recipient only for the purpose for which it was transmitted. It is submitted in confidence and its disclosure to you is not intended to constitute public disclosure or authorization for disclosure to other parties. It may not be copied or communicated without the written consent of Analytical Laboratory Services, Inc.

Rev. 5/05

Method: 19-Rec/Han

Revision: 11

Date: February 6, 2007

Page: 28 of 36

- | | Analyst | Trainer |
|--|---------|---------|
| 11) Analyst is aware that designated refrigerators are available for "holding" micro and volatile samples during processing. | _____ | _____ |
| 12) Analyst has demonstrated this ability on at least five separate projects/workorders. | _____ | _____ |

The workorders used for demonstration are:

Analyst Name Analyst Signature Date

Supervisor Name Supervisor Signature Date

This document is the property of Analytical Laboratory Services, Inc. It may be used by the recipient only for the purpose for which it was transmitted. It is submitted in confidence and its disclosure to you is not intended to constitute public disclosure or authorization for disclosure to other parties. It may not be copied or communicated without the written consent of Analytical Laboratory Services, Inc.

New Employee Orientation

Sample Receiving Training Checklist

Procedure: Receiving Samples

SOP: _____

Revision: _____

	Analyst	Trainer
1) Analyst has read and understands SOP 19-Rec/Han and signed the concurrence form.	_____	_____
2) Analyst will ensure that all samples are accompanied by a chain of custody.	_____	_____
3) If a walk-in client attempts to drop off samples not accompanied by a chain of custody, the analyst provides a chain for them to complete on-site.	_____	_____
4) Analyst receives custody of the sample(s) by signing the chain of custody upon receipt of samples, including the date and time received.	_____	_____
5) When samples are received with a chain of custody from a source other than ALSI, the analyst will confirm client and sample information is present.	_____	_____
6) Analyst checks for the presence of custody seals and whether they are intact and records all findings including any non-conformances appropriately on the chain of custody.	_____	_____
7) Analyst checks the sample temperature and for the presence of ice and records all findings including any non-conformances appropriately on the chain of custody.	_____	_____
8) Analyst checks for breakage and leakage and records all findings including any non-conformances appropriately on the chain of custody.	_____	_____

Method: 19-Rec/Han

Revision: 11

Date: February 6, 2007

Page: 30 of 36

	Analyst	Trainer
9) Analyst compares the information on the chain of custody to the information on the bottle labels and records all findings including any non-conformances on the chain of custody.	_____	_____
10) Analyst checks to see that the sample containers are appropriate for the analyses requested and records all findings including any non-conformances on the chain of custody.	_____	_____
11) Analyst checks all volatile containers for headspace and records all findings including any non-conformances on the chain of custody.	_____	_____
12) Analyst properly checks for preservatives and records all findings including any non-conformances on the chain of custody.	_____	_____
13) Analyst knows what analytes have short holding times and notifies the appropriate departments upon receipt.	_____	_____
14) Analyst has demonstrated this ability on at least five separate projects/workorders.	_____	_____

The workorders used for demonstration are:

Analyst Name Analyst Signature Date

Supervisor Name Supervisor Signature Date

Appendix F

Receipt of Soil Volatile Samples in Encores or Soil Jars

Volatile soil samples received in soil jars or encores must be extracted into sodium bisulfate and/or methanol vials within 48 hours from the time of collection. Samples received with little time remaining prior to expiration require immediate attention. Proceed as follows:

- 1) When these types of samples are received, check the sample containers against the COC. Following procedure, note any discrepancies on the COC.
- 2) Log the samples into Horizon.
- 3) Initiate the ALSI Encore Internal COC Logbook. Even if the samples cannot be logged into Horizon for any reason, continue with this step. In such a case, use the sample description in lieu of a Horizon sample number.
- 4) Transfer the samples to the GC/MS laboratory along with a copy of the COC.

NOTE: Samples must be delivered to an actual person who acknowledges receipt of the samples by initialing the Encore Internal COC Logbook. The samples cannot be left on a counter or placed in the refrigerator without documented receipt. If an individual qualified to extract encores is not available, refer to the GC/MS phone list located in sample receiving and contact an individual to come in and perform the extractions.

- 5) Complete the Encore Internal COC Logbook. (See Appendix G)

SOP Change Summary

<u>Section No.</u>	<u>Section</u>	<u>Reason for Change</u>
Revision 7:		
5.9	Apparatus and Materials	Correction to current SOP
6.0	Reagents	Correction to current SOP
10.2.8	Procedure	A2LA Audit Response
10.6-10.7	Procedure	Horizon LIMS Implementation
Revision 8:		
10.2.4	Procedure	New Jersey DEP audit response
Revision 9:		
2	Summary of Method	DoD audit response 5/13/05
4	Safety	DoD audit response 5/13/05
5	Apparatus and Materials	DoD audit response 5/13/05
6	Reagents	DoD audit response 5/13/05
7	Instrument Calibrations	DoD audit response 5/13/05
8	Quality Control	DoD audit response 5/13/05
10	Procedure	DoD audit response 5/13/05
Appendix C	Sample Collection, Containers & Preservation For Organic Contaminants	DoD audit response 5/13/05
Appendix D	ALSI Chain of Custody	DoD audit response 5/13/05
Appendix E	DC-2 Data Package Inventory Checklist	DoD audit response 5/13/05

SOP Change Summary (continued)

Section No. Section Reason for Change

Revision 10: 10/06/2006

Revised refrigeration temperatures to read "...above the freezing point of water up to 6°C..." to reflect NELAC verbiage.

1.3	Scope and Application	Added use of project specific criteria
4.1	Safety	Added statement about maintaining MSDS
6.4.1, 6.4.2	Reagents	Revised volume of dispenser tops from 1 mL to 2 mL; revised number of squirts to accommodate new dispenser volume
6.4.3.3	Reagents	Revised volume added to TOC bottle
8.1	Quality Control	Added verbiage indicating that the SOP falls under the QA Plan umbrella
8.4, 8.5	Quality Control	Added initial DOC and statement about ongoing proficiency
10.10.4	Procedure	Removed recording of tests required in sample preservation logbook; added generation of logbook in a Microsoft Excel spreadsheet
11	Calculations	Added section
12	Reporting Results	Added section
16	Troubleshooting	Added section
Removed Appendix duplicating QA Plan Appendix B, Container, Preservation, Storage and Holding Times		
C	Appendix	Updated COC to current form
E	Appendix	Added examples of training forms

SOP Change Summary (continued)

Method: 19-Rec/Han

Revision: 11

Date: February 6, 2007

Page: 35 of 36

<u>Section No.</u>	<u>Section</u>	<u>Reason for Change</u>
<u>Revision 11: 02/05/07</u>		
5.8.3	Apparatus and Materials	Added new walk-in refrigerator
6.1	Reagents	Added ALSI generated reagent water
6.3	Reagents	Replaced “well/tap” water with “reagent” water
6.4.10	Reagents	Added Ethylenediamine (EDA) Solution
10.2.1	Procedure	Added specific verbiage for microbial samples
10.11	Procedure	Added verbiage about sample holding times, microbial sample test times, soil samples and corrective actions for samples past holding time
10.13	Procedure	Added Wet Chem as lab for turbidity analysis
10.15.5, 10.15.7	Procedure	Added specific sample storage locations; deleted defunct refrigerators
B	Appendix	Added Fecal Coliform to “Analyze Immediately” column; Added Total Coliform to “24 Hour HT” column
F	Appendix	Added directions for the receipt of soil volatile samples in Encores or Soil Jars
G	Appendix	Added example of Encore Internal COC logbook page

Control Limits

ID 4980 Method M5 8260/5035 Prep Method Y6 SW846 5035

Matrix Sample LCS Instrument Effective 07/23/06 Stop User VED Type % Recovery

Other Criteria None Workorder Profile Client

Value

Note

Analytes

Sort	Analyte	Name	LCL	UCL	LWL	UWL	Mean
1	630-20-6	1,1,1,2-Tetrachloroethane	74	126			
2	919-94-8	tert-Amyl Ethylether	70	130			
3	75-85-4	tert-Amyl Alcohol	70	130			
4	10061-01-5	cis-1,3-Dichloropropene	76	123			
5	108383/106423	mp-Xylene	72	130			
6	71-23-8	n-Propanol	5	227			
7	95-47-6	o-Xylene	75	129			
8	95-49-8	o-Chlorotoluene	70	142			
9	103-65-1	n-Propylbenzene	68	134			
10	104-51-8	n-Butylbenzene	64	141			
11	106-43-4	p-Chlorotoluene	72	131			
12	110-57-6	trans-1,4-Dichloro-2-butene	67	128			
13	10061-02-6	trans-1,3-Dichloropropene	77	123			
14	156-60-5	trans-1,2-Dichloroethene	66	133			
15	75-65-0	tert-Butyl Alcohol	51	152			
16	98-06-6	tert-Butylbenzene	71	133			

Control Limits

ID: 4980 Method: M5 8260/5035 Prep Method: Y6 SW846 5035

Matrix: Sample: LCS Instrument: Effective: 07/23/06 Stop: User: MED Type: % Recovery

Other Criteria: None Workorder Profile Client Value:

Note:

Analytes

Sort	Analyte	Name	LCL	UCL	LWL	UWL	Mean
17	994-05-8	tert-Amyl methyl ether	79	123			
18	135-98-8	sec-Butylbenzene	72	136			
19	99-87-6	p-Isopropyltoluene	69	135			
20	108-90-7	Chlorobenzene	76	125			
21	124-48-1	Chlorodibromomethane	75	124			
22	75-15-0	Carbon Disulfide	47	144			
23	74-87-3	Chloromethane	44	139			
24	67-66-3	Chloroform	73	126			
25	56-23-5	Carbon Tetrachloride	64	136			
26	75-00-3	Chloroethane	1	141			
27	126-99-8	Chloroprene	58	130			
28	637-92-3	Ethyl tert-butyl ether	75	121			
29	97-63-2	Ethyl Methacrylate	78	126			
30	60-29-7	Ethyl Ether	66	137			
31	141-78-6	Ethyl Acetate	43	152			
32	107-39-1	Diisobutylene	23	148			

Control Limits

ID: 4980 Method: M5 8260/5035 Prep Method: Y6 SW846 5035

Matrix: Sample: LCS Instrument: Effective: 07/23/06 Stop: User: VED Type: % Recovery

Other Criteria: None Workorder Profile Client Value:

Note:

Analytes

Sort	Analyte	Name	LCI	UCI	LWL	UWL	Mean
33	108-20-3	Diisopropyl ether	71	128			
34	75-43-4	Dichlorofluoromethane	1	117			
35	75-71-8	Dichlorodifluoromethane	16	152			
36	74-95-3	Dibromomethane	79	128			
37	110-82-7	Cyclohexane	62	143			
38	107-12-0	Propionitrile	68	159			
39	109-66-0	Pentane	30	125			
40	111-65-9	Octane	55	160			
41	98-95-3	Nitrobenzene	52	139			
42	91-20-3	Naphthalene	60	146			
43	75-09-2	Methylene Chloride	68	133			
44	1634-04-4	Methyl t-Butyl Ether	70	118			
45	80-62-6	Methyl methacrylate	59	140			
46	126-98-7	Methacrylonitrile	74	147			
47	98-82-8	Isopropylbenzene	71	137			
48	67-63-0	Isopropyl Alcohol	20	100			

Control Limits

ID: 4980 Method: M5 3260/5035 Prep Method: Y6 SW846 5035

Matrix: Sample: LCS Instrument: Effective: 07/23/06 Stop: User: VED Type: % Recovery

Other Criteria: None Workorder Profile Client Value:

Note:

Analytes							
Sort	Analyte	Name	LCL	UCL	LWL	UWL	Mean
49	78-83-1	Isobutyl alcohol	41	128			
50	74-88-4	Iodomethane	31	182			
51	110-54-3	Hexane	16	165			
52	67-72-1	Hexachloroethane	73	131			
53	87-68-3	Hexachlorobutadiene	63	140			
54	142-82-5	Heptane	55	157			
55	76-13-1	Freon 113	40	109			
56	100-41-4	Ethylbenzene	73	133			
57	75-01-4	Vinyl Chloride	53	141			
58	108-05-4	Vinyl Acetate	30	154			
59	75-69-4	Trichlorofluoromethane	40	130			
60	79-01-6	Trichloroethene	72	129			
61	79-00-5	1,1,2-Trichloroethane	79	123			
62	1330-20-7	Total Xylenes	73	130			
63	108-88-3	Toluene	73	129			
64	109-99-9	Tetrahydrofuran	61	148			

Control Limits

ID: 4980 Method: M5 8260/5035 Prep Method: Y6 SW846 5035

Matrix: Sample: LCS Instrument: Effective: 07/23/06 Stop: User: VED Type: % Recovery

Other Criteria: None Workorder Profile Client Value:

Note:

Analytes

Sort	Analyte	Name	LEL	UCL	LWL	UWL	Mean
65	127-18-4	Tetrachloroethene	58	137			
66	100-42-5	Styrene	77	130			
67	156-59-2	cis-1,2-Dichloroethene	75	128			
68	142-28-9	1,3-Dichloropropane	80	122			
69	542-75-6	1,3-Dichloropropene, Total	77	122			
70	108-67-8	1,3,5-Trimethylbenzene	71	132			
71	541-73-1	1,3-Dichlorobenzene	72	127			
72	106-46-7	1,4-Dichlorobenzene	72	126			
73	123-91-1	1,4-Dioxane	9	267			
74	544-10-5	1-Chlorohexane	71	137			
75	78-93-3	2-Butanone	64	148			
76	107-13-1	Acrylonitrile	64	148			
77	107-02-8	Acrolein	18	139			
78	75-05-8	Acetonitrile	2	177			
79	67-64-1	Acetone	58	146			
80	108-10-1	4-Methyl-2-Pentanone(MIBK)	64	143			

ID: 4980 Method: M5 8260/5035 Prep Method: Y6 SW846 5035
 Matrix: [] Sample: LCS Instrument: [] Effective: 07/23/06 Stop: [] User: VED Type: % Recovery
 Other Criteria: None Workorder Profile Client Value: []
 Note: []

Analytes

Sort	Analyte	Name	LCI	UCL	LWL	UWL	Mean
81	107-05-1	3-Chloro-1-propene	60	145			
82	79-46-9	2-Nitropropane	60	140			
83	591-78-6	2-Hexanone	62	147			
84	74-83-9	Bromomethane	43	148			
85	75-25-2	Bromoform	68	131			
86	75-27-4	Bromodichloromethane	74	127			
87	74-97-5	Bromochloromethane	71	120			
88	108-86-1	Bromobenzene	76	124			
89	100-44-7	Benzyl Chloride	59	145			
90	71-43-2	Benzene	75	132			
91	594-20-7	2,2-Dichloropropane	64	132			
92	71-55-6	1,1,1-Trichloroethane	68	131			
93	79-34-5	1,1,2,2-Tetrachloroethane	72	134			
94	75-34-3	1,1-Dichloroethane	74	131			
95	75-35-4	1,1-Dichloroethene	59	139			
96	563-58-6	1,1-Dichloropropene	70	134			

Control Limits

ID: 4980 Method: M5 B260/5035 Prep Method: Y6 SW846 5035

Matrix: Sample: LCS Instrument: Effective: 07/23/06 Stop: User: VED Type: % Recovery

Other Criteria: None Workorder Profile Client Value:

Note:

Analytes

Sort	Analyte	Name	LCL	UCL	LWL	UWL	Mean
95	75-35-4	1,1-Dichloroethene	59	139			
96	985-95-8	1,1-Dichloropropane	70	134			
97	87-61-6	1,2,3-Trichlorobenzene	68	129			
98	96-18-4	1,2,3-Trichloropropane	74	130			
99	120-82-1	1,2,4-Trichlorobenzene	63	132			
100	95-63-6	1,2,4-Trimethylbenzene	70	131			
101	96-12-8	1,2-Dibromo-3-chloropropane	52	151			
102	106-93-4	1,2-Dibromoethane	76	127			
103	95-50-1	1,2-Dichlorobenzene	75	126			
104	107-06-2	1,2-Dichloroethane	69	132			
105	78-87-5	1,2-Dichloropropane	78	131			
106	1868-53-7	Dibromofluoromethane	62	123			
107	17060-07-0	1,2-Dichloroethane-d4	56	124			
108	2037-26-5	Toluene-d8	59	131			
109	460-00-4	4-Bromofluorobenzene	51	128			
110	540-59-0	1,2-Dichloroethene, Total	75	128			

ID: 4984 Method: M5 8280/5035

Prep Method: Y6 SW846 5035

Matrix: Sample: MSD Instrument: Effective: 07/23/06 Stop: User: VED Type: RPD

Other Criteria: None Workorder Profile Client

Value: []

Note: []

Analytes

Sort	Analyte	Name	LCL	UCL	LWL	UWL	Mean
1	75-71-8	Dichlorodifluoromethane	0	40			
2	919-94-8	tert-Amyl Ethylether	0	40			
3	75-85-4	tert-Amyl Alcohol	0	40			
4	74-87-3	Chloromethane	0	40			
5	75-01-4	Vinyl Chloride	0	40			
6	74-83-9	Bromomethane	0	40			
7	75-00-3	Chloroethane	0	40			
8	75-43-4	Dichlorofluoromethane	0	40			
9	75-69-4	Trichlorofluoromethane	0	40			
10	109-66-0	Pentane	0	40			
11	60-29-7	Ethyl Ether	0	40			
12	107-02-8	Acrolein	0	40			
13	75-35-4	1,1-Dichloroethene	0	40			
14	76-13-1	Freon 113	0	40			
15	67-64-1	Acetone	0	40			
16	74-88-4	Iodomethane	0	40			

Control Limits

ID: 4984 Method: M5 8260/5035 Prep Method: Y6 SW846 5035
 Matrix: Sample: MSD Instrument: Effective: 07/23/06 Stop: User: VED Type: RPD

Other Criteria: None Workorder Profile Client Value:

Note:

Analytes

Sort	Analyte	Name	LCL	UCL	LWL	UWL	Mean
17	75-15-0	Carbon Disulfide	0	40			
18	107-05-1	3-Chloro-1-propene	0	40			
19	75-09-2	Methylene Chloride	0	40			
20	75-65-0	tert-Butyl Alcohol	0	40			
21	107-13-1	Acrylonitrile	0	40			
22	156-60-5	trans-1,2-Dichloroethene	0	40			
23	1634-04-4	Methyl t-Butyl Ether	0	40			
24	110-54-3	Hexane	0	40			
25	75-34-3	1,1-Dichloroethane	0	40			
26	108-05-4	Vinyl Acetate	0	40			
27	126-99-8	Chloroprene	0	40			
28	594-20-7	2,2-Dichloropropane	0	40			
29	156-59-2	cis-1,2-Dichloroethene	0	40			
30	78-93-3	2-Butanone	0	40			
31	74-97-5	Bromochloromethane	0	40			
32	109-99-9	Tetrahydrofuran	0	40			

Control Limits

ID: 4984 Method: M5 8260/5035 Prep Method: Y6 SW846 5035

Matrix: Sample: MSD Instrument: Effective: 07/23/06 Stop: User: VED Type: RPD

Other Criteria: None Workorder Profile Client Value:

Note:

Analytes

Sort	Analyte	Name	LCL	UCL	LWL	UWL	Mean
33	67-66-3	Chloroform	0	40			
34	71-55-6	1,1,1-Trichloroethane	0	40			
35	110-82-7	Cyclohexane	0	40			
36	56-23-5	Carbon Tetrachloride	0	40			
37	563-58-6	1,1-Dichloropropene	0	40			
38	71-43-2	Benzene	0	40			
39	107-06-2	1,2-Dichloroethane	0	40			
40	142-82-5	Heptane	0	40			
41	107-39-1	Diisobutylene	0	40			
42	79-01-6	Trichloroethene	0	40			
43	78-87-5	1,2-Dichloropropane	0	40			
44	74-95-3	Dibromomethane	0	40			
45	75-27-4	Bromodichloromethane	0	40			
46	79-46-9	2-Nitropropane	0	40			
47	110-75-8	2-Chloroethylvinyl ether	0	40			
48	10061-01-5	cis-1,3-Dichloropropene	0	40			

Control Limits

ID: 4984 Method: M5 8260/5035 Prep Method: Y6 SW846 5035

Matrix: Sample: MSD Instrument: Effective: 07/23/06 Stop: User: VED Type: RPD

Other Criteria: None Workorder Profile Client Value:

Note:

Analytes

Sort	Analyte	Name	LCL	UCL	LWL	UWL	Mean
49	108-10-1	4-Methyl-2-Pentanone(MIBK)	0	40			
50	108-88-3	Toluene	0	40			
51	111-65-9	Octane	0	40			
52	10061-02-6	trans-1,3-Dichloropropene	0	40			
53	542-75-6	1,3-Dichloropropene, Total	0	40			
54	79-00-5	1,1,2-Trichloroethane	0	40			
55	127-18-4	Tetrachloroethene	0	40			
56	142-28-9	1,3-Dichloropropane	0	40			
57	591-78-6	2-Hexanone	0	40			
58	124-48-1	Chlorodibromomethane	0	40			
59	106-93-4	1,2-Dibromoethane	0	40			
60	108-90-7	Chlorobenzene	0	40			
61	544-10-5	1-Chlorohexane	0	40			
62	630-20-6	1,1,1,2-Tetrachloroethane	0	40			
63	100-41-4	Ethylbenzene	0	40			
64	108383/106423	mp-Xylene	0	40			

ID: 4984 Method: M5 8260/5035

Prep Method: Y6 SW846 5035

Matrix: Sample: MSD Instrument: Effective: 07/23/06 Stop: User: VED Type: RPD

Other Criteria: None Workorder Profile Client

Value:

Note:

Analytes

Sort	Analyte	Name	LCL	UCL	LWL	UWL	Mean
65	95-47-6	o-Xylene	0	40			
66	1330-20-7	Total Xylenes	0	40			
67	100-42-5	Styrene	0	40			
68	75-25-2	Bromoforn	0	40			
69	98-82-8	Isopropylbenzene	0	40			
70	79-34-5	1,1,2,2-Tetrachloroethane	0	40			
71	108-86-1	Bromobenzene	0	40			
72	96-18-4	1,2,3-Trichloropropane	0	40			
73	110-57-6	trans-1,4-Dichloro-2-butene	0	40			
74	103-65-1	n-Propylbenzene	0	40			
75	95-49-8	o-Chlorotoluene	0	40			
76	108-67-8	1,3,5-Trimethylbenzene	0	40			
77	106-43-4	p-Chlorotoluene	0	40			
78	98-06-6	tert-Butylbenzene	0	40			
79	95-63-6	1,2,4-Trimethylbenzene	0	40			
80	135-98-8	sec-Butylbenzene	0	40			

Control Limits

ID: 4984 Method: M5 8260/5035 Prep Method: Y6 SW846 5035

Matrix: Sample: MSD Instrument: Effective: 07/23/06 Stop: User: VED Type: RPD

Other Criteria: None Workorder Profile Client Value:

Note:

Analytes

Sort	Analyte	Name	LCL	UCL	LWL	UWL	Mean
81	541-73-1	1,3-Dichlorobenzene	0	40			
82	99-87-6	p-Isopropyltoluene	0	40			
83	106-46-7	1,4-Dichlorobenzene	0	40			
84	100-44-7	Benzyl Chloride	0	40			
85	104-51-8	n-Butylbenzene	0	40			
86	95-50-1	1,2-Dichlorobenzene	0	40			
87	96-12-8	1,2-Dibromo-3-chloropropane	0	40			
88	120-82-1	1,2,4-Trichlorobenzene	0	40			
89	87-68-3	Hexachlorobutadiene	0	40			
90	91-20-3	Naphthalene	0	40			
91	87-61-6	1,2,3-Trichlorobenzene	0	40			
92	108-20-3	Diisopropyl ether	0	40			
93	637-92-3	Ethyl tert-butyl ether	0	40			
94	994-05-8	tert-Amyl methyl ether	0	40			
95	75-05-8	Acetonitrile	0	40			
96	80-62-6	Methyl methacrylate	0	40			

ID: 4984 Method: M5 3260/5035 Prep Method: Y6 SW846 5035
 Matrix: Sample: MSD Instrument: Effective: 07/23/06 Stop: User: VED Type: RPD

Other Criteria: None Workorder Profile Client Value: _____

Note: _____

Analytes

Sort	Analyte	Name	LCL	UCL	LWL	UWL	Mean
33	627-92-3	Ethyl tert-butyl ether	0	40			
34	994-05-8	tert-Amyl methyl ether	0	40			
95	73-05-8	Acetonitrile	0	40			
96	88-62-6	Methyl methacrylate	0	40			
97	67-72-1	Hexachloroethane	0	40			
98	98-95-3	Nitrobenzene	0	40			
99	107-12-0	Propionitrile	0	40			
100	123-91-1	1,4-Dioxane	0	40			
101	141-78-6	Ethyl Acetate	0	40			
102	126-98-7	Methacrylonitrile	0	40			
103	97-63-2	Ethyl Methacrylate	0	40			
104	71-23-8	n-Propanol	0	40			
105	67-63-0	Isopropyl Alcohol	0	40			
106	78-83-1	Isobutyl alcohol	0	40			
107	79-20-9	Methyl acetate	0	40			
108	108-97-2	Methyl cyclohexane	0	40			

Control Limits

ID: 4966 Method: 40 SWB46 8260B Prep Method:

Matrix: Sample: LCS Instrument: Effective: 07/23/06 Stop: User: VED Type: % Recovery

Other Criteria: None Workorder Profile Client Value:

Note:

Analytes

Sort	Analyte	Name	LCL	UCL	LWL	UWL	Mean
1	630-20-6	1,1,1,2-Tetrachloroethane	78	121			
2	95-63-6	1,2,4-Trimethylbenzene	76	125			
3	106-93-4	1,2-Dibromoethane	80	124			
4	71-55-6	1,1,1-Trichloroethane	66	130			
5	107-06-2	1,2-Dichloroethane	70	133			
6	108-67-8	1,3,5-Trimethylbenzene	76	125			
7	79-34-5	1,1,2,2-Tetrachloroethane	74	135			
8	104-51-8	n-Butylbenzene	71	130			
9	71-43-2	Benzene	80	124			
10	79-00-5	1,1,2-Trichloroethane	82	126			
11	100-41-4	Ethylbenzene	80	124			
12	75-65-0	tert-Butyl Alcohol	17	168			
13	75-34-3	1,1-Dichloroethane	78	124			
14	98-82-8	Isopropylbenzene	73	129			
15	75-35-4	1,1-Dichloroethene	63	128			
16	1634-04-4	Methyl t-Butyl Ether	69	115			

Control Limits

ID: 4966 Method: 40 SW846 8260B Prep Method:

Matrix: Sample: LCS Instrument: Effective: 07/23/06 Stop: User: VED Type: % Recovery

Other Criteria: None Workorder Profile Client Value:

Note:

Analytes

Sort	Analyte	Name	LCL	UCL	LWL	UWL	Mean
17	563-58-6	1,1-Dichloropropene	76	126			
18	91-20-3	Naphthalene	56	134			
19	87-61-6	1,2,3-Trichlorobenzene	61	126			
20	108-88-3	Toluene	80	125			
21	96-18-4	1,2,3-Trichloropropane	75	132			
22	1330-20-7	Total Xylenes	79	125			
23	120-82-1	1,2,4-Trichlorobenzene	67	123			
24	108383/106423	mp-Xylene	79	125			
25	96-12-8	1,2-Dibromo-3-chloropropane	59	133			
26	95-47-6	o-Xylene	79	124			
27	95-50-1	1,2-Dichlorobenzene	82	118			
28	78-87-5	1,2-Dichloropropane	81	127			
29	541-73-1	1,3-Dichlorobenzene	81	118			
30	142-28-9	1,3-Dichloropropane	82	126			
31	542-75-6	1,3-Dichloropropene, Total	80	123			
32	123-91-1	1,4-Dioxane	1	280			

Control Limits

ID: 4966 Method: 40 SW846 8260B Prep Method: _____

Matrix: _____ Sample: LCS Instrument: _____ Effective: 07/23/06 Stop: _____ User: VED Type: % Recovery

Other Criteria: None Workorder Profile Client Value: _____

Note: _____

Analytes

Sort	Analyte	Name	LCL	UCL	LWL	UWL	Mean
33	106-46-7	1,4-Dichlorobenzene	81	116			
34	544-10-5	1-Chlorohexane	73	130			
35	594-20-7	2,2-Dichloropropane	64	129			
36	78-93-3	2-Butanone	50	152			
37	110-75-8	2-Chloroethylvinyl ether	1	150			
38	591-78-6	2-Hexanone	65	154			
39	79-46-9	2-Nitropropane	60	138			
40	107-05-1	3-Chloro-1-propene	59	135			
41	108-10-1	4-Methyl-2-Pentanone(MIBK)	71	146			
42	67-64-1	Acetone	40	151			
43	75-05-8	Acetonitrile	19	130			
44	107-02-8	Acrolein	18	183			
45	107-13-1	Acrylonitrile	71	151			
46	100-44-7	Benzyl Chloride	59	130			
47	108-86-1	Bromobenzene	81	119			
48	74-97-5	Bromochloromethane	73	117			

ID: 4966 Method: 40 SW846 8260B Prep Method:
 Matrix: Sample: LCS Instrument: Effective: 07/23/06 Stop: User: VED Type: % Recovery

Other Criteria: None Workorder Profile Client Value:
 Note:

Analytes

Sort	Analyte	Name	LCL	UCL	LWL	UWL	Mean
49	75-27-4	Bromodichloromethane	79	126			
50	75-25-2	Bromoform	70	123			
51	74-83-9	Bromomethane	45	148			
52	75-15-0	Carbon Disulfide	57	131			
53	56-23-5	Carbon Tetrachloride	62	132			
54	108-90-7	Chlorobenzene	85	117			
55	124-48-1	Chlorodibromomethane	77	122			
56	75-00-3	Chloroethane	51	142			
57	67-66-3	Chloroform	78	122			
58	74-87-3	Chloromethane	38	156			
59	126-99-8	Chloroprene	62	125			
60	110-82-7	Cyclohexane	66	130			
61	74-95-3	Dibromomethane	81	125			
62	75-71-8	Dichlorodifluoromethane	17	166			
63	75-43-4	Dichlorofluoromethane	4	121			
64	107-39-1	Diisobutylene	60	137			

Control Limits

ID: 4966 Method: 40 SW846 8260B Prep Method:

Matrix: Sample: LCS Instrument: Effective: 07/23/06 Stop: User: VED Type: % Recovery

Other Criteria: None Workorder Profile Client Value:

Note:

Analytes

Sort	Analyte	Name	LCL	UCL	LWL	UWL	Mean
65	108-20-3	Diisopropyl ether	74	131			
66	141-78-6	Ethyl Acetate	63	138			
67	60-29-7	Ethyl Ether	71	131			
68	97-63-2	Ethyl Methacrylate	74	128			
69	637-92-3	Ethyl tert-butyl ether	75	123			
70	76-13-1	Freon 113	50	130			
71	142-82-5	Heptane	56	130			
72	87-68-3	Hexachlorobutadiene	55	128			
73	67-72-1	Hexachloroethane	62	130			
74	110-54-3	Hexane	48	142			
75	74-88-4	Iodomethane	37	128			
76	78-83-1	Isobutyl alcohol	37	171			
77	67-63-0	Isopropyl Alcohol	5	172			
78	126-98-7	Methacrylonitrile	68	155			
79	80-62-6	Methyl methacrylate	63	135			
80	75-09-2	Methylene Chloride	76	121			

Control Limits

ID: 4966 Method: 40 SWB46 8260E Prep Method:

Matrix: Sample: LCS Instrument: Effective: 07/23/06 Stop: User: VED Type: % Recovery

Other Criteria: None Workorder Profile Client Value:

Note:

Analytes

Sort	Analyte	Name	LCL	UCL	LWL	UWL	Mean
81	98-95-3	Nitrobenzene	23	139			
82	111-65-9	Octane	58	127			
83	109-66-0	Pentane	33	143			
84	107-12-0	Propionitrile	59	158			
85	100-42-5	Styrene	79	123			
86	127-18-4	Tetrachloroethene	72	124			
87	109-99-9	Tetrahydrofuran	62	152			
88	79-01-6	Trichloroethene	77	124			
89	75-69-4	Trichlorofluoromethane	38	123			
90	108-05-4	Vinyl Acetate	58	136			
91	75-01-4	Vinyl Chloride	27	138			
92	156-59-2	cis-1,2-Dichloroethene	78	125			
93	10061-01-5	cis-1,3-Dichloropropene	81	121			
94	71-23-8	n-Propanol	19	342			
95	103-65-1	n-Propylbenzene	74	122			
96	95-49-8	o-Chlorotoluene	78	126			

Control Limits

ID: 4966 Method: 40 SW846 8260E Prep Method:

Matrix: Sample: LCS Instrument: Effective: 07/23/06 Stop: User: VED Type: % Recovery

Other Criteria: None Workorder Profile Client Value:

Note:

Analytes

Sort	Analyte	Name	LCL	UCL	LWL	UWL	Mean
97	106-43-4	p-Chlorotoluene	78	125			
98	99-87-6	p-Isopropyltoluene	72	123			
99	135-98-8	sec-Butylbenzene	72	127			
100	994-05-8	tert-Amyl methyl ether	75	121			
101	98-06-6	tert-Butylbenzene	72	124			
102	156-60-5	trans-1,2-Dichloroethene	71	122			
103	10061-02-6	trans-1,3-Dichloropropene	78	126			
104	110-57-6	trans-1,4-Dichloro-2-butene	60	141			
105	1868-53-7	Dibromofluoromethane	78	116			
106	17060-07-0	1,2-Dichloroethane-d4	62	133			
107	2037-26-5	Toluene-d8	76	127			
108	460-00-4	4-Bromofluorobenzene	79	114			
109	108-87-2	Methyl cyclohexane	70	130			
110	79-20-9	Methyl acetate	70	130			
111	540-59-0	1,2-Dichloroethene, Total	78	125			
112	75-85-4	tert-Amyl Alcohol	70	130			

Control Limits

ID: 4966 Method: 40 SWB46 8260B Prep Method:

Matrix: Sample: LCS Instrument: Effective: 07/23/06 Stop: User: VED Type: % Recovery

Other Criteria: None Workorder Profile Client Value:

Note:

Analytes

Sort	Analyte	Name	LCL	UCL	LWL	UWL	Mean
98	89-87-6	Propylbenzene	72	123			
99	135-98-8	sec-Butylbenzene	72	127			
100	994-05-8	tert-Amyl methyl ether	75	121			
101	98-06-6	tert-Butylbenzene	72	124			
102	156-60-5	trans-1,2-Dichloroethene	71	122			
103	10061-02-6	trans-1,3-Dichloropropene	78	126			
104	110-57-6	trans-1,4-Dichloro-2-butene	60	141			
105	1868-53-7	Dibromofluoromethane	78	116			
106	17060-07-0	1,2-Dichloroethane-d4	62	133			
107	2037-26-5	Toluene-d8	76	127			
108	460-00-4	4-Bromofluorobenzene	79	114			
109	108-87-2	Methyl cyclohexane	70	130			
110	79-20-9	Methyl acetate	70	130			
111	540-58-8	1,2-Dichloroethene, Total	78	125			
112	75-85-4	tert-Amyl Alcohol	78	130			
113	919-94-8	tert-Amyl Ethylether	70	130			

Control Limits

ID: 4971 Method: 40 SW846 8260B Prep Method:

Matrix: Sample: MSD Instrument: Effective: 07/23/06 Stop: User: VED Type: RPD

Other Criteria: None Workorder Profile Client Value:

Note:

Analytes

Sort	Analyte	Name	LCL	UCL	LWL	UWL	Mean
1	75-71-8	Dichlorodifluoromethane	0	24			
2	74-87-3	Chloromethane	0	27			
3	75-01-4	Vinyl Chloride	0	40			
4	74-83-9	Bromomethane	0	26			
5	75-00-3	Chloroethane	0	24			
6	75-43-4	Dichlorofluoromethane	0	22			
7	75-69-4	Trichlorofluoromethane	0	23			
8	109-66-0	Pentane	0	24			
9	60-29-7	Ethyl Ether	0	19			
10	107-02-8	Acrolein	0	40			
11	75-35-4	1,1-Dichloroethene	0	21			
12	76-13-1	Freon 113	0	26			
13	67-64-1	Acetone	0	40			
14	74-88-4	Iodomethane	0	27			
15	75-15-0	Carbon Disulfide	0	28			
16	107-05-1	3-Chloro-1-propene	0	18			

Control Limits

ID: 4971 Method: 40 SW846 8260B Prep Method:

Matrix: Sample: MSD Instrument: Effective: 07/23/06 Stop: User: VED Type: RPD

Other Criteria: None Workorder Profile Client Value:

Note:

Analytes

Sort	Analyte	Name	LCL	UCL	LWL	UWL	Mean
17	75-09-2	Methylene Chloride	0	17			
18	75-65-0	tert - Butyl Alcohol	0	40			
19	107-13-1	Acrylonitrile	0	16			
20	156-60-5	trans-1,2-Dichloroethene	0	22			
21	1634-04-4	Methyl t-Butyl Ether	0	20			
22	110-54-3	Hexane	0	22			
23	75-34-3	1,1-Dichloroethane	0	15			
24	108-05-4	Vinyl Acetate	0	17			
25	126-99-8	Chloroprene	0	18			
26	594-20-7	2,2-Dichloropropane	0	18			
27	156-59-2	cis-1,2-Dichloroethene	0	21			
28	78-93-3	2-Butanone	0	16			
29	74-97-5	Bromochloromethane	0	19			
30	109-99-9	Tetrahydrofuran	0	20			
31	67-66-3	Chloroform	0	16			
32	71-55-6	1,1,1-Trichloroethane	0	20			

Control Limits

ID: 4971 Method: 40 SWB46 8260R Prep Method: _____

Matrix: _____ Sample: MSD Instrument: _____ Effective: 07/23/06 Stop: _____ User: VED Type: RPD

Other Criteria: None Workorder Profile Client Value: _____

Note: _____

Analytes

Sort	Analyte	Name	LCL	UCL	LWL	UWL	Mean
33	110-82-7	Cyclohexane	0	20			
34	56-23-5	Carbon Tetrachloride	0	17			
35	563-58-6	1,1-Dichloropropene	0	16			
36	71-43-2	Benzene	0	26			
37	107-06-2	1,2-Dichloroethane	0	19			
38	142-82-5	Heptane	0	26			
39	107-39-1	Diisobutylene	0	20			
40	79-01-6	Trichloroethene	0	18			
41	78-87-5	1,2-Dichloropropane	0	15			
42	74-95-3	Dibromomethane	0	16			
43	75-27-4	Bromodichloromethane	0	16			
44	79-46-9	2-Nitropropane	0	17			
45	110-75-8	2-Chloroethylvinyl ether	0	40			
46	10061-01-5	cis-1,3-Dichloropropene	0	16			
47	108-10-1	4-Methyl-2-Pentanone(MIBK)	0	16			
48	108-88-3	Toluene	0	20			

ID: 4971 Method: 40 SW846 8260B Prep Method:
 Matrix: Sample: MSD Instrument: Effective: 07/23/06 Stop: User: VED Type: RPD

Other Criteria: None Workorder Profile Client Value:
 Note:

Analytes

Sort	Analyte	Name	LCL	UCL	LWL	UWL	Mean
49	111-85-9	Octane	0	40			
50	10061-02-6	trans-1,3-Dichloropropene	0	18			
51	542-75-6	1,3-Dichloropropene, Total	0	16			
52	79-00-5	1,1,2-Trichloroethane	0	15			
53	127-18-4	Tetrachloroethene	0	38			
54	142-28-9	1,3-Dichloropropane	0	15			
55	591-78-6	2-Hexanone	0	17			
56	124-48-1	Chlorodibromomethane	0	15			
57	106-93-4	1,2-Dibromoethane	0	19			
58	108-90-7	Chlorobenzene	0	15			
59	544-10-5	1-Chlorohexane	0	17			
60	630-20-6	1,1,1,2-Tetrachloroethane	0	16			
61	100-41-4	Ethylbenzene	0	19			
62	108383/106423	mp-Xylene	0	21			
63	95-47-6	o-Xylene	0	19			
64	1330-20-7	Total Xylenes	0	35			

Control Limits

ID: 4971 Method: 40 SW846 82608 Prep Method:

Matrix: Sample: MSD Instrument: Effective: 07/23/06 Stop: User: VED Type: RPD

Other Criteria: None Workorder Profile Client Value:

Note:

Analytes

Sort	Analyte	Name	LCL	UCL	LWL	UWL	Mean
65	100-42-5	Styrene	0	16			
66	75-25-2	Bromoform	0	16			
67	98-82-8	Isopropylbenzene	0	18			
68	79-34-5	1,1,2,2-Tetrachloroethane	0	16			
69	108-86-1	Bromobenzene	0	17			
70	96-18-4	1,2,3-Trichloropropane	0	19			
71	110-57-6	trans-1,4-Dichloro-2-butene	0	18			
72	103-65-1	n-Propylbenzene	0	20			
73	95-49-8	o-Chlorotoluene	0	17			
74	108-67-8	1,3,5-Trimethylbenzene	0	18			
75	106-43-4	p-Chlorotoluene	0	16			
76	98-06-6	tert-Butylbenzene	0	17			
77	95-63-6	1,2,4-Trimethylbenzene	0	24			
78	135-98-8	sec-Butylbenzene	0	17			
79	541-73-1	1,3-Dichlorobenzene	0	16			
80	99-87-6	p-Isopropyltoluene	0	17			

Control Limits

ID: 4971 Method: 40 SW846 8260B Prep Method:

Matrix: Sample: MSD Instrument: Effective: 07/23/06 Stop: User: VED Type: RPD

Other Criteria: None Workorder Profile Client Value:

Note:

Analytes

Sort	Analyte	Name	LCL	UCL	LWL	UWL	Mean
95	09-95-2	Methoxybenzene	0	37			
96	437-12-0	Propionitrile	0	23			
97	123-91-1	1,4-Dioxane	0	40			
98	141-78-6	Ethyl Acetate	0	18			
99	126-98-7	Methacrylonitrile	0	16			
100	97-63-2	Ethyl Methacrylate	0	16			
101	71-23-8	n-Propanol	0	40			
102	67-63-0	Isopropyl Alcohol	0	40			
103	78-83-1	Isobutyl alcohol	0	40			
104	79-20-9	Methyl acetate	0	18			
105	108-87-2	Methyl cyclohexane	0	18			
106	65-85-0	Benzoic acid	0	40			
107	994-05-8	tert-Amyl methyl ether	0	40			
108	540-59-0	1,2-Dichloroethene, Total	0	40			
109	75-85-4	tert-Amyl Alcohol	0	40			
110	919-94-8	tert-Amyl Ethylether	0	40			

Maintenance

Daily:

Check the interior of the main compartment for leaks and spills. Isolate and repair all leaks.
Clean up any spills or dried reagents inside the main compartment, on top of the instrument, or inside the autosampler.
Check the junction between the pump head and the metal pump casting for liquid leaks. If leak occurs replace piston seals.

Weekly:

Check air lines for crimping or discoloration. Relocate any pinched lines and replace damaged lines. Also check liquid lines for crimping or damage.
Inspect guard column frit for discoloration, replace as needed.

Monthly:

Clean trays for autosampler. Clean the inside of autosampler.
Inspect and/or clean injection valve.
Inspect sample tip for blockage. Replace as needed.
Inspect eluent uptake filter, rinse if discoloration is present.
Check line into instrument from autosampler for blockage. Check waste line out of instrument for blockage.

Quarterly:

Inspect waste lines for blockage or pinching caused by ferrules or union connections.
Inspect autosampler valve port faces for blockage. Clean or replace as needed.

Yearly:

Replace eluent uptake filter.
Rebuild injection valve.
Replace lines between autosampler and instrument. Replace lines between columns. Replace waste line out of instrument.



DEPARTMENT OF THE NAVY

NAVAL FACILITIES ENGINEERING SERVICE CENTER
1100 23RD AVE
PORT HUENEME CA 93043-4370

IN REPLY REFER TO:
NFESC 413
December 4, 2007

Ms. Helen MacMinn
Analytical Laboratory Services, Inc.
Quality Assurance Manager
34 Dogwood Lane
Middletown, PA 17057

Dear Ms. MacMinn,

This correspondence addresses the status of Analytical Laboratory Services, Inc. of Middletown, PA in the Navy Environmental Restoration (ER) Quality Assurance (QA) Program as administered by the Naval Facilities Engineering Service Center (NFESC).

Your laboratory is accepted to perform sample analysis for the methods listed in Table 1. The period of acceptance expires February 28, 2008. This acceptance does not guarantee the delivery of any analytical samples. Acceptance is facility specific and can not be transferred to an affiliated or subcontract laboratory.

The Navy's assessment included a review of the laboratory's QA manual, selected standard operating procedures (SOPs) and SOP master list, list of major analytical instrumentation, performance test (PT) results and onsite assessment documentation¹.

The Navy reserves the right to conduct additional laboratory assessments or to suspend or revoke acceptance status for any or all of the listed parameters if deemed necessary.

Table 1

METHOD	PARAMETER	MATRIX
300.0/9056	Anions: Chloride, Fluoride, Sulfate, Nitrate, Nitrite and Ortho-phosphate	Water/Solid
314.0	Perchlorate	Water
6020/6010B 7000 Series	TAL Metals: Aluminum, Antimony, Arsenic, Barium, Beryllium, Cadmium, Calcium, Chromium, Cobalt, Copper, Iron, Lead, Magnesium, Manganese, Mercury, Molybdenum, Nickel, Potassium, Selenium, Silver, Sodium, Thallium, Vanadium, and Zinc	Water/Solid
8015B	Gasoline Range Organics/Diesel Range Organics by Gas Chromatography	Water/Solid
8021	Halogenated and Aromatic Volatiles	Water/Solid
8081A	Organochlorine Pesticides by Gas Chromatography	Water/Solid

¹ A Navy onsite assessment was conducted on November 28-30, 2007.

NFESC 413
December 4, 2007

8082	Polychlorinated Biphenyls (PCBs) by Gas Chromatography	Water/Solid/Oil
8141/8151A	Herbicides by Gas Chromatography	Water/Solid
8260B	Volatile Organic Compounds by GC/MS	Water/Solid
8270C/8270SIM	Semivolatile Organic Compounds by GC/MS	Water/Solid
8330	Explosives by HPLC	Water/Solid
9012A/9014	Total and Amenable Cyanides by Midi-Distillation	Water/Solid

Acceptance for use for parameters not identified on the table will be determined by Navy project personnel.

The laboratory should notify NFESC if there are parameters not presented on Table 1 that the laboratory expects to run on a routine basis in support of Navy environmental restoration projects. In these circumstances the laboratory's capability to run the tests will be reviewed and the table will be modified accordingly.

Questions concerning the information provided should be directed to the NFESC IR QA Program coordinator, Ms. Patricia Moreno at (805) 982-1659, or via email at pati.moreno@navy.mil.

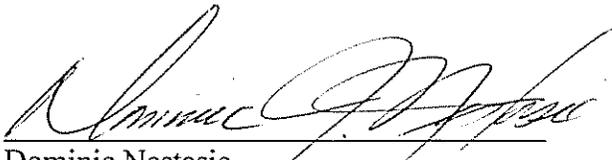
Sincerely,

Robert J. Kratzke
Supervisor, Consultation/Information
Management Branch

Microseeps, Inc.

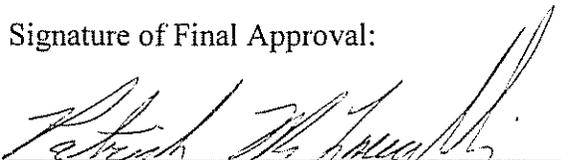
Standard Operating Procedure for Waste Disposal

Controlled Copy No. _____



Dominic Nestasie
Laboratory Director

Signature of Final Approval:



Patrick McLoughlin, PhD.
Technical Director

1-9-04
Date

1.0 Introduction

The purpose of this waste disposal program is to provide compliance with the Federal Resource Conservation and Recovery Act (RCRA) in order to identify, handle, store, treat, and ultimately dispose of laboratory waste. This waste includes analytical process waste, unused sample aliquots, and laboratory hazardous waste, including samples that have been identified as hazardous according to their characteristics or identified as listed hazardous wastes (See Section 3.1 for definitions).

The preferred option is to avoid generating hazardous waste if at all possible. Procedures for waste minimization including inventory control, recovery of materials for reuse, and neutralization to render corrosive wastes non-hazardous and amenable to sanitary sewage disposal are discussed. Another method of waste reduction is returning hazardous samples to the generator upon agreement between the client and the laboratory.

2.0 Responsibility

2.1 Hazardous Waste Coordinator

The only employee authorized to remove any waste from any laboratory is the hazardous waste coordinator. The hazardous waste coordinator is responsible for labeling and moving all containers of hazardous waste into the central storage area. The hazardous waste coordinator is responsible for tracking all hazardous waste in the laboratory and determining and documenting monthly accumulation amounts.

The Hazardous Waste Coordinator shall sample all accumulated sample waste that is unidentified and submit it to the laboratory with a completed chain of custody for a waste characterization analysis. The hazardous waste coordinator is responsible for coordinating waste disposal with an authorized waste hauler. The Waste Coordinator shall obtain the analytical results from the Laboratory Director and submit them to the waste hauler for final waste classification prior to transporting for disposal.

2.2 Laboratory Director

The Laboratory Director or his designee is responsible for notifying the Waste Coordinator of the results of any samples that have hazardous levels of contaminants.

2.3 Lead Analysts

The Lead Analysts are responsible for segregating hazardous samples from non-hazardous at the direction of the Waste Coordinator. The Lead Analysts are responsible for disposing of analyzed samples that are non-hazardous.

2.4 Executive Vice President and Operations Officer

The Executive Vice President shall sign all hazardous waste manifests.

3.0 Definitions

Hazardous wastes are classified according to type and characteristic. In order for hazardous waste to be handled safely and according to regulations, all generated waste must be classified using the EPA hazardous waste codes from 40 CFR 261. There are two types of Hazardous Waste according to the Resource Conservation and Recovery Act (RCRA). They are: listed wastes and characteristic wastes.

3.1 Listed Wastes

There are four classifications of listed wastes (listed in 40 CFR 261):

F Listed Wastes: Hazardous wastes from non-specific sources such as halogenated solvents used in degreasing. These wastes are assigned a hazardous waste number by the EPA beginning with the letter F. (40 CFR 261.31)

K Listed Wastes: Hazardous wastes from specific sources such as wastewater treatment sludge from the production of zinc yellow pigments. These wastes are assigned a hazardous waste number by the EPA beginning with the letter K. (40 CFR 261.32)

P Listed Wastes: Acutely hazardous discarded commercial chemical products, off-specification species, container residues, and spill residues thereof such as an expired container of potassium cyanide. These wastes are assigned a hazardous waste number by the EPA beginning with the letter P (40 CFR 261.33).

U Listed Wastes: Toxic hazardous discarded commercial chemical products, off-specification species, container residues, and spill residues thereof such as an expired container of methylene chloride. These wastes are assigned a hazardous waste number by the EPA beginning with the letter U. (40 CFR 261.33)

3.2 Characteristic Wastes

Wastes that are classified due to their hazard characteristics are classified using the letter D. Characteristic hazardous wastes are defined as follows:

Ignitability: a waste is hazardous if a sample has any of the following properties:

(1) a liquid that has a flash point less than 140° F (60 C); (2) not a liquid and is capable, under Standard Temperature and Pressure (STP) of causing fire through friction, absorption of moisture or spontaneous chemical changes and, when ignited, burns so vigorously and

persistently that it creates a hazard; (3) an ignitable compressed gas; or (4) an oxidizer. These wastes have the EPA Hazardous Waste Number of D001.

Corrosivity: a liquid that has a pH less than or equal to 2 or greater than or equal to 12.5. These wastes have the EPA Hazardous Waste Number of D002.

Reactivity: A waste is hazardous due to the characteristic of reactivity if a substance has any of the following properties: (1) is normally unstable and readily undergoes violent change without detonating; (2) reacts violently with water; (3) forms potentially explosive mixtures with water; (4) when mixed with water, generates toxic gases; (5) a cyanide or sulfide bearing waste; (6) capable of detonation if subjected to a strong initiating source or heated under confinement; (7) capable of detonation or explosive decomposition or reaction at STP; or (8) it is a forbidden, class A or B explosive according to the DOT. These wastes have the EPA Hazardous Waste Number of D003.

Toxicity: If an extract from a sample of the waste meets or exceeds the concentrations listed in 40 CFR Table 1 of 261.24, the waste is considered hazardous due to the characteristic of toxicity. These wastes have the EPA Hazardous Waste Numbers of D004 through D043.

Accumulation Date: The date that the first drop of waste gets placed in a storage container in a hazardous waste storage area.

4.0 Waste Minimization

4.1 Inventory Control

Chemicals shall be ordered in the smallest quantities possible. It is important that the reagents are used up prior to their expiration date. In most cases, at the discretion of the lead analyst, the "first in, first out" rule will be followed. In other words, older reagents should be used up before a newer container is opened. This procedure will virtually eliminate the expense of disposing of expired or off-specification chemicals.

4.2 Material Recovery

The situations are rare when reuse of a chemical or reagent can occur due to the nature of the analytical laboratory business and cross-contamination issues, but when possible, chemicals and/or reagents will be recovered and reused.

4.3 Neutralization

According to the regulations governing the Resource Conservation and Recovery Act, neutralization as a form of waste pre-treatment is acceptable and does not require a permit. Therefore, Microseeps has adopted the policy of neutralizing wastes that are hazardous due either wholly or in part to the characteristic of corrosivity. These wastes shall be neutralized by

pre-treating both acids and bases with bicarbonate of soda or pre-treating acids with sodium carbonate to a pH of not less than 6 and not greater than 9. Those samples only deemed hazardous by the characteristic of corrosivity may be disposed in the sanitary sewer using copious amounts of water. Samples that are hazardous due to other characteristics are containerized for disposal by an authorized waste disposal company.

4.4 Mixing Waste

- Containers of same type wastes are to be consolidated into the smallest container possible to minimize the amount of disposal costs.
- Liquid and solid wastes shall not be combined. If wastes are being mixed from the same or different projects the wastes MUST be compatible and the container MUST be marked with the Proper Waste Identification Code and Accumulation Date.
- Sorbant materials, gloves, and other materials used in hazmat spill cleanups are to be decontaminated after use, if possible. Those items that cannot be decontaminated shall be disposed of as a hazardous waste.
- If wastes are being collected and segregated for disposal based on a suspicion of a particular hazard, do not combine any of the wastes. Keep wastes from each sample separate until the hazards are known. It is possible that only some of the samples are hazardous. If hazardous wastes and non-hazardous wastes are combined, the entire mixture is then deemed hazardous and must be disposed of as a hazardous waste.

5.0 Hazardous Waste Identification

There are three distinct types of materials generated at Microseeps that may fall into the hazardous waste category and they are analytical samples, analytical process waste, expired or off-specification laboratory chemicals. See Exhibit 1 for flow diagrams that outline the decision process for waste identification and final disposition of samples and analytical process waste.

5.1 Identification of Analytical Samples that are Hazardous

The Waste Coordinator shall check each project in the LIMS to see if analyzed samples contain any listed waste or are hazardous by characteristics. The waste coordinator will then segregate the hazardous samples from the non-hazardous. Analytical samples that are deemed hazardous will be stored in drums in the hazardous waste storage area in the UPARC facility. Those containers will be labeled with the appropriate hazardous warning labels, Microseeps name and the accumulation date.

5.2 Identification of Hazardous Process Waste

Analytical process waste that is deemed hazardous either by characteristic or by listing shall be placed into a compatible waste container and labeled with all applicable EPA Hazardous Waste Numbers and the accumulation date.

5.3 Unused, Expired, or Off Specification Laboratory Chemicals

When a hazardous chemical or reagent can no longer be used because it has expired or there is a question about its purity, the Hazardous Waste Coordinator shall be notified. The Hazardous Waste Coordinator shall see that the chemical is labeled with all applicable EPA Hazardous Waste Numbers, and the accumulation date. The Hazardous Waste Coordinator shall remove the chemical from the laboratory, store it in the hazardous waste storage cabinet, and include it in the next scheduled waste pick-up.

6.0 Hazardous Waste Handling

6.1 Administrative Controls

Limiting access only to designated individuals who have specific responsibilities concerning waste shall control exposure to hazardous waste. Safe work practices shall be applied such as limiting the handling of the waste to the Waste Coordinator. Each laboratory has one designated spot for accumulating waste and that waste shall be removed on a weekly basis. The Hazardous Waste Coordinator shall add those wastes to the monthly accumulation report and turn it in to the Quality Systems Coordinator at the end of each month.

6.2 Engineering Controls

Hazardous waste that poses a hazard by inhalation shall be handled under a fume hood with the sash at the designated height. All hazardous waste shall be stored in properly ventilated cabinets until removed to the hazardous waste storage area.

All glass containers that hold waste must be transported in unbreakable carrying cases. Any containers of waste that weigh more than twenty pounds shall be transported or moved by using either a regular dolly, a drum dolly, or a wheeled cart.

6.3 Personal Protection

Personal Protective Equipment must be worn while handling hazardous waste. All employees who handle hazardous waste shall wear safety glasses and chemical resistant gloves.

7.0 Hazardous Waste Storage

All waste that is deemed hazardous shall be stored in compatible containers until prepared by the waste hauler for disposal. The Waste Coordinator shall label all waste as it is placed into the hazardous waste storage area with Microseeps name and the Accumulation Date.

The hazardous waste storage cabinet shall be appropriately marked with a sign denoting the space as hazardous waste storage. All hazard-warning signs that apply shall be posted. The hazardous waste storage cabinet is equipped with secondary containment vessels and proper ventilation. Incompatible waste is segregated so that in the event of leakage or a spill there can be no adverse reaction. Once waste is removed from the laboratories, it shall be stored in the hazardous waste storage cabinet or the UPARC hazardous waste storage area until disposal by an authorized waste hauler.

The Hazardous Waste Coordinator must inspect the hazardous waste storage area weekly, and inspection documentation (see Exhibit 2) shall be kept on file by the Waste Coordinator.

7.1 Accumulation Amounts

Microseeps generates less than 100 kilograms of hazardous waste per month and is designated as a conditionally exempt small quantity generator (CESQG). Total hazardous waste accumulation shall never exceed 1000 kilograms under any circumstances.

As soon as 900 kilograms are accumulated, or the hazardous waste storage cabinet is almost full (as long as the amount does not exceed 900 kilograms) an authorized waste hauler will be contacted to transport the waste to a designated treatment facility.

8.0 Disposal of Laboratory Chemicals and Samples

All hazardous wastes shall be moved to the hazardous waste storage cabinet or UPARC hazardous waste storage area to await disposal by an authorized hazardous waste hauler.

8.1 Treatment of Waste that are Hazardous due only to Corrosivity

Wastes that are generated by the laboratory that have a pH of less than 2 and greater than 12.5 shall be neutralized to a pH of not less than 6 and not greater than 9 prior to sewer disposal using the following procedure. Acids and bases shall always be neutralized separately.

- Put on safety glasses, inner gloves, and over gloves (pvc).
- Place a large plastic bucket into a fume hood and turn the fume hood on.
- Set the sash height according to the arrows on the front of the hood.

- Place approximately 1 liter of tap water into the bucket.
- Slowly pour the acid or the base into the bucket.
- Add soda ash (acids) or sodium bicarbonate (acids or bases) approximately a tablespoon at a time, allowing the reaction to stop prior to adding more.
- Check the pH frequently.
- When the pH meets UPARC's specifications, the solution is amenable to sewer disposal according to the following instructions.

8.2 Sewer Disposal

The only laboratory chemicals and waste that are amenable to sewer disposal are the ones that are deemed non-hazardous, or corrosive wastes that have been treated. Non-hazardous aqueous samples are amenable to sewer disposal. Any time samples or neutralized wastes are disposed using the sanitary sewer; the tap shall be run for a minimum of fifteen minutes.

8.2.1 Procedure for Sewer Disposal of Corrosives with pH not less than 6 and not greater than 9

In order to comply with Microseeps' disposal agreement with Oxford Development, the following procedure shall be used to dispose of dilute corrosives using the sanitary sewer.

- Turn on the cold water in the sink where the waste will be poured 5 minutes prior to the start of disposal to flush the lines.
- While keeping the cold water on, slowly pour the waste into the sink. Triple-rinse the waste container. The container can then be placed in the trash.
- When all of the corrosive wastes have been disposed, keep the water running for an additional 15 minutes to flush the lines before turning off the water.

8.3 Hazardous Waste Disposal

All hazardous waste shall be disposed in accordance with the regulations outlined in 40 CFR Part 262 Subpart B – The Manifest. The contracted waste hauler will complete the manifest and submit it to the Waste Coordinator for inspection and signature. It is the Waste Coordinator's responsibility to ensure that the manifest is complete and correct.

The Waste Coordinator must:

- Obtain the handwritten signature of the Executive Vice President.

- Obtain the signature of the initial transporter and date of acceptance on the manifest.
- Retain one copy of the manifest.
- Give the transporter the remaining copies.
- Give the manifest to the Quality Systems Coordinator for filing.

When packaged for transportation and disposal, the contracted waste hauler will label the container with the proper Department of Transportation label according to hazard classification, and insure that the package is prepared for Transportation in accordance with 49 CFR.

9.0 Record keeping

The Quality Systems Coordinator shall do the following:

- Keep a copy of each signed manifest until Microseeps receives the signed copy from the designated facility that received the waste. This signed copy must be retained as a record for at least three years from the date that the waste was accepted by the initial transporter.
- Keep on file all analytical results relating to the disposed waste a minimum of three years from the date that the waste was accepted by the initial transporter.
- Contact the waste hauler if the completed and signed manifest is not returned from the designated facility that was to receive the waste within 45 days to inquire about the dispensation of the waste.

10.0 References

40 Code of Federal Regulations, Protection of Environment, Parts 260 to 299. Office of the Federal Register National Archives and Records Administration.

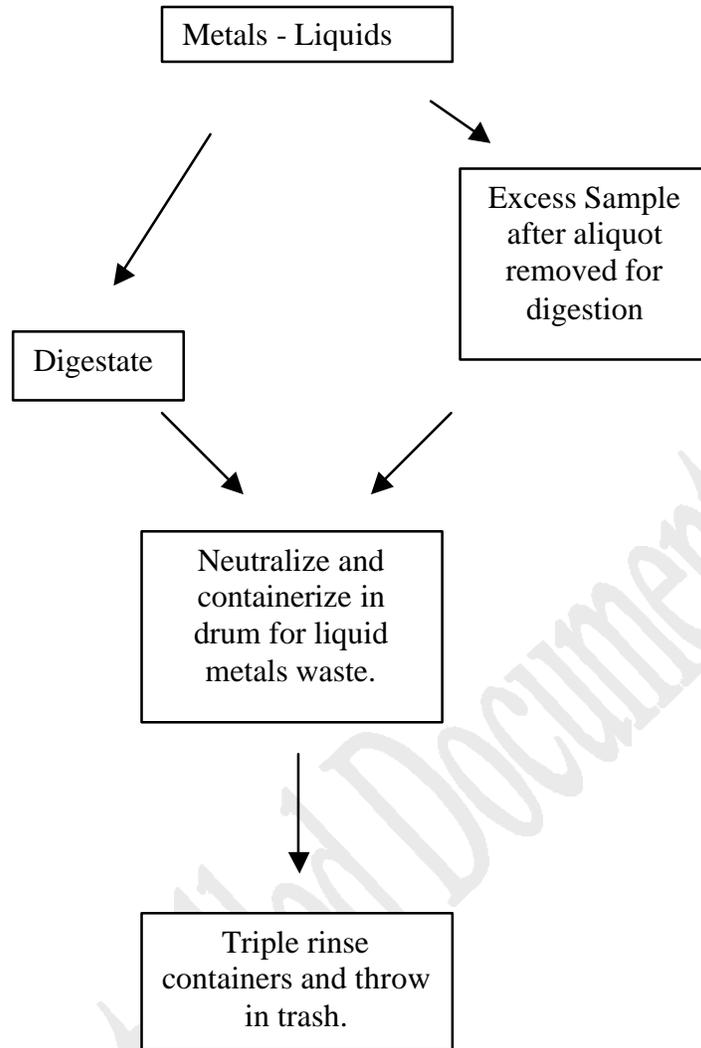
SOP Review Date: June 15, 2005

Exhibit 1

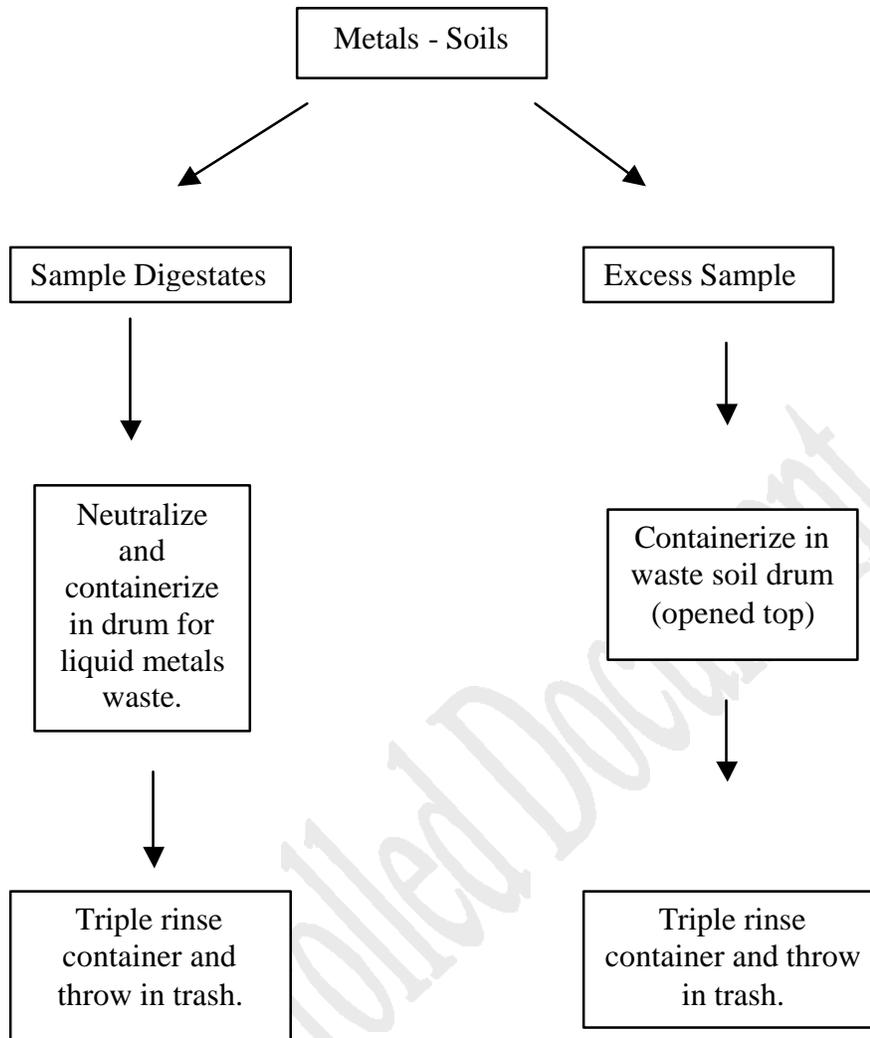
Process Flow Diagrams for Hazardous and Non-hazardous Waste and Sample Disposal

Controlled Document

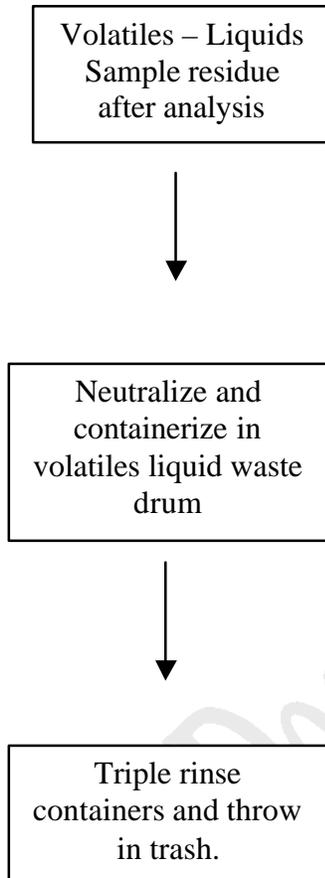
Disposal Flowchart of Water Samples Received for Metals Analysis



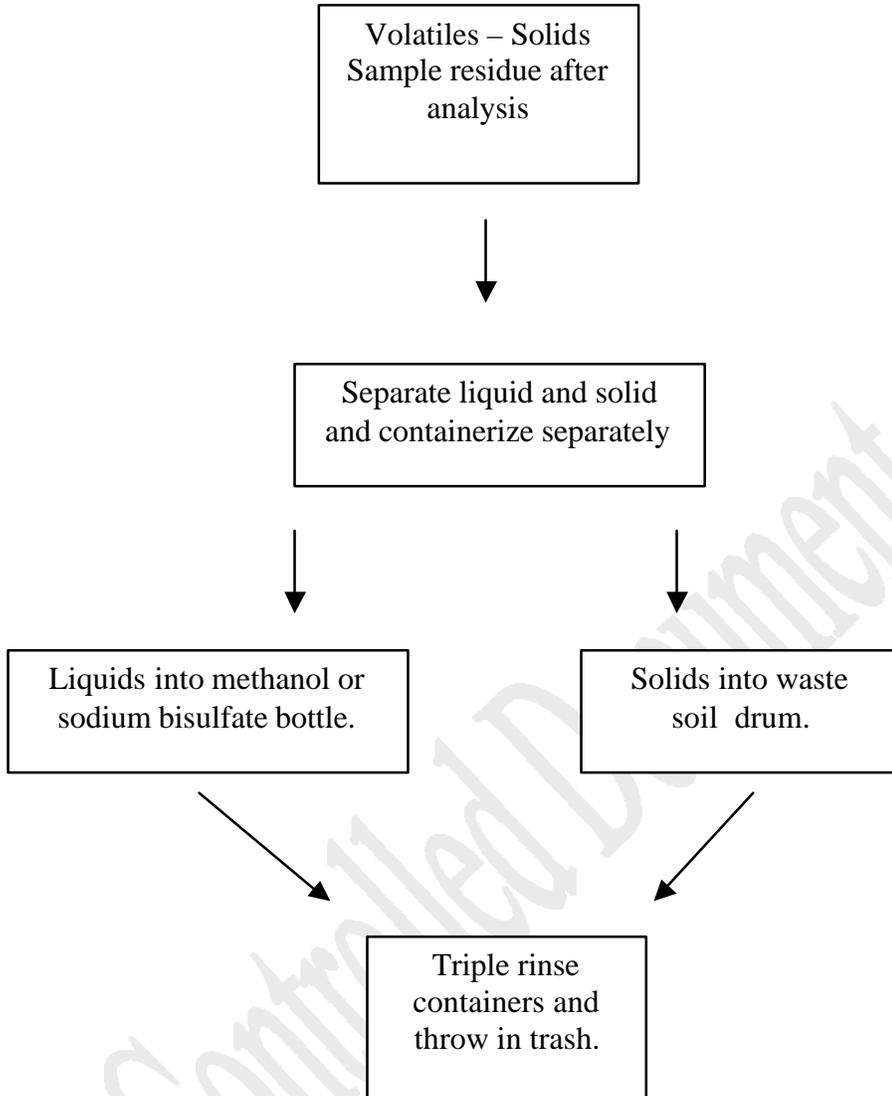
Disposal Flowchart of Soil Samples Received for Metals Analysis



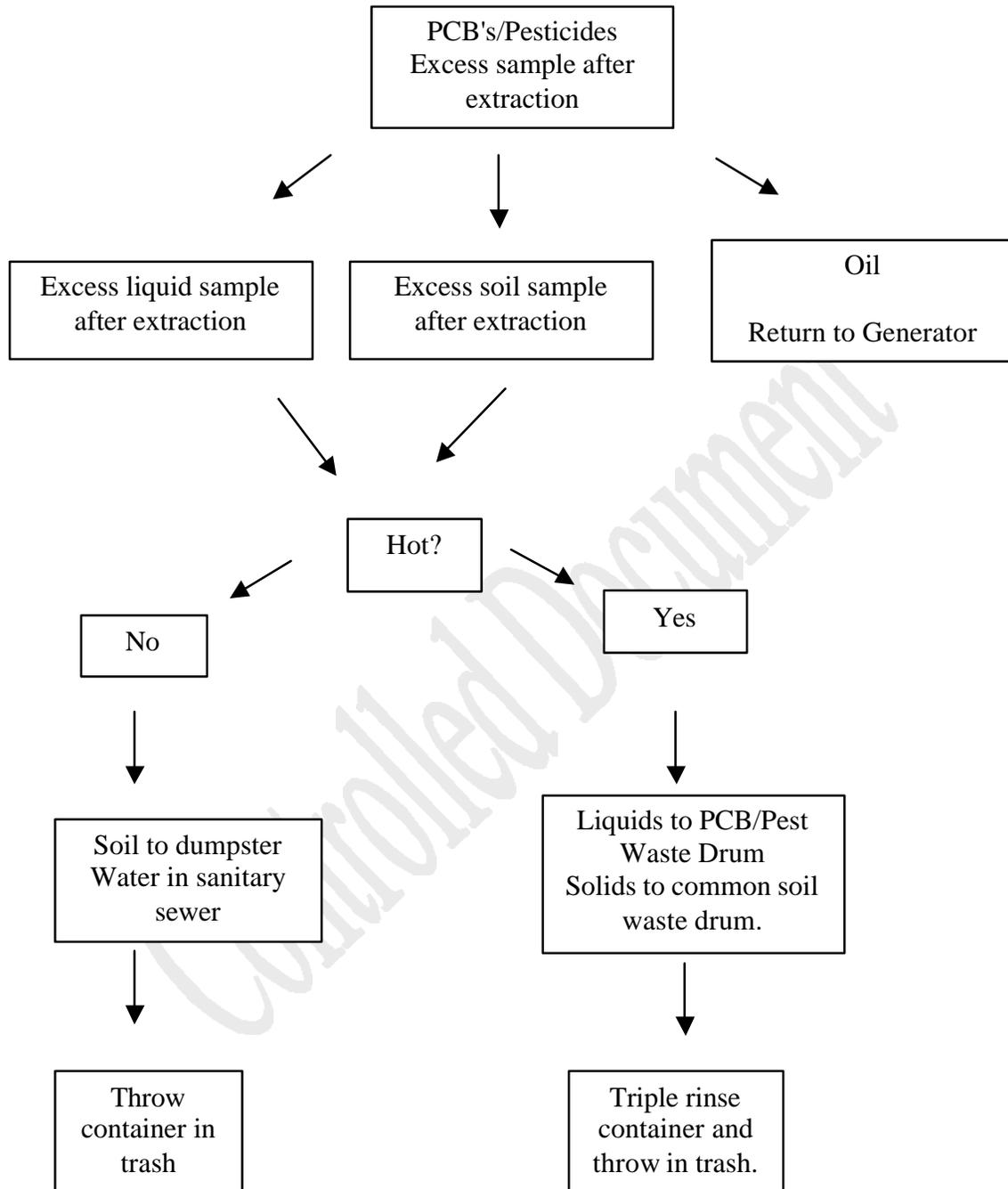
Disposal Flowchart for Water Samples Received for Volatiles Analysis



Disposal Flowchart for Soil Samples Received for Volatiles Analysis



Disposal Flowchart for Unused (un-extracted) Portions of Samples Received for PCB/Pesticide Analysis



Disposal Flowchart for PCB/Pesticide Extracts

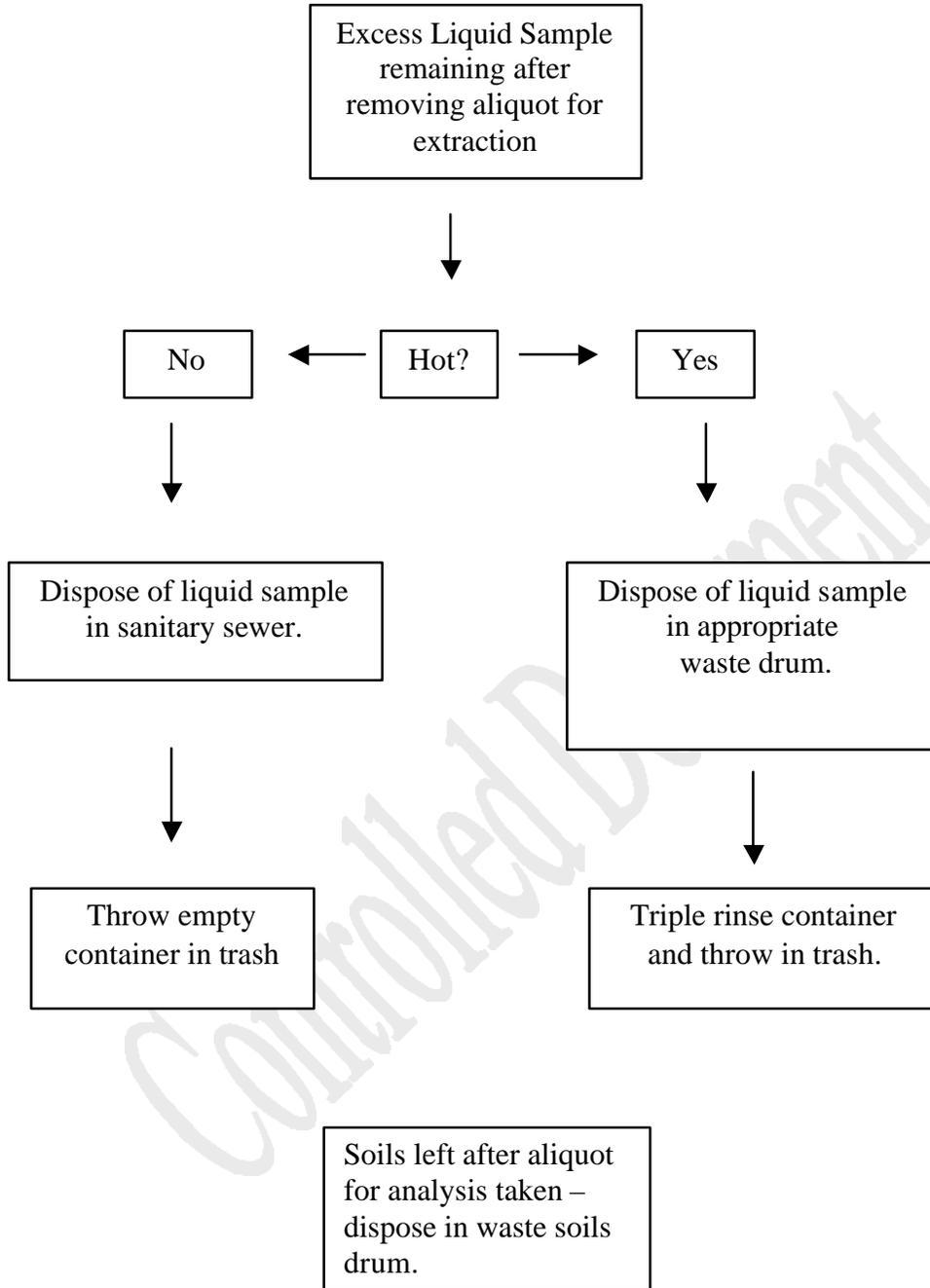
PCB's/Pesticides
Extracts after
sample analysis
(Hexane)



Containerize
autosampler vials in
5-gal. bucket with
lid fastened tightly.
(also labeled
flammable)

Controlled Document

Disposal Flowchart for Excess Samples Received for BNA Analysis



Disposal Flowchart for BNA Extracts

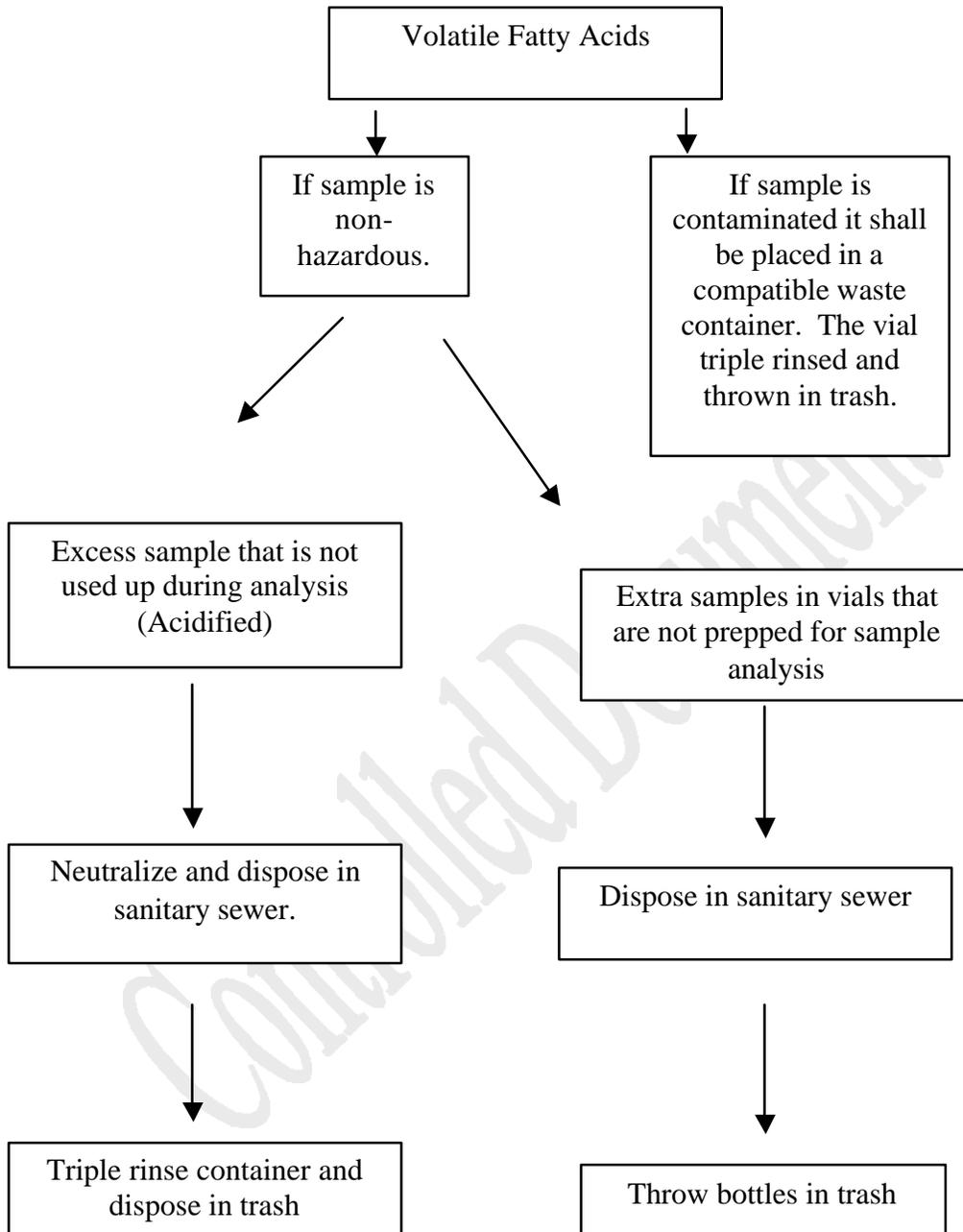
BNA Extracts after
analysis
(methylene chloride)



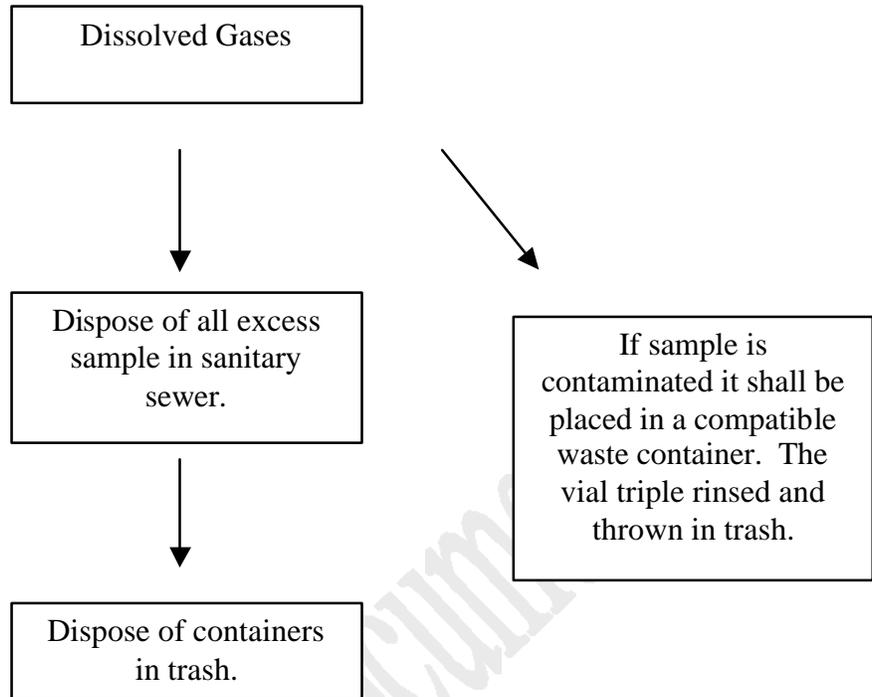
Collect autosampler vials in
Ziploc baggie. Do not open
vials. Place vials in 5 gallon
bucket with lid tightly sealed.
labeled with proper hazardous
waste code and date.

Controlled Document

Disposal Flowchart for Samples Received for VFA Analysis

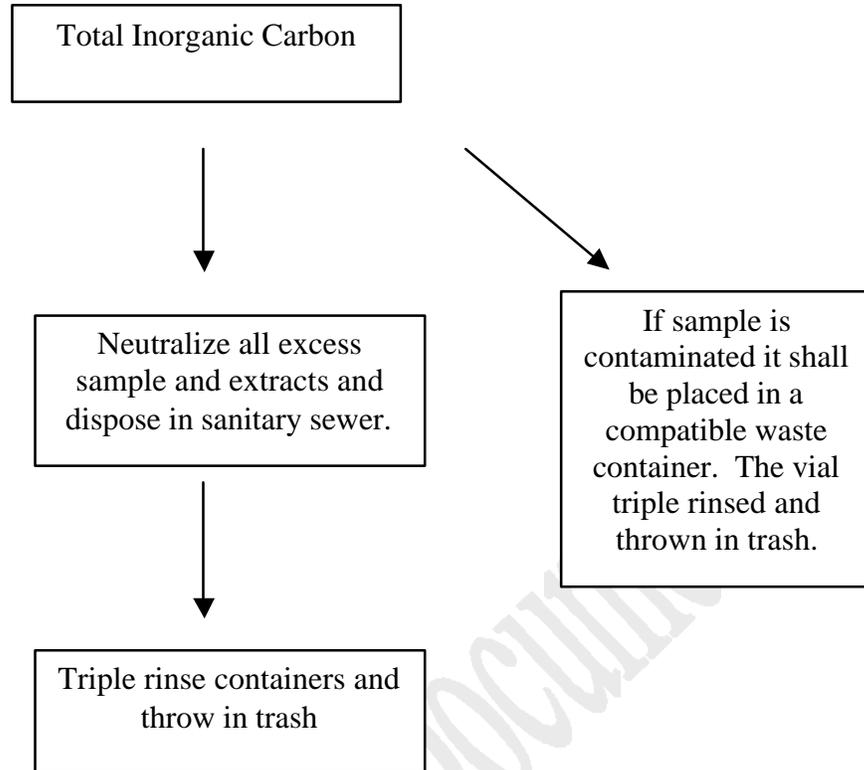


Disposal Flowchart for Samples Received for Dissolved Gas Analysis



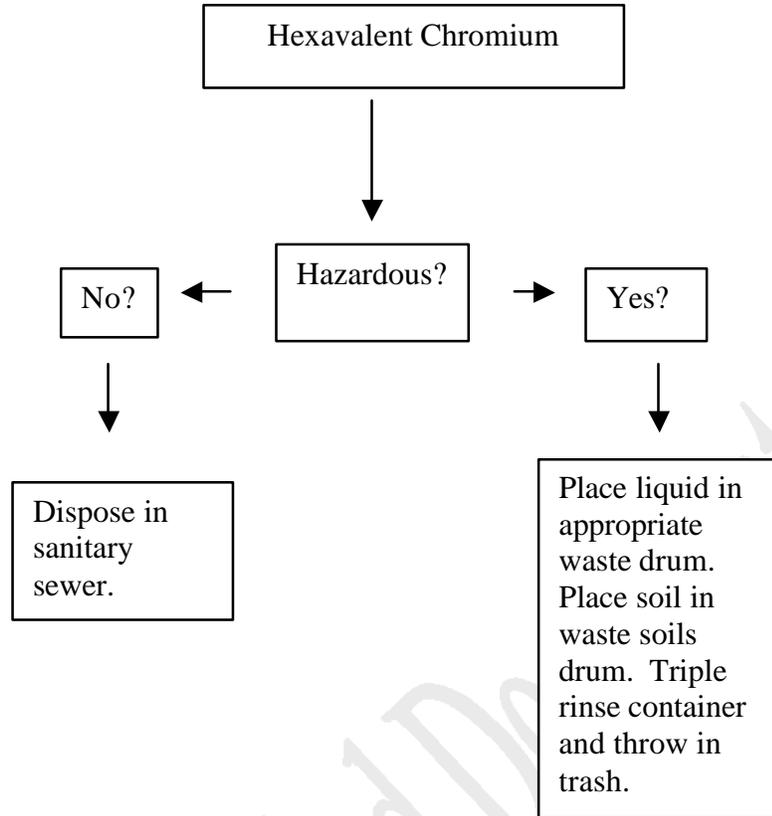
Controlled Document

Disposal Flowchart for Samples Received for TIC Analysis



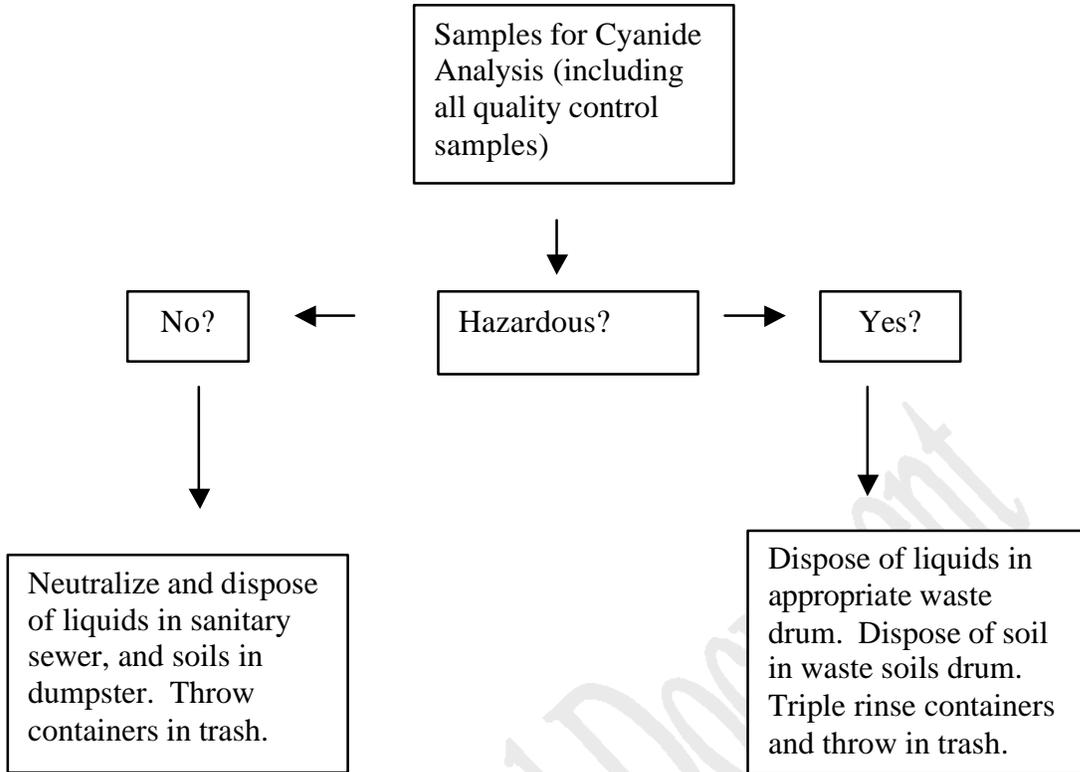
Controlled Document

Disposal Flowchart for Samples Received for Hexavalent Chromium Analysis

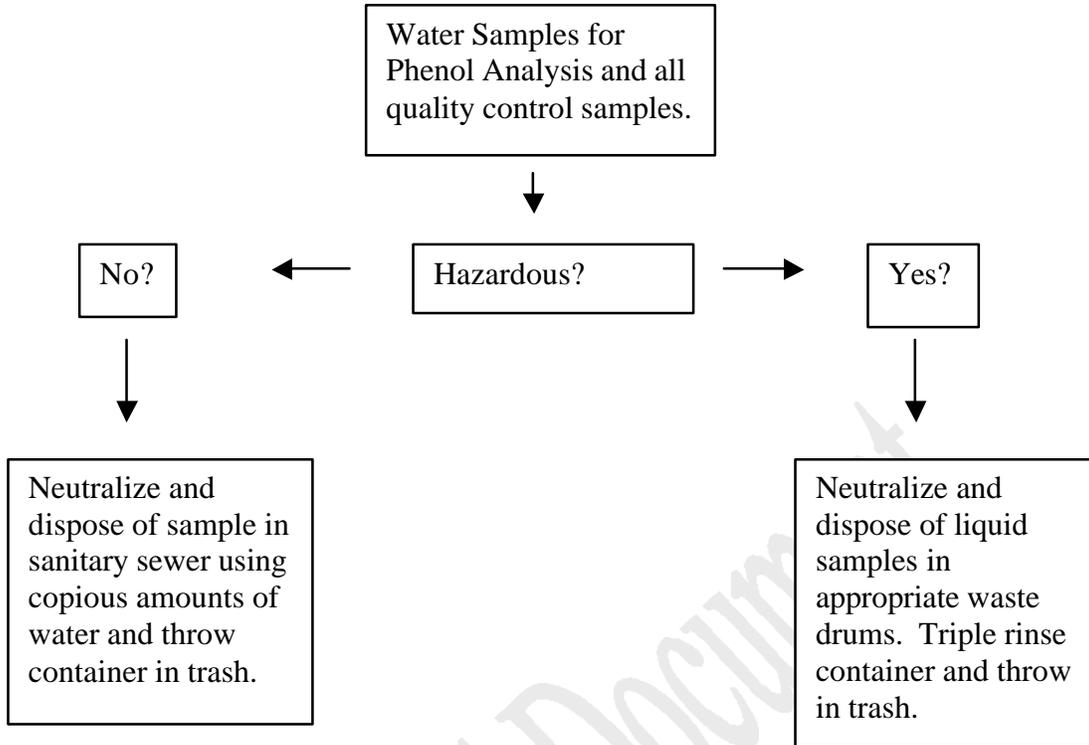


Controlled Document

Disposal Flowchart for Samples Received for Cyanide Analysis

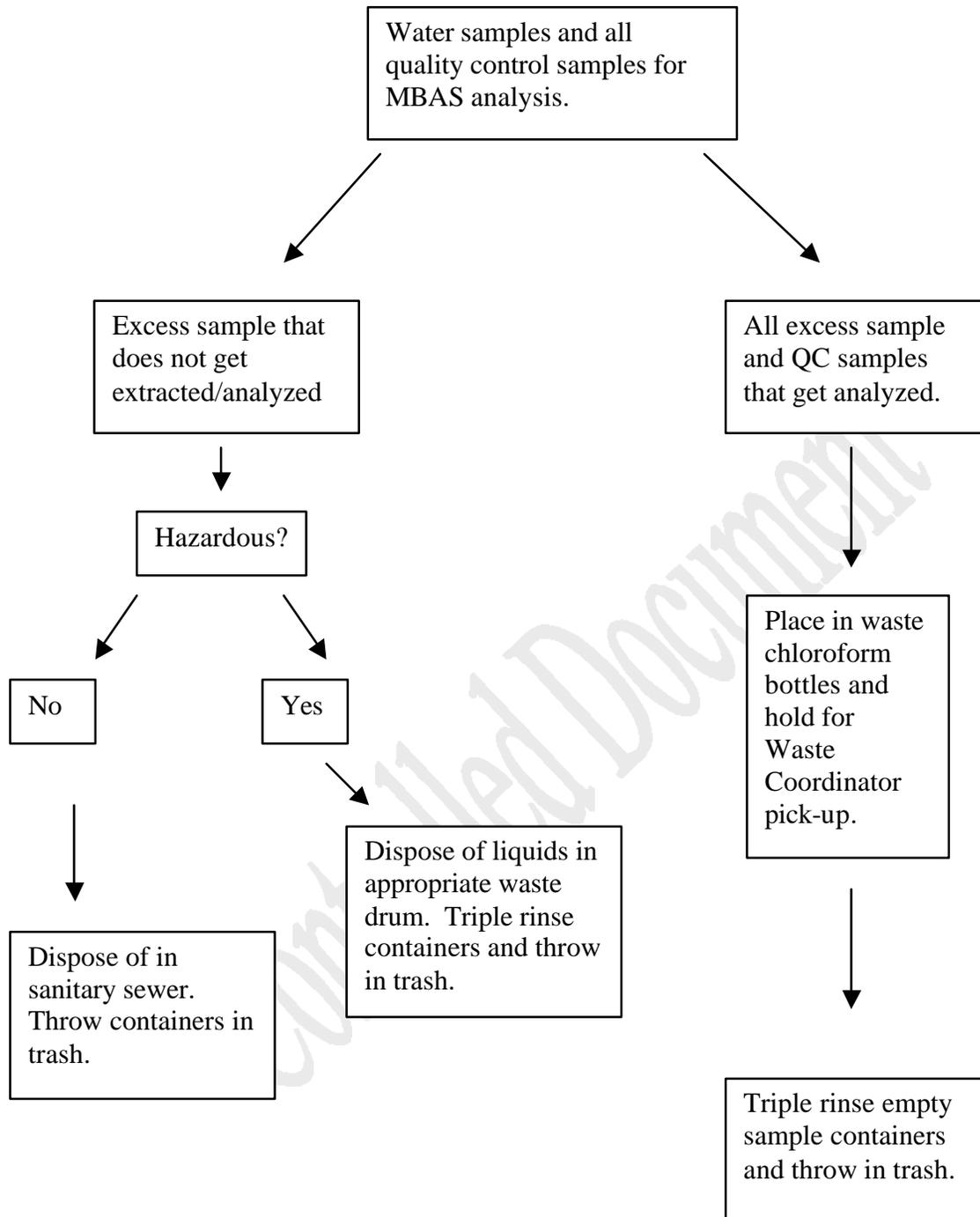


Disposal Flowchart for Samples Received for Phenol Analysis



Controlled Document

Disposal Flowchart for Samples Received for MBAS Analysis



Other Sample Disposal Procedures:

Samples for COD: Place in disposal drum provided in laboratory. When full it shall be disposed by an authorized waste hauler.

All Samples are checked for hazardous characteristics and listed wastes. All aliquots of all samples deemed hazardous are collected in the appropriate waste container and disposed of by an authorized waste hauler.

Controlled Document

Exhibit 2

Hazardous Waste Storage Area Inspection Form

Date: _____

Is cabinet locked? Yes No

Is all waste contained in the cabinet or vessel? Yes No

Is the cabinet marked with hazard warning signs? Yes No

Is the waste inventory posted on the inside door of the cabinet? Yes No

Is the waste inventory up to date? Yes No

Are containers of waste capped? Yes No

Is waste segregated according to compatibility? Yes No

Are containers labeled with EPA waste code and accumulation date? Yes No

Is the cabinet ventilated? Yes No

Is secondary containment adequate to contain 50% of the contents of each container in the event of a leak? Yes No

Do any containers show signs of deterioration? Yes No

How much waste (in kg.) do we currently have? _____

Do we have any acutely hazardous waste? Yes No

If so, how much? _____g.

Any other items of regulatory or safety concern that should be addressed?

Inspector's Signature: _____ Printed Name: _____

Microseeps, Incorporated

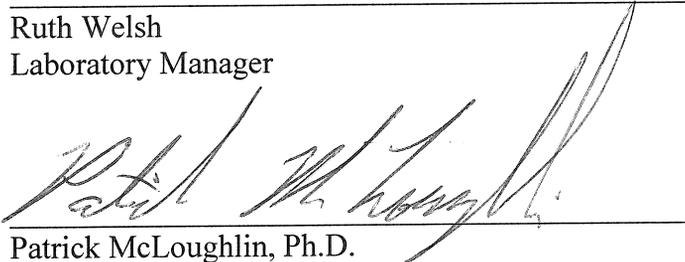
Standard Operating Procedure For the Analyses of Low Level Volatile Fatty Acids by Ion Chromatography

(Reference: Standard Methods 429; ASTM D4327)

Controlled Copy No. _____

Signatures of Final Approval:

Ruth Welsh
Laboratory Manager



Patrick McLoughlin, Ph.D.
Technical Director

2-8-07
Date

1.0 Purpose and Application

When microbes are carrying on reductive dechlorination, they do not use the chlorinated compounds as food. Instead, they use other sources, such as BTEX or natural organic carbon. When food is unavailable, an attempt to enhance the biodegradation by addition of a carbon substrate is a viable option. Once these substrates enter the groundwater and the microbes begin using them as a food source, they rapidly take the form of Volatile Fatty Acids (VFAs). As the groundwater travels down gradient from the injection point, VFA concentrations rapidly drop below the 1-ppm level, however there is still evidence of reductive dechlorination. This suggests that microbes can utilize VFAs at concentrations lower than 1-ppm.

This Standard Operating Procedure addresses the sequential determination of low level VFAs in groundwater. The method used for this procedure is a modification of SW846-9056A.

1.1 Analyte List

**Table 1.1
Analyte List**

Low Level VFA	CAS Number
Lactic acid	50-21-5
Hydroxy-isobutyric acid (HIBA)	594-61-6
Acetic acid	64-19-7
Propionic acid	79-09-4
Butyric acid	107-92-6
Pyruvic acid	127-17-3
i-Pentanoic	503-74-2
n-Pentanoic acid	109-52-4
i-Hexanoic	646-07-1
n-Hexanoic acid	142-62-1

1.2 Matrix

This method is applicable to groundwater.

2.0 Method Summary

Samples are pretreated to remove potential interference. The pretreated samples are then spiked with a mix of compounds that serve as preservatives and internal retention time markers. Samples are then analyzed by ion exchange in an ion chromatograph (IC). The anions are separated on an ion chromatograph and chemically converted to their acid form in an anion self-regenerating suppressor (ARSR). The volatile fatty acids pass through an electrical conductivity

detector. The instrument responds by producing peaks that correspond to the individual VFA concentration.

2.1 Definitions

Analytical Batch: a group of twenty samples or fewer that are prepared and analyzed together.

Analytical Spike: sample duplicate that is spiked with target compounds to aid in identifying observed peaks. The analytical spike is not intended to be used for quantitative recovery calculations. For that, the matrix spike should be used.

Laboratory Control Sample: sample matrix free from analytes of interest, spiked with verified known amounts of analytes. A LCS is used to assess the performance of the measurement system.

Matrix Spike/Matrix Spike Duplicate: samples prepared by adding a known concentration of target analyte to a specific amount of sample. Matrix spikes are used to determine the effect of sample matrix on a method's recovery efficiency.

Method Blank: a sample of similar matrix that is free from the analytes of interest that is processed through all the steps of the analysis with other samples.

Internal Retention Time Marker: a spike of non-target compounds that is placed in a sample and used to determine the relative retention times. Internal Retention time markers are used to compensate for the inevitable retention time shift that occurs in this chromatography. The internal retention time marker is not to be used quantitatively, as a surrogate would be used.

2.2 Method Limitations

Lactic acid (2-hydroxy propionic acid) co-elutes with hydroxy-iso-butyric acid (HIBA, 2 hydroxy-2 methyl propionic acid). Either of these may be a target analyte, but it is unlikely that they would both be found at detectable levels in the same well. Studies have shown that the two compounds respond equally, and the Laboratory Information System (LIMS) is set so that the client report reflects that any observed peak may be lactic acid, HIBA, or both.

The anion exchange chromatography used in this procedure produces considerable retention time shifts. The use of internal retention time markers can help eliminate any uncertainty in peak identification that these shifts introduce. Further confidence can be provided by the use of an analytical spike.

Retention time shifts are due to variations in the pH and the ionic strength of the treated sample. Any increase in ionic strength will shift retention times and may degrade the chromatography. For this reason, surrogates are not used in this method because they would increase the ionic strength of the sample.

Dichloroacetic acid, nitrate and bromide co-elute. Inadvertent introduction of carbonate or carbon dioxide can adversely affect the samples. Additionally, latex gloves must be worn throughout the preparation procedure to minimize the introduction of sulfate and chloride.

3.0 Apparatus, Materials, and Operating Conditions

3.1 Apparatus

- Sample pre-treatment rack
- Dionex[®] Ion Chromatograph DX-500:
 - Chromatography Oven: LC 25
 - Conductivity Detector: CD 20
 - Gradient Pump: GP 40
 - Ion Trap Column: Ion Pac ATC-3, 9 x 24 mm
 - Suppressor: ASRS Ultra II, 4 mm
 - Data collection system: IBM-compatible PC with Dionex Chromeleon software.

3.2 Materials

- Type II deionized degassed water

3.3 Operating Conditions

- Oven temperature: 35°C
- Reservoir pressure: 7psi
- Suppressor current: 50 mA
- Suppressor Regenerant Solution: externally supplied DI
- Eluent concentration program: The total pumping rate is always 1.50 ml/min. The gradient between fixed points is always linear.
- The timed events of the injection and the autosampler triggered by TTL1 of the pump. See Table 3.3 It should be remembered that the AS40 is triggered as TTL1 goes to 0V.
- HPLC Grade Acetone
- HPLC Grade Methanol

4.0 Reagents

All reagents are prepared from water with a resistivity of 18-mega ohm/cm (Mohm); obtained from the Ultra-pure water system in Microseeps wet chemistry laboratory. Reagent grade chemicals are used in preparing all reagents. All standards are labeled and documented in accordance with Microseeps Standard Operating Procedure for Standards and Reference Materials SOP-ADM 15. The following reagents are used:

- Potassium Hydroxide KOH

- Benzalkonium chloride (BAK)
- Degassed deionized water
- UHP Helium
- Formic acid
- HIBA
- Lactic acid
- Pyruvic acid
- Quinic acid

4.1 Standard Preparation Procedures

Standards are purchased as a custom mix from Supelco or another vendor, for the simple alkanolic acids and spiked with a mixture of Lactic acid, Pyruvic acid and HIBA. Initial calibration standards are prepared at five concentrations: low, medium low (MLow), medium (mid-range), medium high (MHigh) and high using 15,000X, 10,000X, 5000X, 1000X and 500X dilutions of the purchased standard mix. This is listed in Table 4.1.

Table 4.1
Calibration Mix Concentrations

Acid	Concentration in ppm	Low 15,000X	MLow 10,000X	Medium 5000X	MHigh 1000X	High 500X
Quinic	-	-	-	-	-	-
Lactic acid	1000	0.0667	0.100	0.200	1.00	2.00
Acetic	610.0	0.0406	0.0610	0.122	0.610	1.22
Propionic	777.1	0.0518	0.0776	0.155	0.776	1.55
Formic	-	-	-	-	-	-
Butyric	888.1	0.0591	0.0887	0.177	0.887	1.77
Pyruvic	1000	0.0667	0.100	0.200	1.00	2.00
i-Pentanoic	1054.1	0.0702	0.105	0.211	1.05	2.11
Pentanoic	1054.1	0.0702	0.105	0.211	1.05	2.11
Chloride	-	-	-	-	-	-
i-Hexanoic	1161.6	0.0773	0.116	0.232	1.16	2.32
Hexanoic	1165.1	0.0776	0.116	0.233	1.16	2.33

Table 4.1 Note: Compounds are listed in order of elution. Quinic acid, formic acid, and chloride are used as internal retention time markers only.

4.2 Preservative Solution Preparation

There are two preservative solutions. One is a solution of BAK in DI that is used as a field preservative. Some or all of that preservative is removed during the preparation described in

section 5.1. That preservative is replenished by the spiking of the prepared solutions. The internal retention time markers are placed into that spiking solution as well.

4.2.1 BAK field Preservative

BAK is an extremely viscous liquid that is completely water soluble (it is soap). Dissolve 12 grams of BAK into 1 liter of DI water.

4.2.2 Preservative and Internal Retention Time Marker Spiking Solution

Place 250 ml DI water in a 400 ml beaker and add 3 g of BAK and let the BAK dissolve. Place 20 ml of that solution into a 40 ml VOA vial. Dissolve 5 mg formic acid and 5 mg quinic acid into that solution. Use DI to fill the rest of the vial and mix.

4.3 Quality Control Sample Preparation

A working stock standard (50x dilution) is made up using the same standard as for the initial calibration.

4.3.1 Laboratory Control Sample

The laboratory control sample (LCS) is a portion of reagent grade water that has been prepared in the same manner as a field sample, but has been spiked with a known amount of the contaminants being monitored. The laboratory control sample (LCS) is prepared at a 1 ppm concentration from purchased stock standards by adding 1ml of the standard solution to 9ml of deionized water.

4.3.2 Matrix Spike/Matrix Spike Duplicate

The matrix spike and spike duplicates are prepared using a 20x dilution of the working stock used for initial calibration by adding 0.5ml of the standard solution to 9.5ml of deionized water. True values of the MS/MSD are listed in Table 4.3.3 below.

4.3.3 Initial and Continuing Calibration Verification

The ICV and CCV are prepared at a concentration of 1 ppm from a source other than that used for the initial calibration. True values of the CCV are listed in Table 4.3.3 below.

Table 4.3
CCV and MS/MSD True Values

Volatile Fatty Acid	TV of MS/MSD & CCV (ppm)
Lactic Acid and HIBA	1.000
Acetic Acid	0.610

Propionic Acid	0.780
Butyric Acid	0.890
Pyruvic Acid	1.000
i-Pentanoic Acid	1.050
Pentanoic Acid	1.050
i-Hexanoic Acid	1.160
Hexanoic Acid	1.160

4.4 Glassware and Storage Requirements for Reagents and Standards

All standards are stored in tightly sealed glass containers and cooled to between 2°C and 6°C when not in use.

5.0 Procedure

Samples are collected in 40 ml glass VOA vial and preserved with the BAK solution described in section 4.2.1. Samples may be stored for up to 14 days at 4°C ± 2°C.

Analysts who use this method have been certified for the method by running Initial Demonstration of Proficiency (IDOP) Samples in accordance with Microseeps Standard Operating Procedure for Administering and Documenting Training in Laboratory Procedures and Instrumentation (SOP ADM 02). IDOP's are run any time there is significant change to an instrument, method, or in the training procedure for training a new analyst.

5.1 Sample Preparation

Prepare the sample bottles by adding 4 drops of prepared BAK solution to a clear glass 40 ml vial. Be sure that the full preservative name of benzalkonium chloride is written on the bottle labels.

All samples, including quality control samples (LCS, MS/MSD), must be field or lab preserved and pre-treated. Preparation of the solution used to accomplish this is described in section 4.2.2.

Inadvertent introduction of carbonate or carbon dioxide can adversely affect the samples. Additionally, spurious introduction of sulfate and chloride should be minimized, though some is inevitable. To minimize that introduction, latex gloves should be worn throughout the preparation procedure.

5.2 Calibration

The calibration proceeds via three steps: standard sample preparation, peak identification and calibration calculation.

5.2.1 Standard Sample Preparation

The standards are prepared according to the following procedure:

- Insure that the instrument is setup as specified in section 3.
- Prepare calibration standards and a blank by labeling six vials with the concentrations specified in section 4.1.
- Cap the vials, being sure to completely insert the filter-cap into the vials (the cap depression tool may be used to facilitate this).
- Add one drop of the ‘post-prep.’ spike
- Load the calibration standards into an autosampler rack and begin a sequence file that identifies each bottle and its contents.

5.2.2 Peak Identification

Initially, the order of elution must be measured from the analysis of single component standards. That order has been found to be the same order in which the acids are listed in table 4.1, with HIBA exactly co-eluting with lactic acid.

With that information, the position and width of the retention time window used to make identifications should be based on measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time can be used to calculate a suggested window size for a compound. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms. The internal retention time markers quinate (from quinic acid), formate (from formic acid), and chloride (from the BAK) are added to each sample. Quinate is an extremely early eluting compound, it even elutes before fluoride. The elution of formate is specified in Table 4.1. In standards containing i-butyric acid, formate is the first component of the partially merged peaks eluting after propionate. Chloride elutes between pentanoic acid and i-hexanoic acid. Analytical spikes may also be useful for difficult analyte assignments.

5.2.3 Calibration Calculations

The standards are analyzed as samples and then the Chromeleon software is used to combine the calibration and the method file. As described in Appendix B, an appropriate calibration is done with a quadratic equation with no offset. The calibration points (concentration vs. instrument response) are fit to a line without intercept via a least-squared-error type regression routine in the software. For further procedural details, please see the Chromeleon software documentation. The correlation of determination (which has the same meaning for a quadratic calibration as for a linear calibration) must be at least 0.995. If this criterion is failed, the system should be inspected and the calibration repeated.

Formula:

The formula used for calculating concentrations is as follows:

$$c = c_{ji} A$$

Where: c = concentration.
 c_{ji} = first coefficient for compound i , determined from the calibration.
 A = area count or instrument response.

5.2.4 Initial Calibration Verification

After initial calibration, the calibration curve must be verified by use of an ICV standard at or near the mid-range of the calibration curve as specified in Section 4.3.3.

5.2.4.1 ICV Acceptance Criteria

The acceptance criteria for the ICV standard must be no greater than $\pm 20\%$ of its true value.

5.2.4.2 Corrective Action

If the calibration curve cannot be verified within the specified limits, the cause must be determined and the instrument recalibrated before samples are analyzed.

5.3 Sample Analysis

Instrument runs employing the autosampler should always start off with two blanks. The purpose of the first is to establish the baseline conditions discussed above and to clean the entire instrument. The data produced from this first analysis is of no use. This run is typically called the "blank XX." To insure that no analytes or samples are left in the analytical path, the last analysis should also be a blank. A method called "shutdown.met" was written to turn off the pump and the ASRS. It should always be in a schedule, and should always be last.

5.3.1 Automatic System Cleaning

During every sample analysis two cleaning procedures are executed. Those procedures, and the provisions that must be made for them, are described below.

5.3.1.1 Rinses

There is an automatic rinse programmed into the method. This rinse occurs five minutes after the start of analysis. It means a vial, filled with only DI water, must follow each sample. The rinse used here is treated only as a rinse by the Ion Chromatograph (IC), not the auto-sampler. That means that the cap on the autosampler vial is to be fully depressed on all rinses.

5.3.1.2 Column "Clean out"

Once the retention time of all analytes of interest has passed, the gradient program passes a high concentration of hydroxide onto the column. The intent of this is to force even highly retained anions off of the column. However, the basic eluent used in this procedure strips the hydronium (H₃O) from the suppressor membranes, and suppressor recovery from this clean-out procedure requires quite a lot of time. For 10.4 minutes after the clean-out procedure, the base line eluent is passed through the column, giving the chromatographic system a chance to re-equilibrate.

5.3.2 Instrument Run Procedure

For a new calibration, a new sequence file should be set up per 5.3.2.1. For a run that is using a pre-existing calibration, the current sequence should simply be appended to the existing sequence file, per 5.3.2.2.

5.3.2.1 Setting Up and Using a New Sequence

- With Chromeleon running, make sure the program is available and note the name of the program.
- On the IC computer, using Chromeleon and the timebase Semivol13, choose File>New>Sequence
- Use the Wizard to set up the sequence, responding to the prompts.

5.3.2.2 Using an Existing Sequence

- Select a sequence on the IC computer.
- Scroll to the last sample and append another below that by selecting the sample, right clicking on it, and choosing "Append".
- In the appended sample, change the sample type (standard or unknown, the last being for QC samples and client samples), the sample name and check the method and program.
- You can always add to a sequence after it has started.
- From the VFA panel choose "Batch" and "Start"

The method is already set up to synchronize the autosampler, IC injection valve, and the data acquisition system.

For aqueous samples with volatile compound concentrations that exceed the range of the initial calibration the sample is to be diluted and reanalyzed until the concentration is within the initial calibration range.

5.4 Quality Control Requirements

The following quality control samples must be run with each analytical batch or more frequently, if noted.

5.4.1 Continuing Calibration Verification

In order to verify the working calibration curve a CCV is run at the beginning of each day and at the beginning and end of each analytical batch. If the instrument response has changed more than $\pm 20\%$, the instrument should be recalibrated.

Corrective Action: If the calibration cannot be verified within the specified limits, discontinue sample analysis, determine the cause, and recalibrate the instrument. All samples analyzed after the last acceptable CCV/CCB must be reanalyzed.

5.4.2 Continuing Calibration Blank

The CCV must be followed by a continuing calibration blank (CCB). The calibration blank must not contain target analytes above the reporting limits.

Corrective Action: If this criterion is not met, inspect all glassware, etc. and then prepare and analyze another blank. Blanks shall be run until this criterion is met. If three blanks are analyzed in succession and this criterion is still not met, the laboratory director shall be notified.

5.4.3 Performance/Method Blank

A performance (PBW) or method blank (18 Mohm water) shall be analyzed for each analytical batch. Any analytes detected in this blank must not be present in concentrations greater than that analyte in the lowest standard.

Corrective Action: If this criterion is not met, inspect all glassware, etc. and then prepare and analyze another blank. Blanks shall be run until this criterion is met. If three blanks are analyzed in succession and this criterion is still not met, the laboratory director shall be notified.

5.4.4 Laboratory Control Sample (LCS)

A Laboratory Control Sample (LCS) shall be analyzed every twenty samples. The LCS should be spiked with each analyte of interest between the low and mid-level standards and must be carried throughout the entire sample preparation and analytical process. Acceptance criterion is a percent recovery between 70% and 130%.

Corrective Action: If the percent recovery for this sample is not between 70% and 130%, a fresh LCS solution should be made, and the LCS should be run again. If it fails again, all samples analyzed after the last acceptable LCS must be prepared again and reanalyzed.

5.4.5 Matrix Spike and Matrix Spike Duplicate

Matrix spike and matrix spike duplicate (MS) samples shall be run for each analytical batch. The percent recovery must be between 70% and 130% and the relative percent difference (RPD) must be $\leq 30\%$.

Corrective Action: If this criterion fails, but all other instrument run criteria are passed, then it is simply noted in the case narrative for the instrument run and the analysis proceeds. Acceptance criteria shall be modified as in-house data is compiled.

5.4.6 Contingency for Handling Out of Control or Unacceptable Data

All samples associated with out of control quality control samples must be reanalyzed. If quality control acceptance criteria cannot be met using the corrective action above, a detailed check of the de-ionized water and chemical purity is made. Reagents, standards, and other quality control samples are re-prepared and analyzed. If problems persist, sample analysis will be halted and the Technical Director shall be contacted immediately to determine the cause and implement corrective action.

Any data submitted with unacceptable quality control sample results shall be qualified in a case narrative. The narrative should indicate the out of control event that occurred, the corrective action that was taken, and any other pertinent information to inform the client of exactly what occurred.

5.5 Capturing and Submitting Data

After the anions are separated on the IC column, they are chemically converted to their acid form. The anion most prevalent in the column eluent is hydroxide (OH^-). The acid form of hydroxide is water. Water has no measurable conductivity. The other acids all have measurable conductivity, so they produce peaks in the conductivity cell as they pass through it.

The peaks are maximized and the noise is minimized if the conversion is perfectly efficient. The conversion occurs in the suppressor (ASRS, or anion self regenerating suppressor). The suppressor shall be cleaned prior to instrument calibration to maximize its efficiency. The procedure for this is given in Appendix A.

The ASRS uses an electrical current to generate the hydronium ions (H^+) that acidify the anions. If this current is too low, the ASRS cannot efficiently neutralize all of the hydroxide. If the current is too high, the useful lifetime of the ASRS is reduced. The minimum setting is 50 mA, and that is the present setting. If the baseline becomes noisier, this can be increased in the method, but such increases should generally be a last resort, and would require a recalibration.

Acquisition of data for all standards, samples, blanks, and laboratory control samples is done using a Windows based personal computer outfitted with Dionex Chromeleon software. The software collects data, plots the peaks, integrates the peaks, calculates the calibration curve associated with the target analytes, and calculates the concentrations of the analytes in mg/L. After review from the analyst, a standard report containing a chromatogram is printed.

The raw data from all analyses, including initial calibration, calibration verification, and method blanks are stored by the analyst in the laboratory where the analyses are performed.

5.5.1 Retention Time Windows

Retention time studies have been conducted for this analysis. These studies are kept on file in the Quality Systems Office. The retention times in Table 5.5.1 below are examples. The exact retention times will vary as a function of sample composition, column type, column age, and column history. For the instruments that use this method, true retention times and retention time windows are taken from the most recent standard analyzed.

**Table 5.5.1
 Retention Time Windows**

Low Level VFA	RT Window (Min.)	RT Window (Min.)
Lactic acid and HIBA	6.683	12.590
Acetic acid	7.259	13.408
Propionic acid	8.840	15.627
Butyric acid	12.318	18.368
Pyruvic acid	13.821	19.539
i-Pentanoic	15.489	20.864
n-Pentanoic acid	19.832	25.355
i-Hexanoic	27.659	30.008
n-Hexanoic acid	29.721	31.539

6.0 Secondary Data Review

The analyst is responsible for insuring that all calibrations, calibration checks, and quality control samples are within the specifications outlined in this SOP.

All data are validated by the analyst and the Lead Analyst. Both signatures are required on the case narrative sheets that are turned in for each analytical batch.

The analyst checks all raw data and calculations for reasonableness and accuracy, making sure that sample dilutions are taken into account. Quality control results are rechecked for compliance with acceptance criteria. If any acceptance criteria cannot be met or if any atypical conditions are encountered, a Case Narrative detailing the conditions is written and handed in with the results.

6.1 Peer Review

All data derived from this method undergoes peer data review prior to being turned into the assistant laboratory director. This review served to catch potential errors prior to the data's entry into the Laboratory Information Management System. This review may only be done by another analyst who is certified in this method or the Group Lead Analyst.

6.2 Laboratory Director Review

The Laboratory Director reviews 10% of all laboratory data and calculations. This review includes sample results, quality control acceptance limits, and a review of the level of quality control required for the project.

6.3 Performance Evaluation Studies

Performance evaluation samples are currently not available. When they become available, they will be analyzed twice annually using this method.

7.0 Reporting Limits

Method detection limit studies are run annually in accordance with Microseeps Standard Operating Procedure for the Determination of Method Detection Limits and PQLs (SOP-ADM 18). Reporting limits for the VFAs are shown in table 7.0 below:

Table 7.0
Volatile Fatty Acid PQLs

VFA	PQL in mg/L
Lactic Acid	0.10
Hydroxy-isobutyric acid (HIBA)	0.07
Acetic acid	0.07
Propionic acid	0.07
Formic acid	0.07
i-Butyric acid	0.07
Butyric acid	0.07
Pyruvic acid	0.07

i-Pentanoic	0.07
Pentanoic acid	0.10
i-Hexanoic	0.10
Hexanoic acid	0.10

8.0 Safety

Safety glasses are required in all laboratory areas. Samples and reagents should always be handled with caution. For other safety concerns, consult Microseeps' Chemical Hygiene Plan. Material Safety Data Sheets (MSDS) for all compounds used in this procedure are available in the Microseeps' conference room.

9.0 Waste

Unused portions of samples are kept for thirty days following analysis. The samples are then removed from the laboratory and stored until disposal according to Microseeps Standard Operation Procedure for Waste Disposal (SOP-ADM 14).

9.1 Waste Minimization

Where possible, Microseeps takes steps to minimize the amount of waste generated by substitution and good chemical handling procedures. For specific information on waste minimization consult SOP-ADM 14.

10.0 References

Citing a reference does not imply that all of the recommendations and/or requirements in those cited methods are required in this Standard Operating Procedure. This section simply refers to sources that were consulted to gather information or knowledge in order to write an informed technical procedure.

Annual Book of ASTM Standards, Volume 11.01 Water D4327, Standard Test Method for Anions in Water by Ion Chromatography, pp. 696-703, 2003.

Standard Methods for the Examination of Water and Wastewater, Method 429, "Determination of Anions by Ion Chromatography with Conductivity Measurement," 16th Edition of Standard Methods.

Appendix A

Contents:

Procedure for Degassing Water

Procedure for Regeneration and cleaning of the ASRS

Instructions for priming the pump

Procedure for Degassing Water

To degas the water use the following procedure:

- Place the water to be degassed into a 4L side-arm vacuum Erlenmeyer flask with a clean, 18 Mohm water rinsed stir bar. Stopper the top of the flask.
- Set the flask atop a stir plate. With the rotation initially at zero, gradually increase it so that the water throughout the entire flask is well stirred.
- Attach the side arm to mechanical vacuum pump inlet and turn on the pump. Make sure that there is a liquid trap between the flask and the pump.
- Pump-on and mix the solution for ~ 10 minutes. Bubbles will continue to come out of the water as the water boils under vacuum, but the rate of bubbling should slow dramatically by the time pumping is ceased.
- Clamp the line from the sidearm to the trap closed and break the connection after the clamp. Turn off the stir plate. Be sure to handle the water with a minimum of agitation and atmospheric exposure.

Instructions for priming the pump

Priming the GP40 pump is a two-step process: priming the eluent manifold and priming the pump heads. Both steps should always be done so the operator is satisfied that the priming is complete.

Priming the eluent manifold:

- Open the Main screen of the GP40 and, using the arrow keys to maneuver the cursor, the select keys to scroll through the choices, and the enter key to finalize the choices, Select LOCAL and then DIRECT CNTRL.
- Verify that the Off/On LED is in the on position.
- Move the cursor to the desired eluent. Enter 100%. This automatically sets the other eluents to 0%.
- Set the flow rate to 0.
- The priming block is below the two pump heads and has a metal lever on its top. Connect a plastic luer-lock syringe to the luer adapter on the priming block.
- Find the metal lever on top of the priming block and open it completely by turning it to your left. If the eluent reservoir is properly pressurized to 8 psi, the eluent should start flowing into the syringe immediately.
- Draw eluent into the syringe, remove the syringe, discard the eluent, reattach the syringe, and repeat until the air bubble formed in the syringe is very small and reproducible.
- Return to step 3, select another eluent, and repeat the priming procedure. It must be done for channels A, B and D.
- When the manifold has been primed with each eluent, move the lever on the priming lock back to the right, fully closed position.
- Reset the flow rate to 1.5 ml/min.

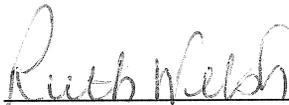
Priming the pump heads:

- Insure that the pump is in LOCAL, DIRECT CNTRL.
- Above the priming block and both pump heads is the pressure transducer. On the front and angled down, is the pressure transducer waste valve. The top of the valve is a plastic knob. Turn that knob counter-clockwise approximately two turns. Opening this valve allows the eluent to travel from the pump heads to waste, rather than from the pump heads to the column.
- Press PRIME on the GP40. The pump will begin pumping at its maximum flow rate of 2.5 ml/min for this micro-bore pump.
- Watch the waste line out of the pump for bubbles. Allow the pump to prime until no bubbles can be seen exiting the pump.
- Press PRIME again to return to the normal, screen specified, flow-rate.
- Close the pressure transducer waste valve by turning it completely clockwise. Once closed, the column pressure on the pump should rapidly rise above 1500 and should be stable. If this is achieved, the eluent system, pump included, is ready for regular operation.

Microseeps, Incorporated

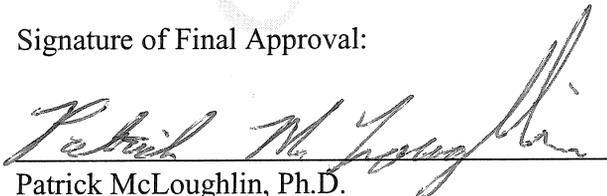
Analytical Method AM20GAx Standard Operating Procedure for the Analysis of Biodegradation Indicator Gases

Controlled Copy No. _____



Ruth Welsh
Laboratory Manager

Signature of Final Approval:



Patrick McLoughlin, Ph.D.
Technical Director

9-21-06

Date

SOP Review Date: September 15, 2006

1.0 Scope and Application

Method AM20Gax is used to determine the concentration of biodegradation indicator gases in vapor samples. Specifically, Method AM20Gax is used to determine the dissolved concentration of the following gases:

Gases	CAS Number
Acetylene	74-86-2
Carbon dioxide	124-38-9
Oxygen	7782-44-7
Nitrogen	7727-37-9
Hydrogen	1333-74-0
Methane	74-82-8
Ethane	74-84-0
Ethene	74-85-1
Propane	74-98-6
Propene	115-07-1
n-Butane	106-97-8
i-Butane	75-28-5
Carbon Monoxide	630-08-0
Total Inorganic Carbon*	

*Total inorganic carbon (TIC) is converted to carbon dioxide using the steps outlined in SOP-PM01. The sample is then analyzed for carbon dioxide according to this SOP. Any differences in method are specified in the appropriate section.

This method is recommended for use by, or under the supervision of, analysts experienced in sample preparation, the operation of gas chromatographs and in the interpretation of chromatograms.

2.0 Method Summary

The sample gas is analyzed with a gas chromatograph capable of simultaneous analysis of all of the target analytes from a single gas sample. A single injection of gas from integral, simultaneously filled sample loops is used to assure consistent injection volume. The permanent gases are analyzed using a thermal conductivity detector (TCD). The light hydrocarbons are analyzed using a flame ionization detector (FID). Hydrogen is analyzed using a reduction gas detector (RGD). The data are transferred to a microcomputer, converted to digital format, stored, and processed using a chromatography data system.

2.1 Definitions

Batch: A batch consists of twenty or fewer samples run during an eight-hour work shift.

Instrument Flush: The front end of the sample loop is flushed with ultra high purity helium injected into the loop directly from the cylinder to remove possible interference by ambient air and to avoid cross contamination between samples.

Method Blank: An injection analyzed by all three detectors that consists of ultra high purity helium. The method blank is free from the analytes of interest

Laboratory Control Sample: A sample of laboratory grade deionized water spiked with verified known amounts of analytes. A LCS is used to assess the performance of the measurement system.

Matrix Spike and Matrix Spike Duplicate: A sample prepared by adding a known concentration of target analyte to a specific amount of sample. Matrix spikes are used to determine the effect of sample matrix on a method's recovery efficiency.

3.0 Apparatus and Materials and Operating Conditions

3.1 Apparatus

Gas Chromatograph: The chromatographs designed and built by Microseeps are equipped with multiple packed columns and multi-port valves, a TCD, a FID, a RGD, and multiple sample loops. The FIDs, which were also built by Microseeps, are of a special design that allows considerably more sensitivity than commercially available models. This instrument provides rapid turn-around for consecutive analyses and a clean baseline for accurate, reproducible results.

3.2 Materials

- Sample vials (Supelco, Inc, Bellefonte, PA or equivalent)
- Syringe: locking gas tight (Hamilton/Alltech, 3, 5, 10, 30 and 60 ml or equivalent)

3.3.1 Interferences

The most likely source of "interference" is ambient air. Due to the relatively high concentrations of oxygen and nitrogen, a very small amount of air as a contaminant will dramatically affect the results. The analyst must take great care to ensure that air is flushed from the gas tight syringe before sample preparation and that no air has entered the syringe or needle prior to injection of the sample into the gas chromatograph.

Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. An unrestricted flow (Instrument flush) of pure carrier gas from a 10 psig source should be allowed to flow through each sample loop for 30 seconds prior to each analysis.

As required, the analyst should demonstrate the absence of carryover contamination by analysis of the contents of the sample loop when purged with carrier gas. This demonstration should be performed when carryover contamination is suspected (after high samples). In the event that 'ghost peaks' (peaks similar to previous sample) appear when a pure carrier gas sample is analyzed (method blank), measures should be taken to eliminate the carryover contamination.

4.0 Reagents

- Helium (UHP Gas)
- Nitrogen (UHP Gas)
- Certified Commercial Gas Standards
- Benzalkonium chloride (BAK) solution – Prepared by dissolving 12.08 g into 1L DI water.
- Tri-sodium phosphate (TSP) – purchased as the dodecahydrate

4.1 Standard Preparation Procedures

Calibration standards are prepared by using the procedures below:

4.1.1 Vial Preparation

Headspace vials used for instrument calibration standards for this method are prepared as follows:

- Crimp and cap each vial, with stopper septa.
- Evacuate each vial to vacuum.
- Flush each vial to atmospheric pressure with the vial balance gas appropriate for the detector being calibrated. (See Table 4.1)

Table 4.1

Detector	Vial Balance Gas
FID	Nitrogen
TCD	Helium
RGD	Nitrogen

4.1.1 Preparing Calibration Standards

The instrument is initially calibrated (ICAL) using dilutions of custom certified gas mixes. Refer to Table 4.1.1 for the correct amounts of standard mix and vial preparation gas to inject into prepared vials.

- Prepare the correct number of vials for the detector being calibrated.
- Each of the three detectors is calibrated with a gas mix from a commercial source.
- Remove the specified amount of standard by extracting it from the standard mix gas cylinder using a gas-tight syringe and injecting it into a prepared vial.
- Add the specified amount of vial balance gas to the same vial.

The dilution factor of one is achieved by directly injecting the standard gas mix from the cylinder into the GC.

Table 4.1.1
Standard Gas and Balance Gas Injection Volumes in ml

Dilution Levels	Standard Gas Mix	Balance Gas	Final Gas Volume
1	N/A	N/A	N/A
2	21	21	42
5	10	40	50
10	5	45	50
25	2	48	50
100	2	198	200
250	1	249	250
2500	20 (of 250x)	180	200
12500	40 (of 2500x)	160	200

4.1.2 Calibration Standard Concentrations

Calibration standards are made up in the following concentrations as specified in Tables 4.1.2 A, B, and C. The true values of the calibration standards vary slightly from cylinder to cylinder. The values below are very close approximations. All standards are prepared using 22 cc headspace vials with stopper septum or 160cc serum bottles.

Table 4.1.2 A
FID Calibration (In PPMV)

Compound	1X	5X	25X	250X	2500X	12,500X
Methane	500	100	20	2	0.2	0.04
Ethane	500	100	20	2	0.2	0.04
Ethene	500	100	20	2	0.2	0.04
Propane	500	100	20	2	0.2	0.04
Propene	500	100	20	2	0.2	0.04
n-Butane	500	100	20	2	0.2	0.04
i-Butane	500	100	20	2	0.2	0.04
Compound	1X	4X	20X	100X	500X	
Acetylene	100	25	5	1	0.2	

Table 4.1.2 B
TCD Calibration (In PPMV)

Compound	1X	2X	10X	50X	100X	250X
Carbon Dioxide	150,000	75,000	15,000	3,000	1,500	600
Oxygen	70,000	35,000	7,000	1,400	700	280
Nitrogen	665,000	332,500	66,500	13,300	6650	2,660
Methane	45,000	22,500	4,500	900	450	180
Carbon Monoxide	70,000	35,000	7,000	1,400	700	280

Table 4.1.2 C
RGD Calibration (In PPMV)

Compound	10X	15X	25X	250X	2500X
Hydrogen	50	33.3	20	2	0.2

4.2 Quality Control Sample Preparation

Quality control samples are prepared as indicated below.

4.2.1 Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV)

The ICV and CCV are prepared from a source different from the source used to prepare the ICAL standards. The concentration of the ICV and CCV is in the middle of the calibration range and is close to that of the ICAL midpoint, but because of the nature of gas standard it is not at exactly that concentration.

4.2.2 Laboratory Control Sample (LCS) and LCS Duplicate (LCSD)

The LCS and LCSD are prepared at a mid-range concentration. The type of LCS/LCSD depends upon the original matrix of the sample. For samples that arrive as vapors, the LCS/LCSD is injected as a gas. For samples that arrive as waters, DI water is spiked with a gas mixture of target analytes and prepared the same as the samples. Water that is free of the principle atmospheric components of nitrogen and oxygen is very difficult to make and similarly difficult to store. Toward that end, LCS/LCSD results for nitrogen or oxygen will not be reported with client data. Table 4.2.2 below gives the true values of the LCS/LCSDs.

4.2.2.1 Total Inorganic Carbon LCS

Mix approximately 0.20g NaHCO₃ into 200ml laboratory grade DI water, prepare according to the TIC procedures outlined in PM01 and analyze in duplicate as a sample. The true value of the spike is calculated as follows:

$$\text{mg/L CaCO}_3 = \frac{\text{Mass}(g)\text{NaHCO}_3}{\text{H}_2\text{O}(L)} \times \frac{100.09}{84.01} \times (1,000,000)$$

4.2.3 Matrix Spike (MS) and Matrix Spike Duplicate (MSD)

- For water samples, MS and MSDs are prepared, analyzed, and reported when clients' request and send sufficient numbers of aliquots to prepare them (e.g. one 40 ml vial each for the MS and another for the MSD).

Table 4.2.2

Compound	Vapor LCS/LCSD (ppmv)	Water LCS/LCSD & MS/MSD
Methane	300.0	822.8 µg/L
Ethane	100.0	41.70 µg/L
Ethene	100.0	38.54 µg/L
Propane	100.0	60.56 µg/L
Propene	100.0	57.81 µg/L

Compound	Vapor LCS/LCSD (ppmv)	Water LCS/LCSD & MS/MSD
iso-Butane	100.0	79.83 µg/L
n-Butane	100.0	79.83 µg/L
Carbon dioxide	50,000	129.3 mg/L
Oxygen	20,000	***
Nitrogen	balance gas	balance gas
Hydrogen	25.00	1376 nM

Notes on Table 4.2.2

- Since oxygen is an ubiquitous “contaminant”, it is not monitored in either the LCS or MS.
- Actual values vary slightly from lot to lot of cylinders of calibration gases.
- MS/MSD prepared by using a standard gas mix instead of He in the headspace prep. procedure.

4.2.3.1 Total Inorganic Carbon MS and MSD

Mix approximately 0.04g NaHCO₃ directly into client samples (when provided and requested), prepare according to the TIC procedures outlined in PM01 and analyze in duplicate as a sample. The true value of the spike is calculated as follows:

$$\text{mg/L CaCO}_3 = \frac{\text{Mass(g)NaHCO}_3}{\text{H}_2\text{O(L)}} \times \frac{100.09}{84.01} \times (1,000,000)$$

4.2.4 Method Blank

Method blanks are made up of ultra high purity helium injected into a vial and then into the instrument.

4.2.4.1 Total Inorganic Carbon Method Blank

The method blank for TIC is made up of deionized water in a 40 ml vial, prepared according to the TIC procedures outlined in PM01, and analyzed as a sample.

4.3 Glassware and Storage Requirements for Reagents and Standards

Reagents are stored at room temperature (70°F ±5°) and all standards are prepared fresh for each use immediately prior to each analysis. Standards are made up from compressed gas cylinders. Those standards expire after 2 years.

5.0 Procedure

Water samples should be cooled upon collection and stored at a temperature of $4^{\circ}\text{C} \pm 2^{\circ}$.

Gas samples are shipped and received at a positive pressure, which eliminates a cross-contamination issue during sample shipment. It is preferable that gas samples be shipped without cooling. However, it is not a sample receipt non-conformance if received vapor samples are packed in ice (sample may experience slight loss in pressure.) Gas samples are stored in the laboratory at room temperature ($70^{\circ}\text{F} \pm 5^{\circ}$). The pressure in gas vials is not checked upon receipt in the laboratory because of the inherent risk of losing sample, or inadvertently introducing atmospheric gases, when the septum is pierced. The number of times the septum is pierced should be as few as absolutely possible. See Section 5.2.2 for a discussion on how the laboratory checks and documents vial pressure. Holding time for both gas and water samples is fourteen days.

Water samples for light hydrocarbon analyses only (methane, ethane, propane, propene, n-butane, i-butane, acetylene) are collected in 40ml VOA vials with zero headspace and preserved with tri-sodium phosphate (TSP). TSP is added as the dodecahydrate at 200 mg/40 ml vial. This results in a sample pH > 10. Water samples collected for either permanent gases only or permanent gases and light hydrocarbon analyses are collected in 40ml amber VOA vials with zero headspace and preserved with four drops of BAK solution.

Analysts who use this method have been certified for the method by running Initial Demonstration of Proficiency (IDOP) Samples in accordance with Microseeps Standard Operating Procedure for Administering and Documenting Training in Laboratory Procedures and Instrumentation (SOP ADM 02). IDOPs are run any time there is significant change to an instrument, method, or in the training procedure for training a new analyst.

5.1 Sample Preparation

Samples that are collected using the Bubble Strip Sampling Technique, Microseeps Sampling Method SM9, do not require additional preparation prior to analysis.

Samples that are collected as waters and are to be analyzed for dissolved gases (methane, ethane, ethene, acetylene, CO_2 , N_2 , O_2 , propane, propene, iso-butane, n-butane, TIC), must be prepared using Microseeps Standard Operating Procedure PM01G.

Samples that are collected as gases, for example from a soil gas survey or from the headspace of a microcosm sample, need not be collected by a Microseeps sampling method, nor do they require additional preparation.

5.2 Analysis

5.2.1 If the sample is prepared via SOP-PM 01, it can be injected from the gastight syringe in which it is prepared by inserting the needle of the syringe through the septum on the "sample in" port. If the sample is a calibration standard, a bubble strip sample (SM9), or a gas, the septum inlet to the "sample in" port of the GC must be removed and a luer-lock needle receptacle is plumbed to the "sample in" port in place of the needle. A needle is attached to the luer-lock receptacle and inserted through the septa of the calibration standard, bubble stripped sample, or gas sample.

5.2.2 In order to initiate analysis and introduce the sample into the GC sample loop, a needle is attached to the entry port on the GC and inserted through the sample septum. The flow through the sample loop is monitored by a flow meter connected to the sample-loop vent-port on the gas chromatograph.

When a vial is sufficiently filled, the ball in the flow meter will shoot to the top of the column. This indicates that there is sufficient pressure in the vial to fill the sample loop. If the loop is not properly pressurized, this is reflected on the flow meter immediately. The ball in the flow meter will go up the column part way and drop back to the bottom. This indicates there is not sufficient pressure in the sample vial. If this happens, the analyst will remove the vial from the inlet port as quickly as possible and withdraw 10 – 12ccs of sample from the sample vial using a locking syringe. This is then injected into the instrument. The lack of sufficient pressure in the vial and the means of sample injection are then documented on the case narrative.

5.2.3 Once the flow out of the sample loop ceases (3 seconds if SOP-PM 01 is used) the sample loop valves are activated.

5.2.4 Once the sample loop valves are activated, the ports to and from the sample loop are flushed with ultra high purity helium injected into the loop directly from the cylinder to remove any interference from ambient air and to avoid cross contamination between samples.

5.3 Calibration and Results

5.3.1 The standard calibration gas should be introduced in the same manner as described in section 5.2.1 above. Measured peak areas are converted to concentrations using certified commercial gas standards. Dilutions are made to achieve multi-point calibration curves for each detector.

Methane can be detected on both the FID and the TCD. If the methane concentration causes an FID signal output level of 8000 millivolts, then any output exceeding that is quantified on the TCD.

5.3.2 Initial calibration is accomplished by analyzing multiple standards of appropriate calibration ranges.

Note: Due to the nature of preparing custom gas standards, the component concentration can fluctuate between purchased lots. This is accounted for during method/calibration development. These results will be used to establish a multi-point calibration curve.

Acceptance Criteria: A linear fit to an area response versus concentration plot is formed with the origin forced to zero, and the calibration is accepted for use if r^2 , the coefficient of determination is ≥ 0.995 . If this criterion can not be met using a linear fit, a quadratic can be used. For the quadratic fit, the acceptance criteria is also $r^2 \geq 0.995$.

Corrective Action: If the acceptance criteria specified above is not met, the reason is determined and a new set of calibration standards are analyzed.

5.3.3 An Initial Calibration Verification (ICV) standard immediately follows the initial calibration. Acceptance criterion for the ICV is an instrument response within $\pm 20\%$ drift. Since the instrumentation used at Microseeps routinely monitors the percent recoveries and in this instance percent drift is equal to percent recovery less 100%, the control limits are 80%-120% recovery for the ICV.

$$\text{Percent Recovery} = \frac{\text{Measured Value}}{\text{True Value}} \times 100\%$$

Acceptance Criteria and Corrective Action: If the instrument response for the ICV standard is outside the acceptance window of 80-120%, the analyst will not analyze samples until either the reason is determined and the problem is corrected, or a new multi-point calibration is analyzed and an acceptable ICV is run using that calibration.

5.3.4 An initial calibration blank follows the ICV. The blank is made up of the carrier gas. Compounds must not be detected above the reporting limits.

Corrective Action: If the blank does not meet the acceptance criterion, another blank is injected until the results are within the acceptance criterion.

5.3.5 The analytes of this method are indicators. Every attempt to achieve and deliver precise results is made. However, it is realized that for indicator parameters measuring the range of the analyte concentration (*i.e.* is the concentration of methane gas >1 mg/l or < 0.1 mg/l) is the primary goal of employing these analyses. The calibration range is chosen to extend over most of the bio-indicator concentration range. If the concentration of an analyte exceeds that of the highest calibration standard, but does not saturate the instrument response, the concentration is calculated by assuming detector response linearity and using an extrapolation of the calibration

plot. If the instrument response is saturated the sample is diluted to bring the analyte concentration into the calibration range.

5.4 Quality Control

The following quality control samples shall be analyzed with each analytical batch of twenty or fewer samples.

5.4.1 A Continuing Calibration Verification: The CCV is made up from a source other than what was used to make up the initial calibration. The acceptance criterion for the CCV is a percent recovery of 80-120%

Corrective Action: If the CCV fails, a new CCV is prepared and analyzed. If the new CCV falls within the acceptance criterion, analysis continues. If the new CCV fails, the instrument shall be recalibrated, and all samples since the last acceptable calibration shall be reanalyzed, provided sufficient sample volume is present and the samples have not been compromised by exposure to air.

5.4.2 A Continuing Calibration Blank: A CCB follows each CCV. The blanks are made up of the carrier gas. The acceptance criterion for the blank is the result must be less than the reporting limits for all compounds.

Corrective Action: If the blank does not meet the acceptance criterion, another blank is injected until the results are within the acceptance criterion.

5.4.3 Laboratory Control Sample and Laboratory Control Sample Duplicate: The LCS and LCSD are prepared and analyzed at a mid-calibration range. Both an LCS and an LCSD are to be run with each batch.

Acceptance Criteria: Percent recovery is required to be between 75% and 125%, inclusive. An Acceptance criterion is based upon the percent recovery and the RPD as calculated by:

$$\text{Percent Recovery} = \frac{\text{Measured Value}}{\text{True Value}} \times 100\%$$

$$\text{RPD} = \frac{|C1 - C2|}{\frac{C1 + C2}{2}} \times 100\%$$

Where: C1=LCS
C2=LCSD

RPD (Relative Percent Difference) is required to be less than or equal to 20%.

Corrective Action: If the LCS fails, a new LCS is prepared and analyzed. If the new LCS falls within the acceptance criterion, analysis continues. If the new LCS fails, analysis is stopped and the instrument is checked with a series of standards to determine the cause. Once the cause is determined and the instrument repaired, calibration is conducted and analysis continues.

5.4.4 Matrix Spike and Matrix Spike Duplicate: Matrix spikes and spike duplicates are analyzed for water samples only when requested by a client and sufficient sample aliquots are provided. Acceptance criterion is a percent recovery between 70% and 130%, and a relative percent difference (RPD) of less than or equal to 20%.

Corrective Action: If the matrix spike and spike duplicate fail but all the other quality control samples are within the acceptance criteria, matrix interference is noted in the Case Narrative.

5.4.5 Method Blank: A method blank is analyzed with each sample batch. The blanks are made up of UHP helium for all of the gases except for blanks for TIC. TIC blanks are made up of deionized water. The acceptance criterion for the blank is the result must be less than the reporting limits for all compounds.

Corrective Action: If the blank does not meet the acceptance criterion, another blank is injected until the results are within the acceptance criterion.

5.4.6 Contingency for Handling Out of Control or Unacceptable Data

If the requirements set forth in section 5.4 are not met, the analytical program will be terminated until the cause is determined and a solution is affected. All samples associated with out of control quality control samples (with the exception of matrix interference) must be reanalyzed provided another vial of sample has been provided by the client. If quality control acceptance criteria cannot be met using the corrective action above, a detailed check of the analytical system is made. Reagents, standards, and other quality control samples are re-prepared and analyzed. If problems persist, sample analysis will be halted and the Laboratory Manager shall be contacted immediately to determine the cause and implement corrective action.

Any data submitted with unacceptable quality control sample results shall be qualified in a case narrative. The narrative should indicate the out of control event that occurred, the corrective action that was taken, and any other pertinent information to inform the client of exactly what occurred.

5.4.7 An experienced analyst shall examine all chromatograms.

5.4.8 Through out analysis the gas samples are injected mechanically into the GC flow path utilizing a sample loop to achieve a uniform sample size from a flow directly from the sample preparation syringe. The uniform sample size achieved using the sample loop assures consistent

and accurate results. Table 5.4.8 (see next page) gives example data from a study performed via this analysis. That data can also be used for accuracy and precision estimates.

Controlled Document
Confidential

Table 5.4.8
Example Data for Precision and Accuracy Studies

	Carbon Dioxide	Oxygen	Nitrogen	Methane	Hydrogen	Methane	Ethane	Ethylene	Propane	Propylene	Iso-Butane	N-Butane
REP. #	(%v)	(%v)	(%v)	(%v)	(PPMV)	(PPMV)	(PPMV)	(PPMV)	(PPMV)	(PPMV)	(PPMV)	(PPMV)
1	0.1221	0.0670	0.5744	0.0410	0.1118	0.2512	0.0525	0.0453	0.0461	0.0581	0.0473	0.0358
2	0.1267	0.0690	0.6020	0.0428	0.1122	0.2608	0.0518	0.0468	0.0521	0.0465	0.0439	0.0407
3	0.1207	0.0657	0.5838	0.0446	0.1247	0.2812	0.0509	0.0485	0.0529	0.0588	0.0436	0.0405
4	0.1193	0.0667	0.6036	0.0444	0.1244	0.2779	0.0549	0.0460	0.0461	0.0536	0.0549	0.0476
5	0.1261	0.0703	0.5860	0.0439	0.1120	0.2894	0.0551	0.0497	0.0520	0.0549	0.0417	0.0460
6	0.1193	0.0665	0.5861	0.0478	0.0943	0.2970	0.0515	0.0467	0.0458	0.0542	0.0435	0.0514
7	0.1227	0.0732	0.5748	0.0353	0.1296	0.3053	0.0532	0.0473	0.0485	0.0584	0.0483	0.0535
AVERAGE	0.1224	0.0683	0.5872	0.0428	0.1156	0.2804	0.0528	0.0472	0.0491	0.0549	0.0462	0.0451
KNOWN	0.1500	0.0700	0.6649	0.0450	0.0999	0.1500	0.0500	0.0500	0.0500	0.0500	0.0500	0.0500
STD. DEV.	0.003	0.003	0.012	0.004	0.012	0.019	0.002	0.001	0.003	0.004	0.004	0.006

5.4.9 The gas matrix for this analysis minimizes the opportunity for matrix effects. If the gas is prepared from a matrix other than that which is injected into the GC (*e.g.* prepared through headspace extraction via Microseeps SOP-PM01), the client should request that matrix spike (MS) and matrix spike duplicate (MSD) analyses be conducted and should supply sufficient sample volume. Since matrix effects are extremely site dependent, the MS and MSD are not part of the regular analytical quality assurance program.

5.4.10 All of the target analytes are gases at room temperature so the opportunity for carry over is small. This is further reduced by the flushing of the sample loop, by the “backflush” configuration of the GC plumbing, and by the nightly bake-out procedure. These combine to keep carry-over concentrations to less than half of the reporting limits.

5.5 Capturing and Submitting Data

The output of the chromatograph is directed to a microcomputer where the signal is converted to digital format, stored, and processed using a chromatography data system.

Automated valve control: Digital control is provided by the microcomputer through the chromatography data-system software. This control provides constant start and stop times for directing carrier gas flow. The event times are programmed and saved using the method editor module of the software.

5.5.1 Total Inorganic Carbon Result Calculation

The total inorganic carbon result is calculated as follows:

$$\text{TIC as mg/L CaCO}_3 = (\% \text{CO}_2)(\text{Volume headspace})(2.08) + 43.3$$

This analysis produces concentration of the analyzed gas in % V.

5.5.2 Retention Time Windows

Retention time studies have been conducted for this analysis. These studies are kept on file in the Quality Systems Office. The retention times in Table 5.5.2 below are examples. The exact retention times will vary as a function of column type, column age, and column history. For the instruments that use this method, true retention times and retention time windows are taken from the most recent retention time window study conducted.

Table 5.5.2
Retention Time Windows

Compound	RT Window (Min.)	RT Window (Min.)	RT Window (Min.)	RT Window (Min.)
	BioRem I Unit		BioRem II Unit	
Carbon Dioxide	5.171	5.340	4.058	4.635
Oxygen	6.537	7.015	5.686	5.721
Nitrogen	7.200	7.626	6.510	6.570
Methane	9.523	9.933	8.874	8.999
Carbon Monoxide	10.475	10.841	10.938	11.302
Methane	0.586	0.609	0.420	0.420
Ethane	0.809	0.835	0.730	0.730
Ethene	1.027	1.050	1.029	1.064
Propane	1.545	1.570	1.871	1.962
Propene	2.822	2.850	3.942	4.225
iso-Butane	3.763	3.807	5.804	6.230
n-Butane	4.351	4.399	6.855	7.379
Hydrogen	4.404	4.480	NA	NA

5.6 Bake-out Procedure

Either overnight, through the weekend or whenever the instrument is not going to be used for several hours, the instrument is put in “bake-out”. With carrier gas continuous flushing through the GC, the temperature on the oven is manually turned up to 210 degrees or as high as the instrument column oven can maintain.

6.0 Secondary Data Review

All analytical data must undergo a minimum of a two-tiered review. The analyst first reviews the data for completeness and accuracy. The data is then submitted to the Group Lead Analyst for final review and the data is entered into the LIMS. Once approved at this level, the data is released as a final report.

7.0 Reporting Limits

The reporting limits for this analysis are listed in Table 7.0 below. Method detection limit studies are run annually in accordance with Microseeps Standard Operating Procedure for the Determination of Method Detection Limits and PQLs (SOP-ADM 18).

Those MDLs must be less than the reporting limits specified below. MDL studies are also performed when there is reason to suspect that method sensitivity has changed. The MDL studies are kept on file in the Quality Systems Office.

Reporting Limits
Table 7.0

Parameter	Reporting Limit	Units
Carbon Dioxide	0.2	% V
Oxygen	0.1	% V
Nitrogen	0.1	% V
Hydrogen	0.5	ppmv
Acetylene	0.1	ppmv
Methane	0.2	ppmv
Ethane	0.02	ppmv
Ethene	0.03	ppmv
Propane	0.05	ppmv
Propene	0.1	ppmv
n-butane	0.07	ppmv
i-butane	0.05	ppmv

7.1 Conversion Factors

This procedure is used to measure the volume concentration of the analytes in a gas. Two methods are used to extract that gas from the groundwater. The conversion factors that are used to convert the concentration of the analytes in the water from the concentration of the analytes as they are measured using this method, are specific to the collection or preparation method and can be found in either SOP-SM9 or SOP-PM 01.

8.0 Safety

Gloves, proper eye protection, and a laboratory coat shall be worn when handling samples and standards. The major hazard in this laboratory area is stick from needles. All needles must be

capped when not in use and when moving about the laboratory. The proper way of capping a needle is to place the cap on the laboratory bench and direct the needle into the cap. A needle is never to be directed into a cap while the cap is being held.

All compressed gases are to be moved using a dolly made for transporting gases and shall be chained in place when in the laboratory. The chain shall be tightened sufficiently to keep the cylinder upright if jostled.

9.0 Laboratory Waste

Samples are kept for 30 days following analysis. Samples are disposed according to Microseeps Standard Operation Procedure for Waste Disposal (SOP-ADM 14).

9.1 Waste Minimization

Where possible, Microseeps takes steps to minimize the amount of waste generated in the laboratory by using substitution, where possible, and good chemical handling procedures. For specific information on waste minimization consult SOP-ADM 14.

10.0 References

Citing a reference does not imply that all of the recommendations and/or requirements in those cited methods is required in this Standard Operating Procedure. This section simply refers to sources that were consulted to gather information or knowledge in order to write an informed technical procedure.

U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste. SW-846, 3rd ed., Office of Solid Waste and Emergency Response, Washington, DC. 1986.

Newel, B.S., RSKSOP-175, Sample Preparation and Calculations for Dissolved Gas Analysis in Water Samples using a GC Headspace Equilibration Technique. Revision No. 0, August 1994.

Newel, B.S., RSK-SOP-147, Gas Chromatographic Analysis of Gaseous Samples for Part-Per-Million Levels of Nitrous Oxide, Methane, Ethylene, and Ethane. Revision No. 0, August 1993.

American Society for Testing and Materials, Standard Practice for Analysis of Reformed Gas by Gas Chromatography. Annual Book of ASTM Standards. Vol. 14.02, 1994.

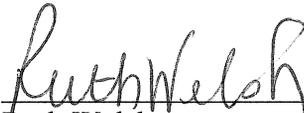
Kampbell, D.H. and Vandegrift, S.A., Analysis of Dissolved Methane, Ethane, and Ethylene in Ground Water by a Standard Gas Chromatographic Technique. Journal of Chromatographic Science. Vol. 36, May 1998.

Microseeps, Inc.

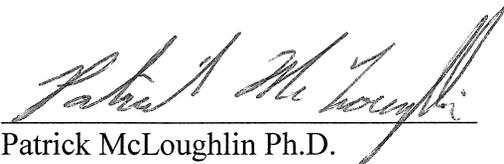
Standard Operating Procedure for Sample Receiving

Controlled Copy No. _____

Signature of Final Approval:



Ruth Welsh
Laboratory Manager



Patrick McLoughlin Ph.D.
Technical Director

Date: 5-31-07

1.1 Purpose

The purpose of this Standard Operating Procedure is to outline the procedures for sample receipt and storage.

1.2 Applicability

This Standard Operating Procedure applies specifically to the sample custodian and/or his or her representative during activities of sample receipt. This Standard Operating Procedure also applies to the Customer Service Office and Project Management Staff and bottle preparation personnel.

2.0 Definitions

Aliquot: a portion of a sample

Background Radiation: naturally occurring radiation.

Chain of Custody Form: record that documents the possession of the samples from the time of collection to receipt in the laboratory. The record may include: number and types of containers; the mode of collection; time of collection; preservation; requested analyses; and sampler's printed name and signature.

Holding Time: the maximum time that samples may be held prior to analysis.

Non-Conformance: samples or sample documentation that are received with incorrect, incomplete, or inadequate information or properties.

Preservation: refrigeration and/or reagents added prior to sample collection to maintain the chemical, physical, and/or biological integrity of the sample.

Shall: denotes a requirement that is mandatory.

Short Holding Time: samples that must be analyzed within 48 hours or less of sample collection.

Temperature Blank: a bottle of water that accompanies the samples in each cooler. This blank is used to monitor cooler temperature upon receipt of samples in the laboratory.

Trip Blank: a set of 40mL VOA vials filled with deionized water that travel with samples to be analyzed for volatile organic compounds. These samples are analyzed to determine if cross contamination occurred during transport.

3.0 Responsibilities

The responsibility of the sample custodian is to sign and date all appropriate receiving documents including chains of custody and shipping papers. Signing the chain of custody indicates that the laboratory accepts the samples in their condition upon arrival. Conditions under which chains of custody will be signed or left unsigned are discussed in Section 4.0 of this Standard Operating Procedure. The Sample Custodian is also responsible for notifying the Customer Service Department for all discrepancies that occur during the sample receipt process.

In all cases, the client shall be consulted to resolve all discrepancies involving their samples. The Sample Custodian shall log samples in accurately and in a timely manner. The Sample Custodian reports to the Laboratory Director.

3.1 Reporting Level Requirements and Responsibility

Microseeps' Project Manager(s) have the responsibility to alert the Sample Custodian, in writing or through the Laboratory Information Management System, of any projects that require more than standard reporting requirements prior to the delivery of those samples. Microseeps identifies the specific reporting levels that are outlined in Exhibit 1. Quality control reporting levels other than standard must be arranged with a member of Customer Service prior to delivery of the samples. That member of Customer Service will then serve as the project manager for that project. If notification is not made prior to sample receipt, the level of reporting requested by the client cannot be assured.

3.2 Non-Standard Custody Requirements and Responsibility

Microseeps' standard custody procedures do not involve intra-laboratory tracking of samples. The Project Manager shall notify the Laboratory Director prior to receipt of samples that will require extra security. If such security is required, it is treated as a client specific quality control reporting requirement. A Microseeps' Project Manger must be assigned to the project, and the Sample Custodian must be made aware of these special requirements prior to the sample receipt.

If notification is not made prior to their delivery, it may not be possible to provide the level of security requested by the client.

3.3 General Documentation Requirements and Responsibility

The responsibility for quality control and documentation during sample receiving requires a diligent effort to ensure that all documentation is present and complete and that security measures are maintained where required. The Sample Custodian is responsible for sample receipt documentation. The Customer Service Office is responsible for client contact and documenting client responses in the event of non-conformance.

All records must be written in ink or printed electronically. When any changes are made, a single line should be drawn through the errors and the corrections should be initialed and dated. All notations written on chains of custody shall be dated and initialed.

4.0 Sample Receipt Procedure

The following policy and procedures are in place to ensure that all samples and chains of custody that are accepted at Microseeps are thoroughly inspected and all discrepancies are fully documented. A Customer Service Representative shall contact the client in the event that there is any discrepancy involved in the condition or the documentation of samples, upon receipt, that may affect the sample's integrity or the analytical process.

A permanent record of sample receipt shall be maintained electronically in the Laboratory Information Management System (LIMS). At a minimum, that record will include: (1) Client Name; (2) Project Name; (3) Date and time of sample receipt; (4) Unique laboratory identification code; (5) Name or initials of the person making the entries; and (6) requested analyses.

4.1 Sample Acceptance Policy

A printed copy of this Sample Acceptance Policy (see Exhibit 2) is forwarded to sample collection personnel as a part of their bottle order shipment.

1. Samples that are shipped to Microseeps must be accompanied by proper, full and complete documentation. This documentation shall be marked on a chain of custody and shall include: sample identification, the location, date and time of collection, sampler's name, preservation type, sample type, specific parameters to be analyzed, and any special remarks concerning the sample.
2. Sample labels shall be supplied by Microseeps or the client. Those labels must be water resistant and completed using indelible ink. Each sample label must include a unique identification number that links it to the chain of custody documentation.
3. Samples shall be in the proper containers with the preservatives that are specific to the type of analysis required.
4. All samples must be received within specified holding times. Clients are requested to notify a Microseeps' Customer Service Representative if samples with short holding times are being shipped.
5. Samples must arrive at Microseeps with sufficient volume to conduct the requested analyses. All bottles should be filled completely.
6. When problems with samples or documentation are found during the sample receiving process, a Non-Conformance Form (Exhibit 3) is completed by the sample custodian and forwarded to the Customer Service Office. A Customer Service Representative will make every attempt to contact the client as soon as possible to make decisions concerning

those discrepancies. The Non-Conformance Form is kept as a permanent part of the project file.

7. If the client cannot be reached, a message will be left either on voice mail or with a receptionist for the client to return the phone call. Correspondence may also occur through the use of email. The samples will be placed in a storage refrigerator and held until Microseeps' Customer Service Representative gets a response from the client. (Exceptions will be made when samples are received that have short holding times and the samples are from a client with whom Microseeps has regular and frequent dealings; or when the samples have short holding times and the samples are from a client with whom Microseeps has a signed contract, work order, or purchase order.)

4.1.1 Non-Conformance Notification

Any analyses that are conducted on samples that do not meet these acceptance criteria will be appropriately qualified in the final report packet. The non-conformance form will be included with the final report.

4.2 Sample Rejection Criteria

The following situations dictate when samples will be rejected:

1. Coolers and samples arrive at Microseeps' facility with no client identification.
2. Samples arrive with no chain of custody and there is no means to obtain one.
3. Samples that fail the radiation screen according to the criteria set forth in the Radiation Screening Procedures in Section 4.3 of this Standard Operating Procedure.
4. Coolers that arrive with hazard labels on them for which Microseeps is not equipped or certified. A chart listing those hazards is posted in the Sample Custodian's Office.

4.3 Sample Receipt Procedures

1. Prior to signing shipping documents from courier, ensure that the number of packages listed on the shipping documents corresponds with the number of packages actually delivered.
2. The courier must document existing discrepancies before the Sample Custodian accepts the packages. Document the discrepancy on a Non-Conformance Form and forward it to the customer service office for immediate action.
3. Put on safety glasses and protective gloves before handling, opening, or unpacking packages and coolers that contain environmental samples.

4. Prior to cooler inspection and opening, determine if any coolers were received from any DOE or DOD facilities. These coolers and packages must be scanned for radiation using the Radiation Alert Monitor 4 according to the following procedures:
5. Turn the Monitor 4 Radiation Instrument to the ON position.
6. Check the battery by sliding the range switch to the BATT position. If the meter indicates a low battery, turn the instrument off and replace the battery with a 9 volt battery and repeat steps 5 and 6.
7. Set the range switch in the X1 position and take a background reading away from the proximity of the packages.
8. Record the date, time, battery check, and background reading in the radiation instrument log. This documentation shall be done at least once a day when the radiation instrument is used.
9. Hold the back of the instrument toward the cooler beginning at a distance of 1 foot and slowly close the distance to one inch. **If the instrument shows a reading above background, leave the room and close both doors. The Technical Director will take appropriate action to remove and/or isolate the cooler.**
10. Inspect all coolers and packages for damaged or broken custody seals. Note any discrepancies on a Non-Conformance Form.
11. Open the cooler and expose the sample bottles. Hold the back of the radiation instrument over the samples within the cooler to a proximity of 1 inch. If there is no reading above background continue with the sample receipt process. **If the instrument shows a reading above background, take the same steps as above in Step 9.**
12. Record cooler temperature on the chain of custody as soon as possible upon opening the cooler. Initial and date the entry. The temperature in the cooler should be $\leq 6^{\circ}\text{C}$. If the temperature is not within those parameters, note the discrepancy on a Non-Conformance Form.
13. Inspect each sample and sample label while removing it from the cooler. Samples containers should be intact. At a minimum, sample labels should be completed with the following information:
 - ◆ Sample Number
 - ◆ Time of Collection
 - ◆ Site Name
 - ◆ Parameters to be analyzed

If samples cannot be properly identified by label inspection, note the discrepancy on a Non-Conformance Form.

14. If samples were received in grouped sets, keep the sets grouped together as they are unpacked. If samples were not received in sets, organize them into sets while unpacking.
15. Match the sample identifications to the Chain of Custody. Note any discrepancies on a Non-Conformance Form
16. If the project requirements specify data reporting above Microseeps' standard level, the custodian must ensure that each group of 20 samples has at least one duplicate and one spike set. Note any discrepancies on a Non-Conformance Form.
17. Make sure that field and trip blanks are present and identified. Document all missing or potentially missing samples on a Non-Conformance Form.
18. Check the Chain of Custody to ensure that all samples are entered and the specific analysis is listed for each bottle. Note all discrepancies on a Non-Conformance Form.
19. Check appropriate samples for proper sample preservative using pH paper according to the procedure outlined in Section 6.0 of this Standard Operating Procedure. Samples that are improperly preserved are to be documented using a Non-Conformance Form.
20. Ensuring that all sample receipt documentation is complete, sign the chain of custody form.

4.3.1 Weekend Sample Receipt Procedures

The weekend analyst will follow the abbreviated sample receipt process below in order to comply with sample holding times. The complete sample receipt process will occur during normal business hours the following Monday.

1. Scan coolers for radiation prior to and during opening following instructions above.
2. Remove samples that have short holding times for immediate analysis.
3. Leave detailed message with Sample Custodian on what bottles were taken. This may be done via email, voicemail, or written note.

4.3.2 After Hours Receipt Procedure

Because the Sample Custodian is not typically present before or after normal business hours, samples that are received during those times shall be placed into the cooler in the Sample Receiving area until the sample custodian processes them on the following business day. If a client wishes to have samples processed outside of normal business hours they must make those

arrangements with Microseeps Customer Service Office prior to sample delivery. The Laboratory Director must approve those arrangements in writing.

4.3.3 Sample Receipt Discrepancies

If there is any discrepancy, problem, or situation with the samples or the above steps that is out of the ordinary, a Non-Conformance Form must be completed immediately and submitted through the proper channels as specified in Section 5.0 of this Standard Operating Procedure.

After all sample receipt documentation has been completed and the samples have been thoroughly examined, the sample custodian creates a batch file and begins the process of Sample Log-In in accordance with the Sample Log-In Standard Operating Procedure.

5.0 Non-Conformance Forms

The Non-Conformance Form is Microseeps' primary documentation tool for sample receipt problems or discrepancies that require client contact or corrective action. It is imperative that Non-Conformance Forms are completed accurately and submitted to the customer service office in a timely manner.

Any non-standard requirements that were not negotiated in advance of sample receipt such as rapid-turnaround, particular reporting limits, or particular sample disposal instructions must be documented on a Non-Conformance Form.

Non-Conformance Forms are to be completed by the Sample Custodian under the following conditions:

- Broken containers
- Label information inadequate to properly identify samples
- Information on COC inadequate to properly log-in samples
- Missing or extra samples
- Conflict between bottle labels and COC (except as noted below)
- Samples received past holding times (except as noted below)
- Improperly preserved samples
- Any other circumstances that are out of the ordinary

Exceptions: In the following situations, non-conformance forms are not required:

- Samples received for pH, total residual chlorine, and oxidation-reduction potential analyses are always received outside of the specified holding time; therefore a non-conformance form is not required. A notice in the narrative portion of the final report must indicate that samples for pH were received out of hold.
- Where the only discrepancy between the sample label and the COC is the sample collection time, and that discrepancy is less than one hour, a non-conformance form shall

not be generated and the sample collection time noted on the COC shall be used for log-in purposes.

5.1 Non-Conformance Form Submissions and Handling

The Customer Service Office is to be notified as soon as possible when a Non-Conformance Form is issued. Customer Service is to expedite their completion of the Non-Conformance Process and return the documentation to the Sample Custodian as quickly as possible. The original and a copy of the Non-Conformance Form is to be placed in the Project.

5.2 Non-Conformance Form Completion

A Non-Conformance Form is to be completed by the Sample Custodian as follows:

1. Complete date, client name, name of person who received samples, and time of sample receipt.
2. Using the lines on the form, ensure adequate information is provided to explain the non-conformance.
3. Add additional information, if necessary, on the back of the Non-Conformance Form.
4. Submit form to Customer Service Office.

The bottom half of the form is to be completed by the Customer Service Office as follows:

1. Customer Service shall document the action taken on the form.
2. Customer Service will then initial and date the form at the bottom.
3. The form will then be returned to the Sample Custodian who will check it for completeness.
4. The Sample Custodian will then log in the samples accordingly, and write the Microseeps project number on the top of the non-conformance form, copy the form, and place the original and the copy in the project file where one copy becomes a permanent part of the project file. The other copy of the Non-Conformance form shall be mailed to the client as a part of their final report package.

6.0 Checking Sample Preservative

In order for water samples to be considered valid, they must be either cooled and/or chemically preserved according to the type of analyses each sample will undergo. All appropriate samples

for analyses that require chemical preservatives shall be checked upon log-in for proper preservative.

Samples that arrive in VOA vials with zero headspace shall not be tested for preservative. VOA vials are not to be opened by the Sample Custodian. Other samples that would degrade upon contact with air shall also not be checked for preservative by the Sample Custodian.

6.1 pH Screening Equipment

Equipment necessary for this procedure is as follows:

1. Disposable pipettes
2. Clean secondary disposable containers
3. pH paper and pH scale

6.2 Acceptable Limits for pH of Preserved Samples

Samples that are preserved with acids should have a pH of two or less. Samples that are preserved with bases should have a pH of twelve or greater.

6.3 Procedures for Checking Sample Preservative

1. Ensure proper personal protective equipment is being worn, i.e. safety glasses and protective gloves.
2. Open sample container to be tested and pull a small amount of the sample into a disposable pipette.
3. Squeeze the aliquot into a clean disposable plastic dish.
4. Immerse the pH paper into the aliquot in the plastic dish.
5. Compare the pH paper to the chart to determine the pH range of the sample aliquot.
6. The pH check shall be documented on the internally generated LIMS cooler receipt form.
7. Dispose of the used pipette and dish. Use a new set for each bottle tested.

6.4 Preservative Check Documentation

All samples that are checked for preservative will be documented in the Laboratory Information Management System (LIMS) on the Cooler Receipt Form. This form is available electronically and becomes a permanent part of the electronic record and is maintained for a minimum of five years.

7.0 Sample Storage

Samples shall be stored according to the conditions specified by preservation protocols. The storage conditions shall be maintained, monitored, and documented. Samples shall be stored away from all standards, reagents, food and other potentially contaminating sources. Samples shall be stored in segregated areas to prevent cross contamination.

7.1 Sample Storage Temperature Documentation and Responsibility

Samples which require thermal preservation shall be stored under refrigeration or frozen. It is the Sample Custodian's responsibility to insure that the refrigerator and freezer temperatures within the sample receipt area are monitored and recorded on the posted temperature log twice a day. A temperature of $\leq 6^{\circ}\text{C}$ is acceptable for a refrigerator that has been closed overnight. A temperature of 0°C or less is acceptable for a freezer. The temperature logs are to be maintained in the Sample Custodian's Office.

7.1.1 Corrective Action for Refrigerator Temperature Beyond Control Limits

If the temperature is outside of the acceptable temperature range limits, the Sample Custodian must immediately notify the Laboratory Manager and the Technical Director. Maintenance will be arranged through the Facility Manager or his representative. If it is apparent that the proper sample temperature cannot be maintained in the area needing maintenance, then every effort will be made to move samples to another cooler that is functioning within the temperature control limits.

7.2 Samples Requiring Extra Security

If the sample custodian has previously been notified by the Microseeps' project manager that the samples require high security, the sample custodian is to complete an internal chain of custody form for each bottle type using our standard chain of custody form. Once the log-in process is complete the samples are to be placed in a cooler in a secure room. A sample tracking record with the appropriate bottle type circled shall be posted on the outside of the cooler. Only Microseeps personnel will have access to that area, and will sign out those samples when they are taken for analysis. Employees who have signed out samples will keep them in their possession at all times, or in a secure area, until such time as the analysis is complete or the remainder of the sample(s) and/or extract(s) is/are returned to the original locked location. A sample tracking record form is displayed in Exhibit 5.

8.0 Safety

Personnel safety is a priority at Microseeps. All employees are required to wear appropriate personal protective equipment in accordance with Microseeps Chemical Hygiene Plan. The Sample Custodian has been provided with safety glasses, protective gloves, and a laboratory coat.

It is required that the Sample Custodian wear gloves and safety glasses while handling all coolers and samples. Coolers shall be opened in a well-ventilated area. If odors are detected upon opening, the cooler shall be closed and moved to an area with a fume hood before proceeding with the sample receipt procedures.

9.0 Equipment and Reagents

The following equipment is to be available for the Sample Custodian to accomplish the procedures outlined in this Standard Operating Procedure:

1. Sink
2. Calibrated thermometer (e.g. temperature gun)
3. Storage Refrigerator
4. Computer with printer
5. Safety Glasses
6. Chemical protective gloves
7. pH Paper
8. Disposable pipettes
9. Disposable plastic containers
10. Radiation Screening Instrument
11. Cooler thermometers

9.1 Radiation Instrument

The instrument used for scanning coolers and packages for radiation is an S.E. International, Inc. Monitor 4 powered by a 9 volt radio battery.

9.1.1 Radiation Instrument Calibration and Frequency

The Monitor 4 Radiation instrument is calibrated yearly by S.E. International by pulse generator and is typically $\pm 15\%$ of full scale relative to Cesium 137.

9.1.2 Background Level Determination

There are many natural factors which affect background radiation levels at any given time therefore; a general background reading is taken in the Sample Receipt room each time the instrument is turned on.

10.0 References and Documentation

10.1 Example Forms and Documentation

1. Exhibit 1 lists Microseeps Standard Data Package
2. Exhibit 2 Sample Acceptance Policy
3. Exhibit 3 Non-conformance Form
4. Exhibit 4 Sample Tracking Record

10.2 References

1. National Environmental Laboratory Accreditation Conference (NELAC) Program Policy and Structure Manual. 2000. Chapter 5, Section 5.11.
2. Radiation Alert, Operation Manual for the Monitor 4, Monitor 4EC, Monitor 5, and MC1K. 1998. pp. 6 - 17.

SOP Review Date: May 29, 2007

EXHIBIT - 1

Standard Data Package

Microseeps, Inc. defines the following QA/QC level as standard for reporting. Requests for reporting beyond "Standard" level QA/QC **MUST** be made during the quotation process.

Standard

- all client sample results (compound, concentration and units)
- volatiles only: trip blanks results *available only if the client provided a trip blank*
- laboratory project and sample ID
- method number
- dilution factor corrected reporting limits
- sample collection date & time *if supplied by the client*

- copy of chain of custody submitted by client
- abbreviated case narrative, *if there are exceptions*

Hard copies of the calibration files from the associated analyses, the raw data (i.e. chromatograms or data sheets) and hard copies of the associated prep log book, analysis log book and standard log book entries are only available by special arrangement and quotation made prior to the start of the project.

Controlled Document

Exhibit 2

Sample Acceptance Policy

1. Samples that are shipped to Microseeps must be accompanied by proper, full and complete documentation. This documentation shall be marked on a chain of custody and shall include: sample identification, the location, date and time of collection, sampler's name, preservation type, sample type, specific parameters to be analyzed, and any special remarks concerning the sample.
2. Sample labels shall be supplied by Microseeps, or the client. Those labels must be water resistant and completed using indelible ink. Each sample label must include a unique identification number that links it to the chain of custody documentation.

3. Samples shall be in the proper containers with the preservatives that are specific to the type of analysis required.
4. All samples must be received within specified holding times. Clients are requested to notify a Microseeps' Customer Service Representative if samples with short holding times are being shipped.
5. Samples must arrive at Microseeps with sufficient volume to conduct the requested analyses. All bottles should be filled completely.
6. When problems with samples or documentation are found during the sample receiving process, a Non-Conformance Form (Exhibit 3) is completed by the sample custodian and forwarded to the Customer Service Office. A Customer Service Representative will make every attempt to contact the client as soon as possible to make decisions concerning those discrepancies. The Non-Conformance Form is kept as a permanent part of the project file.
7. If the client cannot be reached, a message will be left either on voice mail or with a receptionist for the client to return the phone call. Correspondence may also occur through the use of email. The samples will be placed in a storage refrigerator and held until Microseeps' Customer Service Representative gets a response from the client.

Non-Conformance Notification

Any analyses that are conducted on samples that do not meet these acceptance criteria will be appropriately qualified in the final report packet. The non-conformance form will be included with the final report.

Sample Rejection Criteria

The following situations dictate when samples will be rejected:

1. Coolers and samples arrive at Microseeps' facility with no client identification.
2. Samples arrive with no chain of custody and there is no means to obtain one.
3. Samples that fail the radiation screen according to the criteria set forth in the Radiation Screening Procedures in Section 4.3 of this Standard Operating Procedure.
4. Coolers that arrive with hazard labels on them for which Microseeps is not equipped or certified. A chart listing those hazards is posted in the Sample Custodian's Office.

This page left intentionally blank.

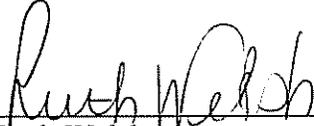
Controlled Document

Microseeps, Incorporated

Standard Operating Procedure for the Determination of Organic Carbon in Water Samples

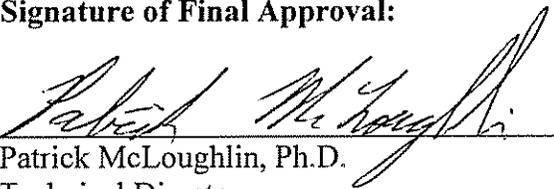
(Ref. Method SW846-9060)

Controlled Copy _____



Ruth Welsh
Laboratory Director

Signature of Final Approval:



Patrick McLoughlin, Ph.D.
Technical Director

Date: 3-1-05

SOP Review Date: March 1, 2005

1.0 Purpose and Application

This Standard Operating Procedure is used as guidance to determine the concentration of non-purge-able organic carbon in water samples. SW846-9060 refers to total organic carbon as will this SOP for the sake of continuity. This method is most applicable to the measurement of organic carbon in homogeneous samples above 1 mg/L.

1.1 Analyte List

This method is applicable to the following:

- Total Organic Carbon (TOC)
- Dissolved Organic Carbon (DOC)

1.2 Matrices

SW 846 9060 is applicable for groundwater, surface and saline waters, and domestic and industrial wastes.

2.0 Method Summary

The HiPerTOC can select either or both wet chemical and combustion methods for oxidation. The sample is reacted with an oxidizer and ultraviolet radiation which releases any organic carbon in the sample in the form of carbon dioxide, or the sample is combusted in a furnace at 1000 degrees Celsius to produce carbon dioxide. The carbon dioxide produced is swept by a stream of oxygen to the detector. During analysis, carbon dioxide levels are measured by a non-dispersive infrared (NDIR) detector. The detector is sensitive to the absorption frequency of carbon dioxide and provides a signal proportional to the instantaneous concentration of carbon dioxide in the carrier gas oxygen, flowing through it. The detector output signal is linear and provides a reading of total organic carbon (TOC). The linear signal is integrated and compared to stored calibration data to compute the sample carbon concentrations in parts per million carbon (ppm C).

2.1 Definitions

Purge: to sparge, to allow gas to flow through a sample to remove volatile elements in a sample.

NDIR: non-dispersive infrared detector; a measuring instrument that provides an electrical signal that is proportional to the concentration of carbon dioxide in the detector.

Oxidation: a reaction in which an element's capability to unite with other substances increased due to the element's loss of electrons.

Sodium Persulfate: ($\text{Na}_2\text{O}_8\text{S}_2$) an oxidizing agent used to convert inorganic carbon into carbon dioxide.

Duplicates: two sub-samples of the same sample analyzed within a short time interval.

Laboratory Control Sample: sample matrix free from analytes of interest, spiked with verified known amounts of analytes. The LCS is used to assess the performance of the measurement system.

Matrix Spike: sample prepared by adding a known concentration of target analyte to a specific amount of sample. Matrix spikes are used to determine the effect of sample matrix on a method's recovery efficiency.

Method Blank: a sample of similar matrix that is free from the analytes of interest that is processed through all the steps of the analysis with other samples. No target analytes should be present at concentrations that impact the analytical results.

2.2 Method Limitations

2.2.1 This procedure is applicable only to homogeneous samples that can be injected into the HiPerTOC. The syringe openings limit the size of particles that can be injected with the sample.

2.2.2 Method limitations include interferences from carbonate and bicarbonate that may be present in the samples. Those substances must either be removed from the sample, or accounted for in the final calculation. This method calls for removal of carbonate and bicarbonate by acidification and purging the acidified sample with nitrogen however, the data user should be aware that this purging may result in the loss of some volatile carbon compounds.

3.0 Apparatus, Materials, and Operating Conditions

3.1 Apparatus

- ThermoElectron HiPer TOC
- Autosampler: 55 position xyz robot with stationary rack design.
- Software: ThermoElectron Theus

3.2 Materials

- 40 ml amber VOA vials
- Compressed oxygen, gas research grade
- Class A volumetric flasks
- Class A volumetric pipettes
- 125 mm Whatman glass microfibre filter

3.3 Operating Conditions

- Oxygen operating pressure: 15-30 psi

4.0 Reagents

All chemicals and reagents used in this procedure are laboratory reagent grade. Stock standards are standardized in the laboratory using ASTM Type II ultra pure deionized water, or purchased as certified standards or stock solution. Reagent and standards logbooks are maintained in the laboratory.

4.1 Standard Preparation Procedures

- **Sodium Persulfate:** Dissolve 238g of ultra pure reagent grade sodium persulfate into one liter of distilled water.
- **Phosphoric Acid Solution:** Dilute 117.6 ml of 85% H₃PO₄ to one liter of distilled water.
- **1000 ppmC TOC Standard:** Purchased from SCP Science AccuSpec Cat#250-250-051
- **Calibration Standards:** Calibration standards can be purchased from a reliable vendor. If prepared in-house, they are prepared by diluting a volume of the 1000 ppm C potassium hydrogen phthalate solution with 100 ml of deionized water in accordance with the following table:

Table 4.1
Calibration Standards

ml of 1000 ppmC Stock	Calibration Standard Concentration	Standard Concentration in µg C
Blank	0 ppm C	0 µg C
0.05	0.5ppm C	0.025µg C
0.1	1.0ppm C	0.5µg C
0.5	5.0ppm C	2.5µg C
1.0	10.0ppm C	5µg C
2.5	25ppm C	12.5µg C
5.0	50ppm C	25µg C

- **Laboratory Control Sample:** The LCS is purchased from ERA as a certified second source solution. Deionized water is added to 5 ml of the purchased solution to 1000 ml.
- **CCV:** Prepared at 25 ppm using the same source and procedure as the calibration standard preparation above but prepared in an independent batch.

4.2 Glassware and Storage Requirements for Reagents and Standards

Stock standards are to be stored in 1000 ml amber glass jars and refrigerated.

4.3 Surrogate Standards

Not applicable.

5.0 Procedure

Samples are to be collected in 250 ml polypropylene bottles and cooled to 4°C. Samples are preserved with sulfuric acid to a pH of less than 2. The holding time is 28 days, and samples should be protected from sunlight. Samples are to be stored refrigerated at 4° C ± 2°.

Analysts who use this method have been certified for the method by running Initial Demonstration of Proficiency (IDOP) Samples in accordance with Microseeps Standard Operating Procedure for Administering and Documenting Training in Laboratory Procedures and Instrumentation (SOP ADM 02). IDOPs are run any time there is significant change to an instrument, method, or in the Training procedure for training a new analyst.

5.1 Sample Preparation

If dissolved or soluble organic carbon are requested, the samples are to be filtered through a 125 mm Whatman glass microfibre filter. It is preferable that all filtering occur in the field prior to sample preservation. A sample should not be filtered once it has been preserved.

5.2 Calibration

Prepare calibration standards in accordance with Table 4.1. 2-ml injections of each standard are analyzed as a samples and the instrument software is used to combine the calibration and method file. The calibration is linear, with the calibration points (concentration vs. instrument response) fit via a linear regression routine. The coefficient of correlation must be 0.9950 or higher for the initial calibration to be acceptable. If this criterion is not met, the system should be inspected, the calibration repeated, and all samples since the last acceptable CCV are rerun.

An ICV is run immediately following the initial calibration. The % drift should not exceed ± 10%.

5.3 Sample Analysis

An instrument run or sample batch is a single set of twenty or fewer samples that is analyzed on a given day. All client samples are homogenized and representative aliquots are transferred into 40 ml VOA vials that can be loaded into the auto sampler. All samples will be analyzed in quadruplicate.

5.3.1 Open **THEus** on computer console and follow the instructions bulleted below:

- Check if main reagent containers and that the waste container is empty. Fill the GLS (gas liquid separator) with DI.
- Manually set the desired temperature in the **System Status** window. This will activate the furnace and other heaters.
- Manually move the XYZ autosampler into a position where vials can be easily removed.
- Fill a few vials with DI Water and prepare standards.
- Open the **Queue Manager** window and build a sample queue and attach the appropriate method file, sample volume, units, calibration line, dilution factor, etc.
- Open the oxygen line and check for leaks.
- Open the **System Status** window and start running a baseline.
- Check that desired temperatures are reached. (indicator light turns green).
- Open the **Queue Manager** window, select appropriate sample queue and press the start button.

5.3.2 Check gas supplies and replenish, if necessary, during warm-up phase. Put all quality control samples into 40 ml vials to be loaded into auto sampler.

5.3.3 After the cleaning procedure, the continuing calibration verification is run followed by the continuing calibration blank, the performance blank, and then the laboratory control sample. After these samples come a series of fifteen client samples (in the 40 ml vials) and then the series is repeated except for the LCS. The LCS is run every twenty samples. Continue loading rack until full or all samples are loaded.

5.3.4 Confirm that the queue selected on the screen matches the auto sampler rack. When analyzed samples are at a concentration >50 ppm, the samples are diluted and re-analyzed.

5.4 Quality Control Requirements

The following are analyzed once per batch unless otherwise specified:

5.4.1 Method blank using 18 Mohm water.

Any TOC detected in the method blank must be present at less than the concentration of TOC in the lowest standard.

Corrective Action: If this criterion is not met, inspect all glassware and equipment, and prepare and analyze another method blank. Method blanks should continue to be run until this criterion is met. If three blanks are analyzed in succession and this criterion is still not met, the laboratory director should be notified.

5.4.2 Laboratory Control Sample

A laboratory control sample is analyzed once for every 20 or fewer client samples. The acceptance criterion for the LCS is a percent recovery between 70% and 130%.

Corrective Action: If the LCS fails a new calibration curve shall be run and all samples since the last acceptable LCS shall be re-analyzed.

5.4.3 CCV

An independently prepared CCV is run every fifteen samples to verify calibration. The CCV shall be followed by a continuing calibration blank. The criterion for acceptance for the CCV is a % recovery of $\pm 10\%$.

Corrective Action: If the CCV fails, the instrument shall be recalibrated, and all samples since the last acceptable CCV shall be re-analyzed.

5.4.4 Matrix Spike and Matrix Spike Duplicate

A matrix spike and spike duplicate are analyzed every 10 client samples. The acceptance criterion is a percent recovery of between 70% and 130%.

Corrective Action: If the matrix spike fails and the rest of the quality control is within the acceptance criteria, matrix interference should be noted in a case narrative.

5.4.5 Contingency for Handling Out of Control or Unacceptable Data

All samples associated with out of control quality control samples (with the exception of matrix interference) must be reanalyzed. If quality control acceptance criteria cannot be met using the corrective action above, a detailed check of the deionized water and chemical purity is made. Reagents, standards, and other quality control samples are re-prepared and analyzed. If problems persist, sample analysis will be halted and the Technical Director shall be contacted immediately to determine the cause and implement corrective action.

Any data submitted with unacceptable quality control sample results shall be qualified in a case narrative. The narrative should indicate the out of control event that occurred, the corrective action that was taken, and any other pertinent information to inform the client of exactly what occurred.

5.5 Capturing and Submitting Data

The software used for the HiPer TOC is specific to the instrument and is called Theus. Analytical results are generated and printed using the THEus software and reported in parts per million.

The formula used for calculating concentration or instrument response is:

$$y = mx + b$$

Where: y = signal
 m = slope
 x = concentration (area count)
 b = y intercept

The instrument run log is generated by the THEus software. These logs are printed and placed into a three-ringed binder and kept in the laboratory. The current and previous years' raw data are maintained in the laboratory; older records are archived in the Records Storage Area and are maintained for five full years. All instrument maintenance is recorded in the Instrument Maintenance Logbook, which is kept in the laboratory.

5.5.1 Blank Correction

A blank is run prior to calibration. This blank will contain normal reagent volumes and the calibration volume of DI water. The measured area of this blank is saved and subtracted from the area of the calibration standard. This causes the carbon coefficient of the standard to represent only the desired carbon content of the standard. After the wash cycle is complete, a reagent blank is run. This blank comprises normal reagent volumes but no DI volume. The measure area of this blank is saved and subtracted from all samples run in the method. All of the raw data for blanking and calibration are stored in the data file.

6.0 Secondary Data Review

It is the analyst's responsibility to insure that all calibrations, calibration verifications, matrix spikes, laboratory control samples, duplicates, and blanks are within the acceptance criteria outlined in this Standard Operating Procedure.

Precision is monitored by calculating the relative percent difference (RPD) between a sample and a sample duplicate.

Accuracy is monitored by calculating the percent recovery of a spiked sample.

6.1 Peer Review

A peer review is not currently conducted on the data derived from this analysis. Therefore, a more thorough review is conducted by the Laboratory Manager or their designee.

6.2 Laboratory Manager Review

The Laboratory Manager reviews 10% of all laboratory data. This review includes sample results, quality control acceptance limits, and a review of the level of quality control required for the project. This review does not necessarily occur in real time.

6.3 Performance Evaluations

Performance evaluation studies are conducted on this method annually, or more often, in order to maintain laboratory certifications or accreditations.

7.0 Reporting Limits

The reporting limit for TOC is 5.0 ppm. Reporting limits are typically 3 to 5 times the MDL.

Method detection limit studies are run annually in accordance with Microseeps Standard Operating Procedure for the Determination of Method Detection Limits and PQLs (SOP-ADM 18).

8.0 Safety

Safety glasses are required in all laboratory areas. For further information regarding safety issues, please refer to the Microseeps Chemical Hygiene Plan.

9.0 Waste

All sample waste and laboratory-generated waste shall be handled in accordance with Microseeps' Standard Operating Procedure for Waste Disposal.

9.1 Waste Minimization

Waste minimization is to be implemented where applicable. For more specific information concerning waste minimization, please consult Microseeps' Standard Operating Procedure for Waste Disposal.

10. References

U.S. Environmental Protection Agency, 1986, Test Methods for Evaluating Solid Waste, Volume C. Third Ed., SW 846-9060. Washington D.C.

HiPerTOC User Manual, Thermo Electron Corporation.

Controlled Document

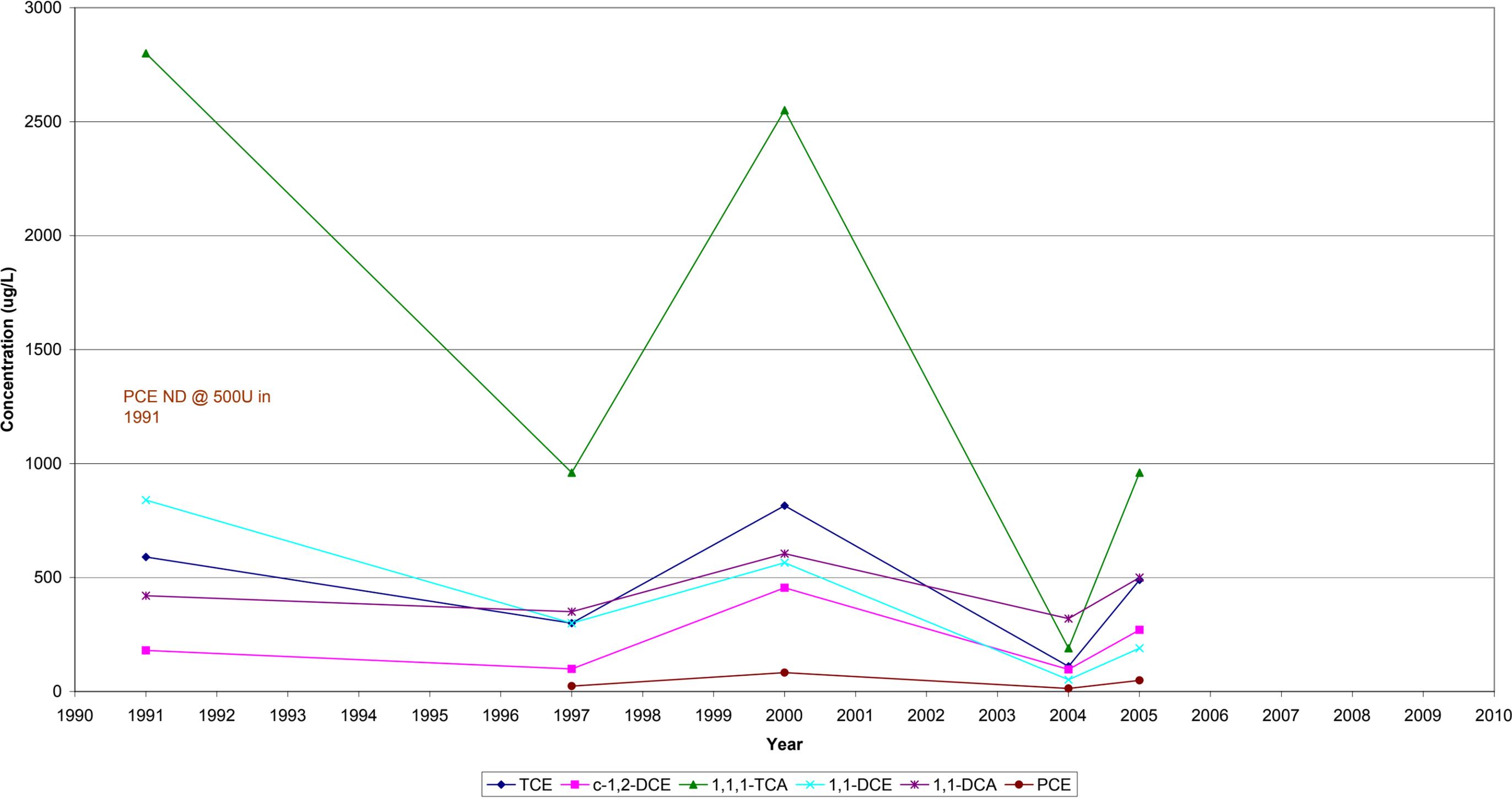
Project-Specific SAP
Site Name/Project Name: NAS JRB Willow Grove Site 5
Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study
Revision Number: 0
Revision Date: September 2008

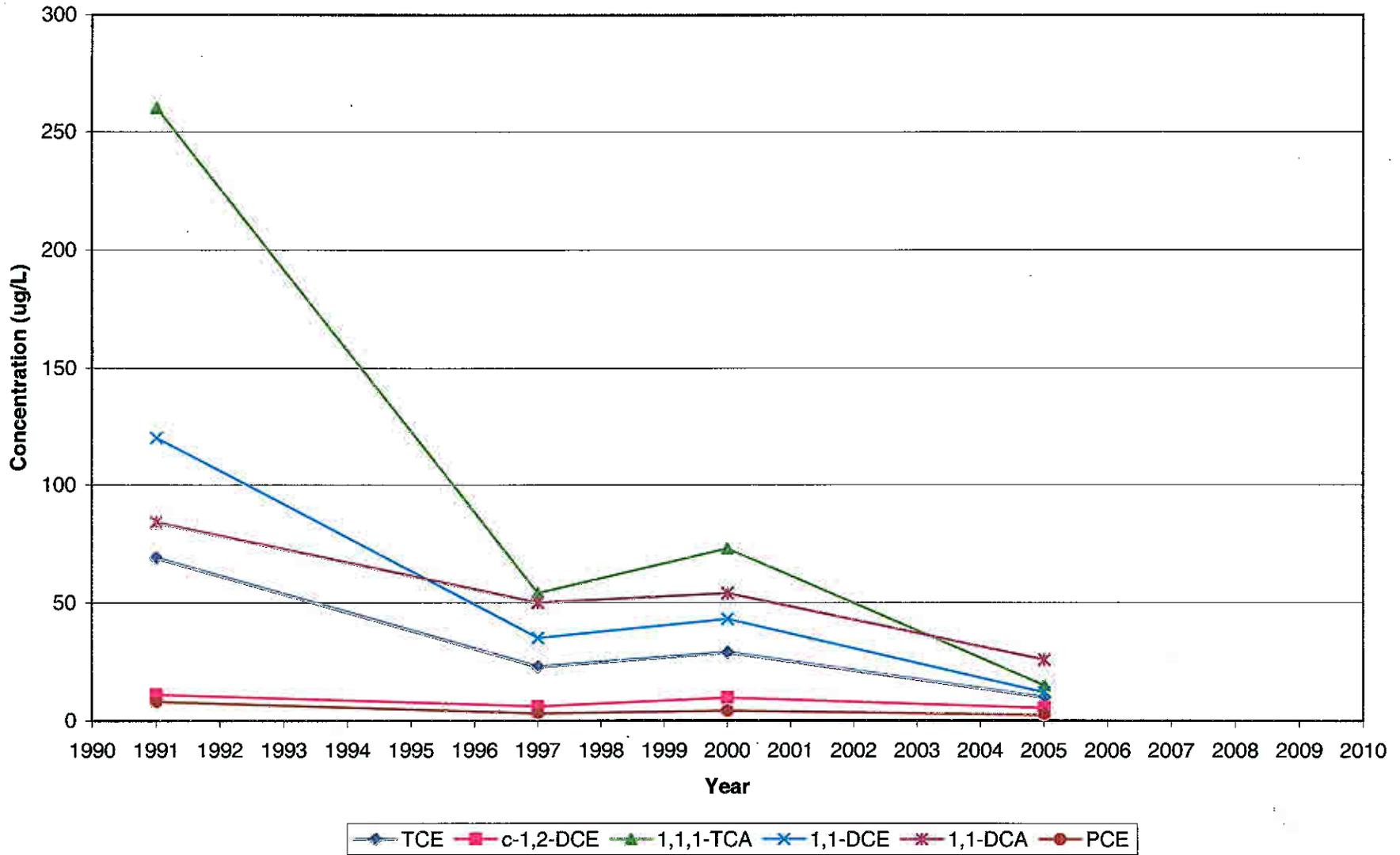
APPENDIX C

HISTORICAL CONCENTRATION TRENDS for SELECTED SITE WELLS

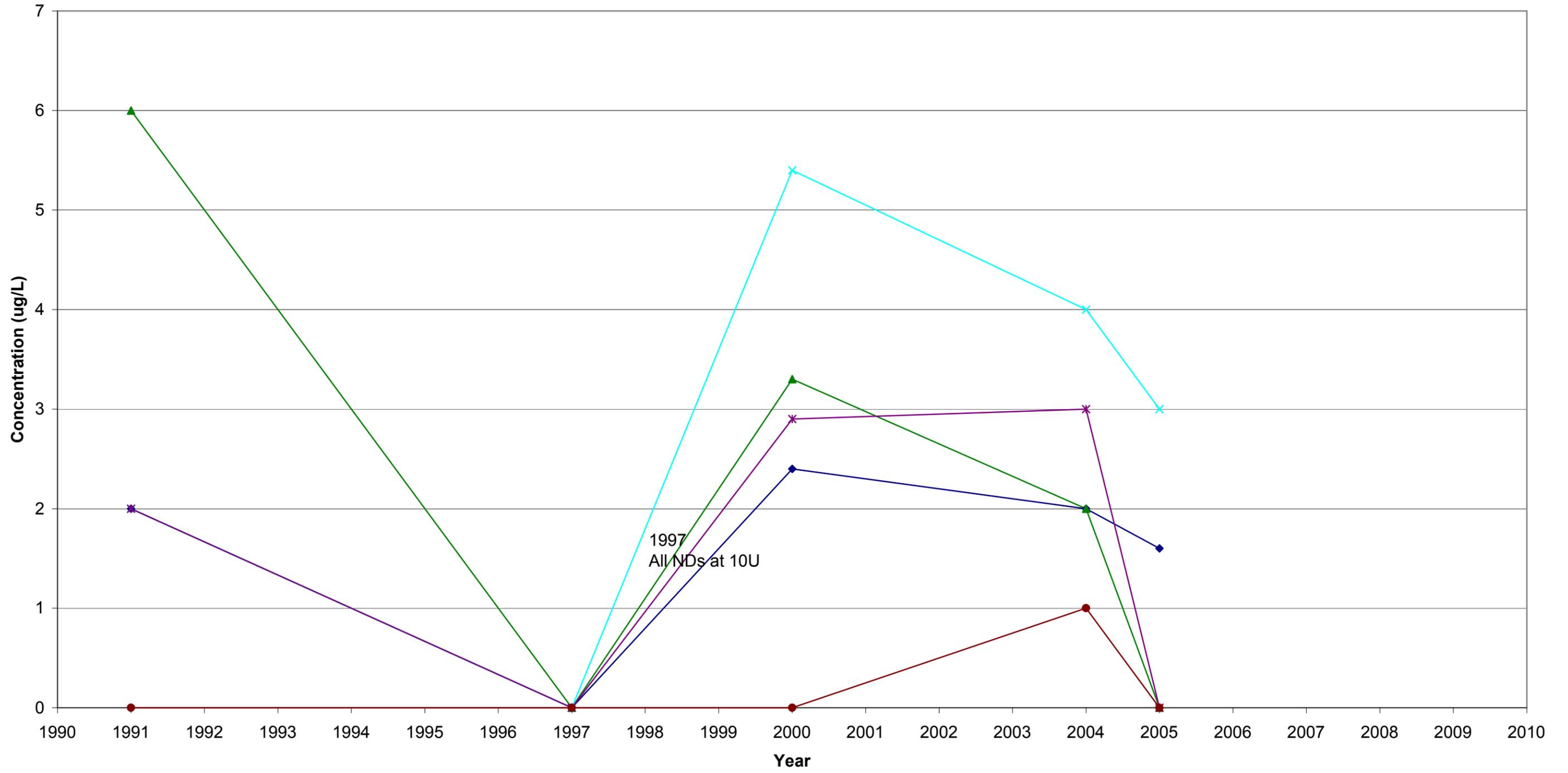
Monitoring Well 05MW01S



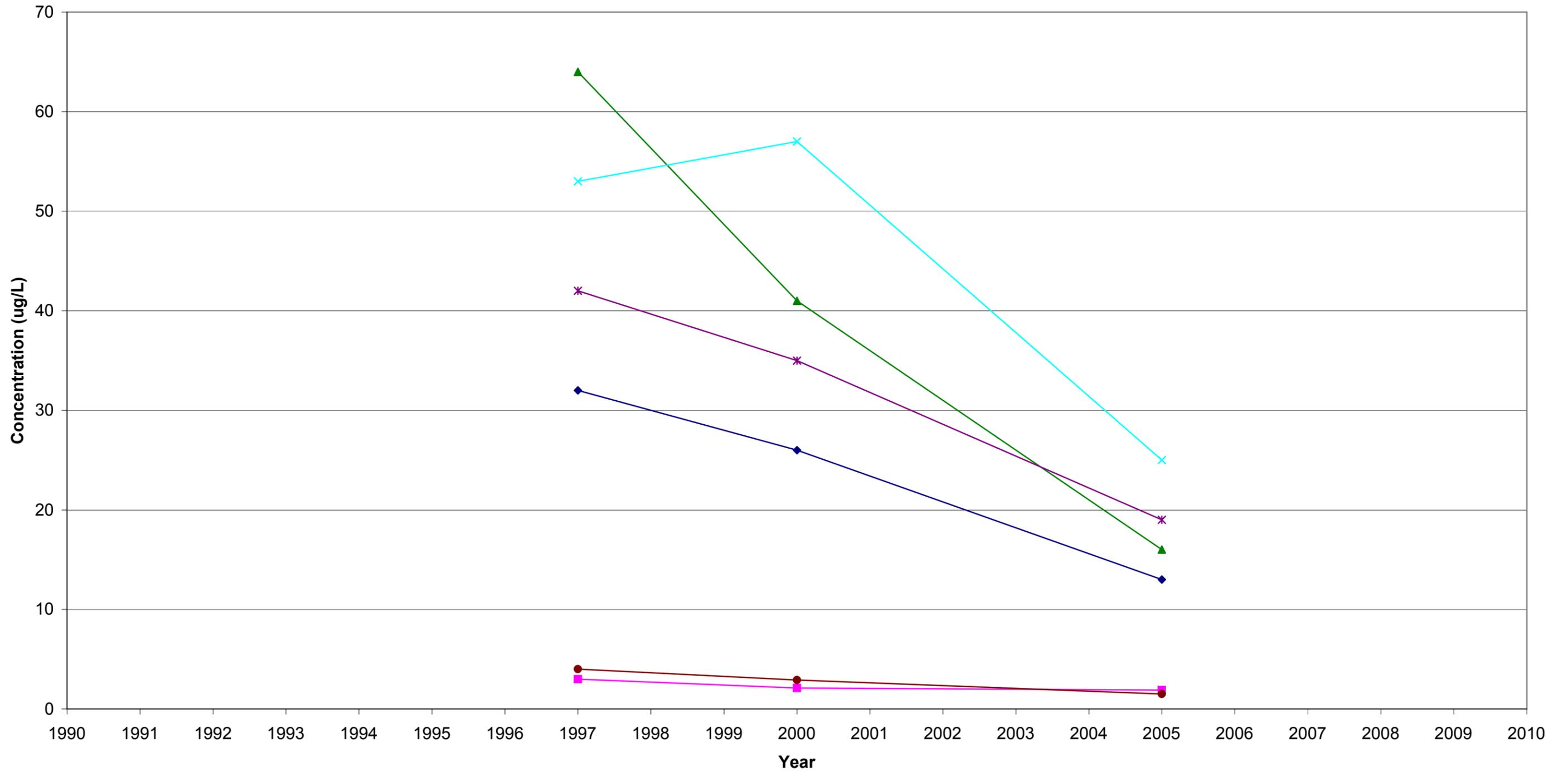
Monitoring Well 05MW01SI



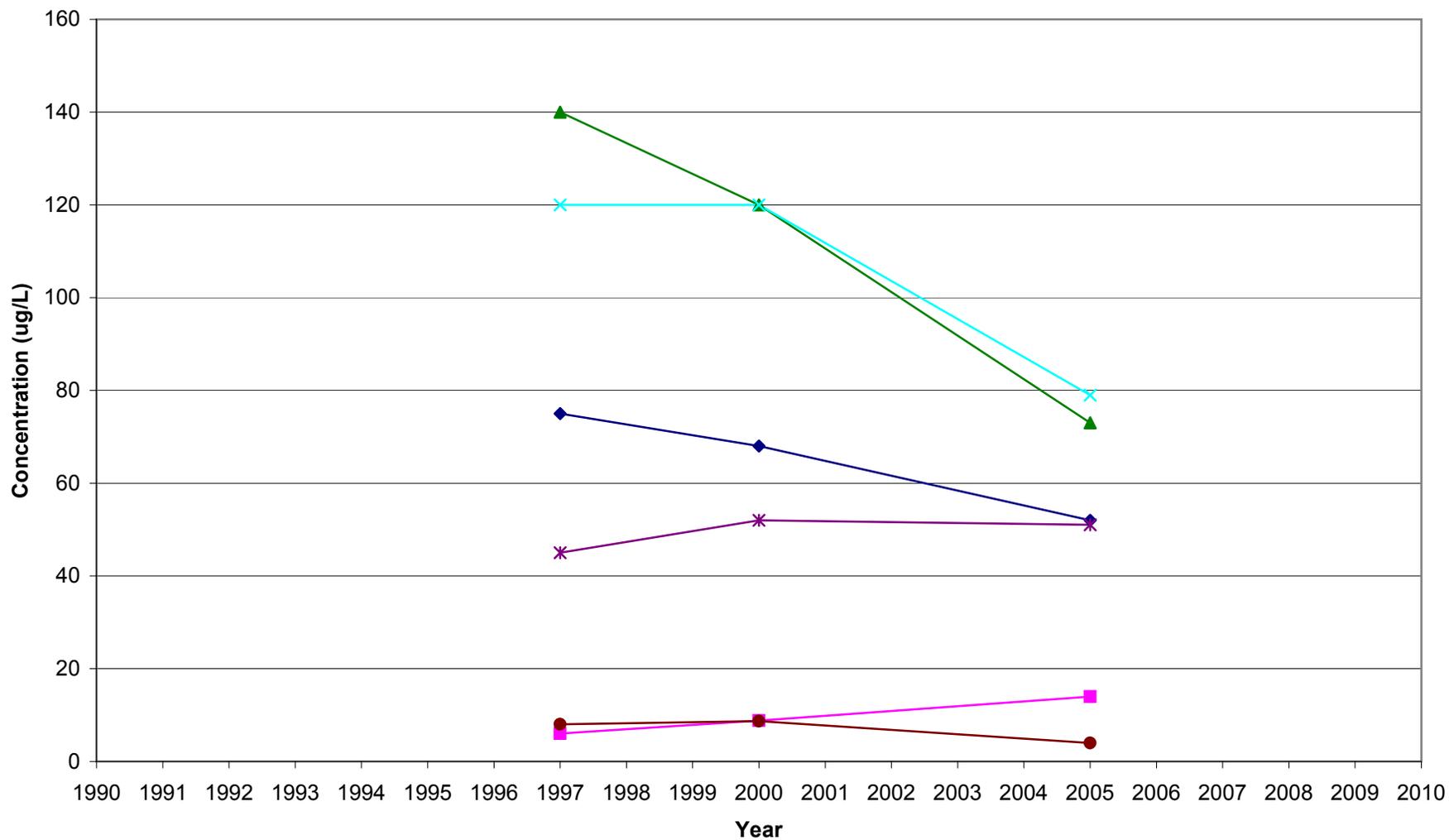
Monitoring Well 05MW07I



Monitoring Well 05MW09SI



Monitoring Well 05MW10SI



Project-Specific SAP
Site Name/Project Name: NAS JRB Willow Grove Site 5
Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study
Revision Number: 0
Revision Date: September 2008

APPENDIX D

MATERIAL SAFETY DATA SHEETS (MSDS)

[About the ILO](#) |
 [Departments and Offices](#) |
 [Regions](#) |
 [Themes](#) |
 [What we do](#)

International Occupational Safety and Health Information Centre (CIS)



[\[List of Chemicals\]](#) |
 [\[Risk Notes\]](#) |
 [\[Risk Phrases\]](#) |
 [\[Safety Phrases\]](#) |
 [\[Danger Symbols\]](#)



SODIUM BICARBONATE	ICSC: 1044
	April 2004
Carbonic acid monosodium salt Baking soda Bicarbonate of soda Sodium hydrogen carbonate Sodium acid carbonate	
CAS No: 144-55-8 RTECS No: VZ0950000	NaHCO_3 Molecular mass: 84.0

TYPES OF HAZARD / EXPOSURE	ACUTE HAZARDS / SYMPTOMS	PREVENTION	FIRE FIGHTING
FIRE	Not combustible.		In case of fire in the surroundings: use appropriate extinguishing media.
EXPLOSION			
EXPOSURE			
Inhalation			

Skin			
Eyes	Redness.	Safety spectacles.	First rinse with plenty of water for several minutes (remove contact lenses if easily possible), then take to a doctor.
Ingestion			

SPILLAGE DISPOSAL	PACKAGING & LABELLING
Sweep spilled substance into containers; if appropriate, moisten first to prevent dusting. Wash away remainder with plenty of water.	

EMERGENCY RESPONSE	STORAGE
	Separated from acids.

IMPORTANT DATA	
<p>Physical State; Appearance WHITE SOLID IN VARIOUS FORMS.</p> <p>Chemical dangers The solution in water is a weak base. Reacts with acids.</p> <p>Occupational exposure limits TLV not established. MAK not established.</p>	<p>Routes of exposure The substance can be absorbed into the body by ingestion.</p> <p>Inhalation risk Evaporation at 20°C is negligible; a nuisance-causing concentration of airborne particles can, however, be reached quickly when dispersed, especially, if powdered.</p> <p>Effects of short-term exposure The substance is mildly irritating to the eyes.</p>

PHYSICAL PROPERTIES	ENVIRONMENTAL DATA
<p>Melting point (decomposes): 50°C Density: 2.1 g/cm³ Solubility in water, g/100 ml at 20°C: 8.7</p>	

NOTES

IPCS
International
Programme on
Chemical Safety



Prepared in the context of cooperation between the International Programme on Chemical Safety and the European Commission
● IPCS 2004

LEGAL NOTICE

Neither the EC nor the IPCS nor any person acting on behalf of the EC or the IPCS is responsible for the use which might be made of this information.



Updated by AS. Approved by EC. Last update: 05.07.2004

For further information please contact the International Occupational Safety and Health Information Centre
at Tel: +41.22.799.6740, Fax: +41.22.799.8516 or E-mail: ois@ilo.org

[[SafeWork Home](#) | [Protection Home](#)]

International Labour Organization (ILO): [Contact us](#) | [Site map](#) |

Copyright and Permissions 1996-2007 International Labour Organization (ILO) - Disclaimer

[About the ILO](#) |
 [Departments and Offices](#) |
 [Regions](#) |
 [Themes](#) |
 [What we do](#)

International Occupational Safety and Health Information Centre (CIS)



[\[List of Chemicals\]](#) |
 [\[Risk Notes\]](#) |
 [\[Risk Phrases\]](#) |
 [\[Safety Phrases\]](#) |
 [\[Danger Symbols\]](#)



SODIUM CARBONATE (ANHYDROUS)	ICSC: 1135
	October 2004
Carbonic acid disodium salt Soda ash	
CAS No: 497-19-8 RTECS No: VZ4050000 EC No: 011-005-00-2	Na_2CO_3 Molecular mass: 106.0

TYPES OF HAZARD / EXPOSURE	ACUTE HAZARDS / SYMPTOMS	PREVENTION	FIRST AID / FIRE FIGHTING
FIRE	Not combustible.		In case of fire in the surroundings: use appropriate extinguishing media.
EXPLOSION			
EXPOSURE		PREVENT DISPERSION OF DUST!	
Inhalation	Cough. Sore throat.	Local exhaust or breathing protection.	Fresh air, rest.
Skin	Redness.	Protective gloves.	Rinse skin with plenty of water or shower.
	Redness. Pain.	Safety goggles.	First rinse with plenty of water for several

Eyes			minutes (remove contact lenses if easily possible), then take to a doctor.
Ingestion	Burning sensation in the throat and chest. Abdominal pain.	Do not eat, drink, or smoke during work.	Rinse mouth. Give plenty of water to drink. Refer for medical attention.

SPILLAGE DISPOSAL	PACKAGING & LABELLING
Sweep spilled substance into sealable containers; if appropriate, moisten first to prevent dusting. Personal protection: P2 filter respirator for harmful particles.	Xi Symbol R: 36 S: (2-)22-26

EMERGENCY RESPONSE	SAFE STORAGE
	Dry. Well closed. Separated from incompatible materials. See Chemical Dangers.

IMPORTANT DATA	
<p>Physical State; Appearance WHITE HYGROSCOPIC POWDER</p> <p>Chemical dangers The solution in water is a medium strong base. Reacts violently with acids. Reacts with magnesium, phosphorous pentoxide causing explosion hazard. Reacts with fluorine causing fire hazard.</p> <p>Occupational exposure limits TLV not established. MAK not established.</p>	<p>Inhalation risk A harmful concentration of airborne particles can be reached quickly especially if powdered.</p> <p>Effects of short-term exposure The substance is irritating to the eyes, the skin and the respiratory tract.</p> <p>Effects of long-term or repeated exposure The substance may have effects on the respiratory tract, resulting in perforation of the nasal septum. Repeated or prolonged contact with skin may cause dermatitis.</p>

PHYSICAL PROPERTIES	ENVIRONMENTAL DATA
Melting point: 851°C Density: 2.5 g/cm³ Solubility in water, g/100 ml at 20°C: 30	

NOTES

IPCS
International
Programme on
Chemical Safety



Prepared in the context of cooperation between the International Programme on Chemical Safety and the European Commission
© IPCS 2004

LEGAL NOTICE

Neither the EC nor the IPCS nor any person acting on behalf of the EC or the IPCS is responsible for the use which might be made of this information.



Updated by (ModOper). Approved by (ModAppr). Last update: (ModDate)

For further information please contact the International Occupational Safety and Health Information Centre
at Tel: +41.22.799.6740, Fax: +41.22.799.8516 or E-mail: ois@ilo.org

[[SafeWork Home](#) | [Protection Home](#)]

International Labour Organization (ILO): [Contact us](#) | [Site map](#) |

Copyright and Permissions 1996-2007 International Labour Organization (ILO) - [Disclaimer](#)

Project-Specific SAP
Site Name/Project Name: NAS JRB Willow Grove Site 5
Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study
Revision Number: 0
Revision Date: September 2008

APPENDIX E

CHEMICAL SOLUTION MAKEUP TABLE

WILLOW GROVE
SITE 5
PILOT STUDY
CHEMICAL SOLUTION MAKEUP TABLE

Sodium Lactate (with sodium lactate solution)
 200 g/L (Based on typical 60% stock solution, sg = 1.3)
 1.67 lb/gal

Gallons of water	gal of stock solution	L of stock solution	Final volume, gal
5	1.48	5.60	5.8
10	2.96	11.20	11.5
15	4.44	16.81	17.3
20	5.92	22.41	23.1
25	7.40	28.01	28.8
30	8.88	33.61	34.6
35	10.36	39.21	40.4
40	11.84	44.82	46.2
45	13.32	50.42	51.9
50	14.80	56.02	57.7

Sodium Carbonate / Bicarbonate
 62.4 g/L
 0.52 lb/gal

Gallons of water	lb
5	2.6
10	5.2
15	7.8
20	10.4
25	13.0
30	15.6
35	18.2
40	20.8
45	23.4
50	26.0
55	28.6
60	31.2
65	33.8
70	36.4
75	39.0
80	41.6
85	44.2
90	46.8
95	49.4
100	52.0
105	54.6
110	57.2
115	59.8
120	62.4
125	65.0
130	67.6
135	70.2
140	72.8
145	75.4
150	78.0
155	80.6
160	83.2
165	85.8
170	88.4
175	91.0
180	93.6
185	96.2
190	98.8
195	101.4
200	104.0

Project-Specific SAP
Site Name/Project Name: NAS JRB Willow Grove Site 5
Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study
Revision Number: 0
Revision Date: September 2008

APPENDIX F

ESTIMATED PROCESS EQUIPMENT LIST

**ESTIMATED
PROCESS EQUIPMENT LIST
SITE 5
WILLOW GROVE**

1.0 TANKS

ITEM NUMBER	NUMBER REQUIRED	NAME/DESCRIPTION
T-1	1	<u>Electron Donor Feed Tank</u> <ul style="list-style-type: none">• Configuration: Vertical, cylindrical, open-top with lid• Features: top-mounted mixer• Materials of fabrication: HDPE• Dimensions: 20 inch diameter x 40 inch SSH – 40 gal• Vendor: LMI (provided by feed pump vendor)
T-2	1	<u>Sodium Bicarbonate Feed Tank</u> <ul style="list-style-type: none">• Configuration: Vertical, cylindrical, open-top with lid• Features: angle-mounted mixer, tank stand, mixer mount• Materials of fabrication: HDPE• Dimensions: 32 inch diameter x 48 inch SSH – 150 gal

IMPORTANT NOTE: ALL EQUIPMENT SIZES INDICATED IN THE ABOVE LIST ARE PRELIMINARY IN NATURE AND SUBJECT TO CHANGE

ESTIMATED
PROCESS EQUIPMENT LIST
SITE 5
WILLOW GROVE

2.0 MIXERS

ITEM NUMBER	NUMBER REQUIRED	NAME/DESCRIPTION
M-1	1	<u>Electron Donor Feed Tank Mixer</u> <ul style="list-style-type: none">• Type: high-speed, propeller type• Configuration: top-mounted• Motor: 1/20 HP, 120 AC plug• Vendor: LMI (provided by feed pump vendor)
M-2	1	<u>Sodium Bicarbonate Feed Tank Mixer</u> <ul style="list-style-type: none">• Type: high-speed, propeller type• Configuration: angle-mounted, mount and stand are required.• Motor: 1/3 HP, field wire• Vendor: USABluebook• Model: Neptune Economy Batch Mixer, 1/3 HP

IMPORTANT NOTE: ALL EQUIPMENT SIZES INDICATED IN THE ABOVE LIST ARE PRELIMINARY IN NATURE AND SUBJECT TO CHANGE

**ESTIMATED
PROCESS EQUIPMENT LIST
SITE 5
WILLOW GROVE**

3.0 STATIC MIXER

ITEM NUMBER	NUMBER REQUIRED	NAME/DESCRIPTION
SM-1	1	<u>Static Mixer</u> <ul style="list-style-type: none">• Type: Inline, static, 1-inch diameter• Configuration: threaded ends• Materials of fabrication: PVC• Vendor: Koflo• Model: 328 – 6 element

IMPORTANT NOTE: ALL EQUIPMENT SIZES INDICATED IN THE ABOVE LIST ARE PRELIMINARY IN NATURE AND SUBJECT TO CHANGE

**ESTIMATED
 PROCESS EQUIPMENT LIST
 SITE 5
 WILLOW GROVE**

4.0 PUMPS

ITEM NUMBER	NUMBER REQUIRED	NAME/DESCRIPTION
P-1	1	<p><u>Extraction Well Pump (two)</u></p> <ul style="list-style-type: none"> • Type: Submersible • Rating: 3 gpm @ 115 ft TDH • Motor: 1/3 HP, 200-240/1/60, field wire • Vendor: Grundfos • Model: 5SQ 3A-90, 1" NPT
P-2	1	<p><u>Electron Donor Feed Pump</u></p> <ul style="list-style-type: none"> • Type: chemical metering pump • Rating: 0.42 GPH max; 110 psi; 1:100 turndown • Vendor: LMI • Model: LMI P031-392B1, 0.375" OD tubing, 120 AC plug end • Include combination valve for anti-siphon, pressure relief, back pressure, and drain
P-3	1	<p><u>Sodium Bicarbonate Feed Pump</u></p> <ul style="list-style-type: none"> • Type: chemical metering pump • Rating: 4 GPH max; 100 psi; 1:100 turndown • Vendor: LMI • Model: LMI C121-363B1, 0.375" OD tubing; 120 AC plug end • Include combination valve for anti-siphon, pressure relief, back pressure, and drain

IMPORTANT NOTE: ALL EQUIPMENT SIZES INDICATED IN THE ABOVE LIST ARE PRELIMINARY IN NATURE AND SUBJECT TO CHANGE

**ESTIMATED
PROCESS EQUIPMENT LIST
SITE 5
WILLOW GROVE**

5.0 CONTROL PANELS

ITEM NUMBER	NUMBER REQUIRED	NAME/DESCRIPTION
CP-1	1	<u>System Control Panel</u> NEMA 4 enclosure with face-mounted instruments and controls. Features: <ul style="list-style-type: none">• Extraction Well Pump HS (HOA) and run indication• Injection Well IW-1 High Level Alarm Light• Injection Well IW-2 High Level Alarm Light

IMPORTANT NOTE: ALL EQUIPMENT SIZES INDICATED IN THE ABOVE LIST ARE PRELIMINARY IN NATURE AND SUBJECT TO CHANGE

**ESTIMATED
PROCESS EQUIPMENT LIST
SITE 5
WILLOW GROVE**

6.0 INSTRUMENTATION

ITEM NUMBER	NUMBER REQUIRED	NAME/DESCRIPTION
FI-1	1	<p><u>Extraction Flow Indicator</u></p> <ul style="list-style-type: none"> • Discmeter with totalizer and flow rate indicators • Vendor: Hays
LSH-1, LAH-1	1	<p><u>IW-1 High Level Alarm</u></p> <ul style="list-style-type: none"> • One (1) high level float switch (LSH-1) • One (1) panel-mounted high level alarm (LAH-1) • Vendor: • Model: Vertical Float
LSH-2, LAH-2	1	<p><u>IW-2 High Level Alarm</u></p> <ul style="list-style-type: none"> • One (1) high level float switch (LSH-2) • One (1) panel-mounted high level alarm (LAH-2) • Vendor: • Model:
PI -1	1	<p><u>Extraction Well Flow Pressure Gauge</u></p> <ul style="list-style-type: none"> • Extraction well (2) pressure gauge (0 to 30 psi) (PI-1) • Model: Bourdon Tube, stainless Steel
PI -2	1	<p><u>Control Valve Pressure Gauge</u></p> <ul style="list-style-type: none"> • One (1) pressure gauge (0 to 60 psi) (PI-2) • Model: Bourdon Tube, stainless Steel
PI -3	1	<p><u>Injection Well Flow Pressure Gauge</u></p> <ul style="list-style-type: none"> • One (1) pressure gauge (0 to 60 psi) (PI-3) • Model: Bourdon Tube, stainless Steel

END OF EQUIPMENT LIST

IMPORTANT NOTE: ALL EQUIPMENT SIZES INDICATED IN THE ABOVE LIST ARE PRELIMINARY IN NATURE AND SUBJECT TO CHANGE

Project-Specific SAP
Site Name/Project Name: NAS JRB Willow Grove Site 5
Site Location: Willow Grove, Pennsylvania

Title: SAP for Site 5 Bioremediation Pilot Study
Revision Number: 0
Revision Date: September 2008

APPENDIX G

DAILY LOG SHEET

DAILY LOG SHEET

Date _____ Weather _____
 Personnel _____

Well	EW*	IW							
DTW, feet									
DO, mg/L									
pH									
ORP, mV									
Spec. Cond., mS/cm									
YSI stabilization time, minutes									
* Recorded from in-line YSI meter.									

In-Line YSI Meter (from extraction well)	Temp., °C	Spec. Cond., mS/cm	DO, %

Extraction Well flow rate		
Rate, gpm		
Totalizer		
Time of day		

Run meter	
Reading	
Time of day	

Pressure Gauges	PI-1 (filter influent)	P1-2 (post-filter)	P1-3 (effluent)
Reading, psig			

Chemical Feed	Tank level, feet		Tank level, gallons		Pump stroke setting, %		Pump Speed, %		Pump rate, GPH	
	start of day	end of day	start of day	end of day	start of day	end of day	start of day	end of day	start of day	end of day
Lactate					 	 				
Bi/carbonate					 	 				
Normal settings: Lactate pump = 0.2 GPH; Bi/carbonate pump = 1.46 GPH										
Maximum pumps output capacities: Lactate pump = 0.4 GPH; Bicarbonate pump = 4.0 GPH										

Chemical tanks refilled?:	Lactate	gal solution add:	gal water added:
	Bi/carbonate	lb added:	gal water added:

Chemical Feed Off:	Time Stopped:	Time Restarted:
Filter check (Y/N):		

Samples Collected	

Notes: _____

