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

for

SUPPLEMENTAL REMEDIAL INVESTIGATION OF 1,4-DIOXANE IN THE EASTERN PLUME AND BEDROCK

Naval Air Station Brunswick
Brunswick, Maine

Naval Facilities Engineering Command Mid-Atlantic

Contract Number N62472-03-D-0057
Contract Task Order 069

October 2008

I have reviewed the geological sections of the attached document entitled *Sampling and Analysis Plan (Field Sampling Plan and Quality Assurance Project Plan) for Supplemental Remedial Investigation of 1,4-Dioxane in the Eastern Plume and Bedrock, Naval Air Station Brunswick, Brunswick, Maine*, prepared by Tetra Tech NUS, Inc. (TtNUS). TtNUS Quality Assurance Project Plan specialists developed this plan.

STATE OF MAINE
CHARLES O. RACE
243
CERTIFIED GEOLOGIST

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

SAMPLING AND ANALYSIS PLAN (Field Sampling Plan and Quality Assurance Project Plan) FOR SUPPLEMENTAL REMEDIAL INVESTIGATION OF 1,4-DIOXANE IN THE EASTERN PLUME AND BEDROCK NAVAL AIR STATION BRUNSWICK, MAINE

OCTOBER 2008

Prepared for:
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Prepared under:
CONTRACT NUMBER N62472-03-D-0057
CONTRACT TASK ORDER 069

Review Signatures:

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Approval Signatures:

Todd Bober 10/7/08	BOWERS. KENNETH. A.1230092474 Digitally signed by BOWERS.KENNETH.A.1230092474 DN: cn=BOWERS.KENNETH.A.1230092474, c=US, o=U.S. Government, ou=DoD, PKI, USN Reason: I have reviewed this document Date: 2008.10.10 14:13:04 -04'00'
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Michael Daly
Environmental Protection Agency, Region 1
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EXECUTIVE SUMMARY

This Uniform Federal Policy Sampling and Analysis Plan (UFP-SAP) encompasses Field Sampling Plan (FSP) requirements and Quality Assurance Project Plan (QAPP) requirements for the supplemental Remedial Investigation (RI) of 1,4-dioxane and other volatile organic compounds (VOCs) in the Eastern Plume and bedrock at Naval Air Station (NAS) Brunswick, Maine. This document constitutes the planning document, addressing specific protocols for sample collection, sample handling and storage, chain of custody, laboratory and field analyses, data validation, and data reporting.

This UFP-SAP has been prepared by Tetra Tech NUS, Inc. (TtNUS) on behalf of the Base Realignment and Closure (BRAC) Program Management Office Northeast (PMO NE) under the Comprehensive Long-Term Environmental Action Navy (CLEAN) Contract Number N62472-03-D-0057, Contract Task Order (CTO) 069. This SAP was generated for and complies with applicable United States Navy (Navy), State of Maine, and United States Environmental Protection Agency (EPA) Region 1 requirements, regulations, guidance, and technical standards, especially those concerning the UFP set forth by the EPA and Department of Defense/Department of Energy (DoD/DOE)/EPA (March, 2005). To comply with UFP requirements, the SAP is presented in the format of standard worksheets specified in the UFP-QAPP Manual.

The Eastern Plume groundwater contamination has been attributed to leaching from past solvent disposal practices at Site 4 (Acid/Caustic Pit), Site 11 (Former Fire Training Area), and Site 13 (Defense Reutilization and Marketing Office area). The dissolved-phase plume associated with these disposal activities consisted primarily of chlorinated VOCs including tetrachloroethene, 1,1,1-trichloroethane, and trichloroethene. A groundwater extraction and treatment system is in place to remediate both the northern and southern lobes of the plume and to provide hydraulic control (via extraction) of the plume and remove dissolved-phase VOCs from groundwater. Since 2004, 1,4-dioxane has been added as a chemical of concern and selected monitoring wells in the Eastern Plume are sampled for this compound. 1,4-Dioxane is present in the Eastern Plume at groundwater concentrations greater than the State of Maine Maximum Exposure Guideline (MEG) of 32 ug/L for drinking water. The purpose of the SAP is to refine delineation of the extent of 1,4-dioxane contamination exceeding the State criterion in support of future remediation efforts. Secondarily, data will be collected for other known volatile organic compounds (VOCs) of concern to refine understanding of the Eastern Plume. The investigation will include both the overburden and bedrock to better understand geology/hydrogeology and migration potential.

The basis for the scope of work was a Data Quality Objectives (DQO) meeting held in February 2008. The ensuing scope of field work described herein will be conducted in a phased sequence, as follows, with the EPA and Maine Department of Environmental Protection (MEDEP) involved in the decision-making process:

- Pore water sampling of 1,4-dioxane and other chlorinated VOCs along Merriconeag Stream to aid in refining transects locations for groundwater profiling. Note this task is being conducted by MEDEP (August 2008), with requirements addressed by MEDEP under separate cover from the subject SAP, and supported by EPA analysis of the samples.

- Groundwater profiling of the overburden (temporary wells) and a concurrent bedrock investigation (permanent monitoring wells) to better delineate the Eastern Plume, refine geology/hydrogeology, and aid in determining contaminant migration potential. Slug tests and water-level measurements from the new wells will aid in the hydrogeology investigation.

- Installation and sampling of permanent overburden monitoring wells and comprehensive evaluation including most recent data from existing Eastern Plume wells that are part of the long-term monitoring program. Also, select existing monitoring wells will be included that are not part of the long-term monitoring program but will aid in the evaluation process. Slug tests and water-level measurements from the new wells will aid in the hydrogeology evaluation. Water-level measurements from new and existing wells throughout the Eastern Plume will be collected to aid in evaluation potentiometric flow.

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- **Figure 17-1, Revision 2**
- **Table**

Appendix A – Attachment 2
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- **Figure 17-2A, Revision 0**

Appendix B Conceptual Site Model

Appendix C Supporting Information
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C-2 Historical Geology/Hydrogeology
C-3 Historical 1,4-Dioxane Area Nature and Extent of Contamination
C-4 February 2008 DQO Meeting Day 2 DQOs

Appendix D Field Standard Operating Procedures

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Appendix F Analytical Laboratory Standard Operating Procedures
(provided on compact disk)

Acronyms

AA	Atomic Absorption
ANSI/ASQ	American National Standards Institute/American Society for Quality
ASTM	American Society for Standards and Materials
BASCE	Brunswick Citizens for a Safe Environment
bgs	Below ground surface
BOD	Biological Oxygen Demand
BRAC	Base Realignment and Closure
CA	Corrective Action
CCC	Continuing calibration compound
CCV	Continuing calibration verification
CDC	Center for Disease Control
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CFR	Code of Federal Regulations
CG	Certified Geologist
CLEAN	Comprehensive Long-Term Environmental Action
CLP	Contract Laboratory Program
COC	Contaminant of Concern or Chain-of-Custody form
CRDL	Contract-Required Detection Limit
CSM	Conceptual Site Model
CTO	Contract Task Order
CWA	Clean Water Act
DoD	Department of Defense
DOE	Department of Energy
DCE	Dichloroethene
DCN	Document Control Number
DNAPL	Dense non-aqueous phase liquid
DPT	Direct-Push Technology
DQI	Data Quality Indicator
DQO	Data Quality Objective
DVM	Data Validation Manager
EC	Electrical conductivity
EDD	Electronic data deliverable
EGIS	Environmental Geographic Information System
ENU	Elemental Neutralization Unit
EPA	Environmental Protection Agency
ESD	Explanation of Significant Differences
FOL	Field Operations Leader
FS	Feasibility Study
FSP	Field Sampling Plan
FTMR	Field Task Modification Request
GAC	Granular activated carbon
GC	Gas Chromatograph
GC/MS	Gas Chromatograph/Mass Spectrometer
GIS	Geographic Information System
GPC	Gel Permeation Chromatography

gpm	gallon per minute
GPS	Global Positioning System
GW	Ground Water
GWETS	Groundwater extraction and treatment system
HASP	Health and Safety Plan
HQ	Headquarters
HSM	Health and Safety Manager
ICOC	Internal Chain-of-Custody
ICP	Inductively Coupled Plasma
ID	Inside diameter
IDW	Investigation-derived waste
IDQTF	Intergovernmental Data Quality Task Force
IR	Installation Restoration
IRIS	Integrated Risk Information System
IS	Internal Standard
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LFB	Laboratory Fortified Blank
LIMS	Laboratory Information Management Systems
LTMP	Long-Term Monitoring Plan
MCL	Maximum Contaminant Level
MDL	Method Detection Limit
MEDEP	Maine Department of Environmental Protection
MEG	Maximum Exposure Guideline (State of Maine)
MNA	Monitored natural attenuation
MPC	Measurement Performance Criteria
MQO	Measurement Quality Objectives
MS/MSD	Matrix spike/Matrix spike duplicate
msl	Mean Sea Level
MSR	Management Systems Review
NA	Not Available
NAS	Naval Air Station
NAVFAC	Naval Facilities
NCP	National Contingency Plan
NEIC	National Enforcement Investigations Center
NEL	Northeast Laboratory
NFA	No Further Action
NFESC	Naval Facilities Engineering Service Center
NIRIS	Naval Installation Restoration Information Solutions
NIST	National Institute of Standards and Technology
NPL	National Priorities List
NSF	National Sanitation Foundation
OSHA	Occupational Safety and Health Administration
OU	Operable Unit
PARCC	Precision, Accuracy, Representativeness, Completeness, and Comparability
PCE	Tetrachloroethene
PDF	Portable document format
PG	Professional Geologist
PM	Project Manager

PMO NE	Program Management Office Northeast
POTW	Public-owned treatment works
PPE	Personal protective equipment
PQO	Project Quality Objective
PRG	Preliminary Remediation Goal
PRQL	Project-Required Quantitation Limit
PT	Proficiency Testing (previously known as performance evaluation (PE) sample)
PVC	Polyvinyl Chloride
QA	Quality assurance
QAM	Quality Assurance Manager
QAO	Quality Assurance Officer
QAPP	Quality Assurance Project Plan
QC	Quality control
QL	Quantitation limit
QS	Quality System
QSM	Quality Systems Manual
RCRA	Resource Conservation and Recovery Act
RA	Remedial Action
RD	Remedial Design
REG	Regulatory
RfC	Reference Concentration
RfD	Reference Dose
RI	Remedial Investigation
RIC	Reconstructed Ion Chromatogram
ROD	Record of Decision
RPD	Relative percent difference
RPM	Remedial Project Manager
RSD	Relative standard deviation
RSL	Regional Screening Level
RT	Retention Time
RTW	Retention Time Window
SAP	Sampling and Analysis Plan
SD	Standard Deviation
SDG	Sample delivery group
SDWA	Safe Drinking Water Act
SIM	Selective Ion Monitoring
SOP	Standard Operating Procedure
SPCC	System performance check compounds
SQLs	Sample Quantitation Limits
SRM	Standard Reference Material
SVOA	Semivolatile Organic Analytes
SVOC	Semivolatile Organic Compound
SW	Surface Water
TCA	Trichloroethane
TCE	Trichloroethene
TCL	Target Compound List
TCLP	Toxicity Characteristic Leaching Procedure
TBD	To Be Determined
TOC	Total organic compound

TSA	Technical Systems Audit
TtNUS	Tetra Tech NUS, Inc.
UFP	Uniform Federal Policy
UV	Ultraviolet
VOA	Volatile Organic Analytes
VOC	Volatile organic compound

SAP Worksheet #2 -- SAP Identifying Information

(UFP-SAP Manual Section 2.2.4)

Site Name/Number: **Eastern Plume, Naval Air Station (NAS) Brunswick**
Operable Unit: **OU 5**
Contractor Name: **Tetra Tech NUS, Inc. (TtNUS or Tetra Tech)**
Contract Number: **N62472-03-D-0057**
Contract Title: **NAVFAC Mid-Atlantic CLEAN**
Work Assignment Number: **CTO 069**

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

2. Identify regulatory program: National Contingency Plan (NCP); Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA)

3. This SAP is a project-specific SAP.

4. List dates of scoping sessions that were held:

Scoping Session	Date
• Alternative Means (phone calls, e-mails, and correspondence): Scoping was first based on review of data gaps from previous investigations and consensus agreements between the Navy and Stakeholders documented in correspondence.	August-October 2008
• DQO Meeting – 1,4-Dioxane and Bedrock	February 26-28, 2008

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

Title	Date
Revised Proposed 1,4-Dioxane Sampling Plan – April 2007 (ECC)	March 27, 2007
Draft SAP for Supplemental Investigation of 1,4-Dioxane in the Eastern Plume	October 2007 (superceded by subject SAP)
Final Long-Term Monitoring Plan Eastern Plume (ECC)	February, 2008

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

MEDEP (regulatory stakeholder)
EPA Region 1 (regulatory stakeholder)
NAS Brunswick (property owner)
Brunswick Area Citizens for a Safe Environment (BACSE)
Midcoast Regional Redevelopment Authority (MRRA)

7. Lead organization

Navy, BRAC PMO NE

8. No SAP elements have been omitted.

UFP-SAP Worksheet #	Required Information	Crosswalk to Related Information
A. Project Management		
Documentation		
1	Title and Approval Page	NA
2	Table of Contents SAP Identifying Information	NA
3	Distribution List	NA
4	Project Personnel Sign-Off Sheet	NA
Project Organization		
5	Project Organizational Chart	NA
6	Communication Pathways	NA
7	Personnel Responsibilities and Qualifications Table	NA
8	Special Personnel Training Requirements Table	NA
Project Planning/ Problem Definition		
9	Project Planning Session Documentation (including Data Needs Tables) Project Scoping Session Participants Sheet	NA
10	Problem Definition, Site History, and Background. Site Maps (historical and present)	NA
11	Site-Specific Project Quality Objectives	NA
12	Measurement Performance Criteria Table	NA
13	Sources of Secondary Data and Information Secondary Data Criteria and Limitations Table	NA
14	Summary of Project Tasks	NA
15	Reference Limits and Evaluation Table	NA
16	Project Schedule/Timeline Table	NA
B. Measurement Data Acquisition		
Sampling Tasks		
17	Sampling Design and Rationale	NA
18	Sampling Locations and Methods/SOP Requirements Table Sample Location Map(s)	NA
19	Analytical Methods/SOP Requirements Table	NA
20	Field Quality Control Sample Summary Table	NA
21	Project Sampling SOP References Table Sampling SOPs	NA

UFP-SAP Worksheet #	Required Information	Crosswalk to Related Information
22	Field Equipment Calibration, Maintenance, Testing, and Inspection Table	NA
Analytical Tasks		
23	Analytical SOPs Analytical SOP References Table	NA
24	Analytical Instrument Calibration Table	NA
25	Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table	NA
Sample Collection		
26	Sample Handling System, Documentation Collection, Tracking, Archiving and Disposal Sample Handling Flow Diagram	NA
27	Sample Custody Requirements, Procedures/SOPs Sample Container Identification Example Chain-of-Custody Form and Seal	NA
Quality Control Samples		
28	QC Samples Table Screening/Confirmatory Analysis Decision Tree	NA
Data Management Tasks		
29	Project Documents and Records Table	NA
30	Analytical Services Table Analytical and Data Management SOPs	NA
C. Assessment Oversight		
31	Planned Project Assessments Table Audit Checklists	NA
32	Assessment Findings and Corrective Action Responses Table	NA
33	QA Management Reports Table	NA
D. Data Review		
34	Verification (Step I) Process Table	NA
35	Validation (Steps IIa and IIb) Process Table	NA
36	Validation (Steps IIa and IIb) Summary Table	NA
37	Usability Assessment	NA

SAP Worksheet #3 -- Distribution List
(UFP-SAP 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)
Todd Bober	Remedial Project Manager (RPM)	Navy BRAC PMO NE 4911 South Broad Street Philadelphia, PA 19112	215-897-4911	todd.bober@navy.mil	Not applicable
Paul Burgio	BRAC Environmental Coordinator	Navy BRAC PMO NE 4911 South Broad Street Philadelphia, PA 19112	215-897-4915	paul.burgio@navy.mil	Not applicable
Carolyn LePage	Technical Advisor to BASCE	LePage Environmental Services 731 Hotel Road Auburn, ME 04210	207-777-1049	calepage@adelphia.net	Not applicable
Victoria Boundy	Planning and Environmental Manager (MMRA)	Midcoast Regional Redevelopment Authority 5450 Fitch Avenue Brunswick, ME 04011	207-798-6512	victoriab@mrra.us	Not applicable
(shared copy) Jennifer Wright and Amy Van Dercook Sherri Eng	Environmental Technical Support Biologist Environmental Scientist Chemist	NAVFAC Atlantic Attn: Code EV32JW 6506 Hampton Blvd Norfolk, VA 23508-1278 Code EV32JW (Jennifer) Code EV32AV (Amy) Code EV32SE	 757-322-8428 757-322-4764 757-322-4366	 Jennifer.H.Wright@navy.mil Amy.vandercook@navy.mil sherri.eng@navy.mil	Not applicable
David Barclift	Navy BRAC PMO NE Technical Support	Navy BRAC PMO NE 4911 South Broad Street Philadelphia, PA 19112	215-897-4913	david.barclift@navy.mil	Not applicable
Bonnie Capito (cover letter only with document storage via Glenn Wagner of TtNUS)	Librarian (Navy Admin Record)	NAVFAC Atlantic LRA Bldg. A 6506 Hampton Blvd Norfolk, VA 23508-1278	---	bonnie.capito@navy.mil	Not applicable
Michael Fagan	NAS Brunswick IR Coordinator	NAS Brunswick Environmental Office 437 Huey Drive, Bldg. 53 Brunswick, ME 04011-5008	207-921-1717	Michael.Fagan1@navy.mil	Not applicable

Name of SAP Recipients	Title/Role	Organization	Telephone Number (Optional)	E-Mail Address or Mailing Address	Document Control Number (Optional)
Lisa Joy	NAS Brunswick Environmental Director	NAS Brunswick Environmental Office 437 Huey Drive, Bldg. 53 Brunswick, ME 04011-5008	207-921-1720	lisa.joy@navy.mil	Not applicable
Michael Daly	Remedial Project Manager	EPA Region 1 Federal Facilities Superfund Section 1 Congress Street, Suite 1100 (HBT) Boston, MA 02114-2023	617-918-1386	Daly.Mike@epamail.epa.gov	Not applicable
Claudia Sait	Remedial Project Manager	Maine Department of Environmental Protection Bureau of Remediation & Waste Management State House, Station 17 Augusta, ME 04333-0017	207-287-7713	claudia.b.stait@maine.gov	Not applicable
Chris Evans	MEDEP Project Hydrogeologist	Maine Department of Environmental Protection Bureau of Remediation & Waste Management State House, Station 17 Augusta, ME 04333-0017	207-287-7656	Gordon.C.Evans@maine.gov	Not applicable
Peter Golonka	EPA Contractor	Gannett Fleming Suite 210 199 Wells Avenue Newton, MA 02459	---	pgolonka@gfnet.com	Not applicable
Dave McTigue	EPA Contractor	Gannett Fleming Suite 210 199 Wells Avenue Newton, MA 02459	---	dmctigue@gfnet.com	Not applicable
Helen Cavanaugh	Environmental Scientist	ECC 33 Boston Post Road West Suite 340 Marlborough, MA 01752	508-229-2270	HCavanaugh@ecc.net	Not applicable
Robert Phinney	Treatment Plant Operator	**FedEx**: NAS Brunswick Building 50 Brunswick, ME 04011 **Regular Mail:** PMB 289 13 Gurnet Road Brunswick, ME 04011	207-798-5977	rphinney@ecc.net	Not applicable

Name of SAP Recipients	Title/Role	Organization	Telephone Number (Optional)	E-Mail Address or Mailing Address	Document Control Number (Optional)
Pam Doughty	Total Quality Manager	Northeast Laboratory Services, Inc. P.O. Box 788 Waterville, ME 04903-0788	(207) 873-7711 ext. 310 (207) 873-7022	pdoughty@nelabservices.com	Not applicable
Linda Klink	TtNUS Project Manager (PM)	TtNUS NUS, Inc. 661 Andersen Drive, Foster Plaza 7 Pittsburgh, PA 15220	412-921-8650	linda.klink@tetratech.com	Not applicable
Chuck Race	TtNUS Maine Certified Geologist (C.G.)	TtNUS NUS, Inc. 55 Jonspin Road Wilmington, MA 01887-1020	(978) 474-8437	charles.race@tetratech.com	Not applicable
Tom Johnston (shared copy with TtNUS PM)	TtNUS Quality Assurance Manager (QAM)	TtNUS NUS, Inc. 661 Andersen Drive, Foster Plaza 7 Pittsburgh, PA 15220	412-921-8615	tom.johnston@tetratech.com	Not applicable
Kelly Carper	TtNUS Project Chemist	TtNUS NUS, Inc. 661 Andersen Drive, Foster Plaza 7 Pittsburgh, PA 15220	412-921-7273	kelly.carper@tetratech.com	Not applicable
Joseph Samchuck Lee Leck (shared copy)	TtNUS Data Validation Manager (DVM) TtNUS Data Manager (DM)	TtNUS NUS, Inc. 661 Andersen Drive, Foster Plaza 7 Pittsburgh, PA 15220	412-921-8510 412-921-8856	joseph.samchuck@tetratech.com lee.leck@tetratech.com	Not applicable
Matt Soltis [Health and Safety Plan (HASP) only]	TtNUS Health and Safety Manager (HSM)	TtNUS NUS, Inc. 661 Andersen Drive, Foster Plaza 7 Pittsburgh, PA 15220	412-921-8912	matt.soltis@tetratech.com	Not applicable
John Trepanowski	TtNUS Program Manager	TtNUS NUS, Inc. 234 Mall Boulevard, Suite 260 King of Prussia, PA 19406	610-491-9688	john.trepanowski@tetratech.com	Not applicable
TBD	TtNUS Field Operations Leader (FOL) and field support personnel	TtNUS NUS, Inc. 661 Andersen Drive, Foster Plaza 7 Pittsburgh, PA 15220	TBD	TBD	Not applicable
Janet Fullerton (Final only)	Head of Reference Desk (NASB Admin Record)	Curtis Memorial Library 23 Pleasant Street Brunswick, Maine 04011	---	---	Not applicable

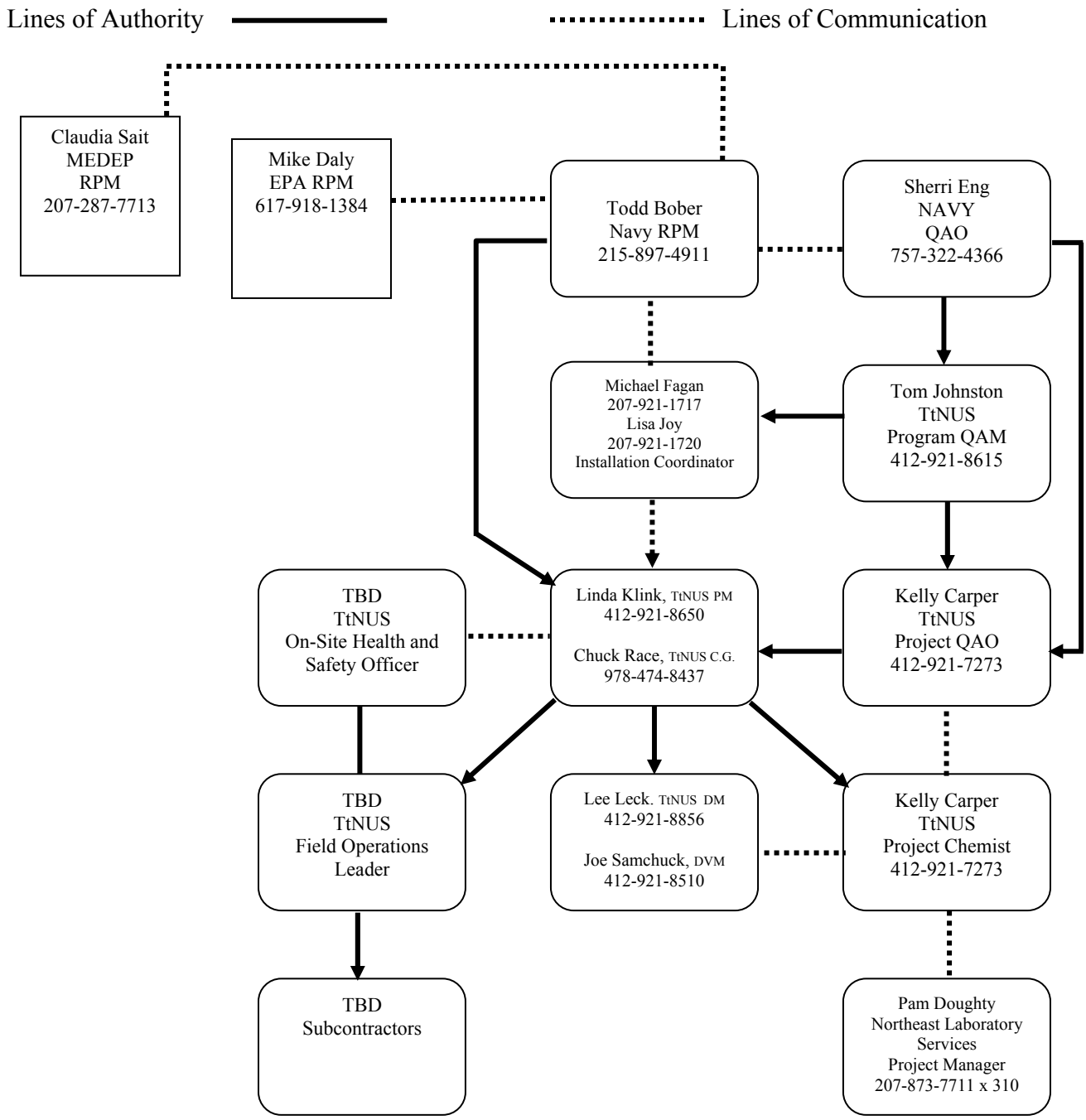
SAP Worksheet #4 -- Project Personnel Sign-Off Sheet

(UFP-SAP Manual Section 2.3.2)

Name	Organization/Title/Role	Telephone Number (optional)	Signature/E-mail receipt	SAP Section Reviewed	Date SAP Read
Linda Klink	TtNUS, Project Manager (PM)	412-921-8650	See Worksheet #1 Title and Approval Page	All	
Matt Soltis	TtNUS Health & Safety Manager (HSM)	412-921-8912	HASP signature page (under separate cover)	HASP (under separate cover)	
Charles Race	TtNUS Maine Certified Geologist	978-474-8437	See Worksheet #1 Title and Approval Page	All	
Tom Johnston	TtNUS Project Quality Assurance Manager (QAM)	412-921-8615	See Worksheet #1 Title and Approval Page	All	
Kelly Carper	TtNUS Project Chemist	412-921-7273		Worksheet #s 12, 15, 19, 23-28, and 30-36	
TBD	TtNUS FOL/Site Safety Officer	TBD		All except Worksheet #s 12, 15, 19, 23-28, and 30-36	
TBD	TtNUS field tech	TBD		Same as above	
TBD	TtNUS field tech	TBD		Same as above	
Joseph Samchuck	TtNUS DVM	412-921-8510		Worksheet #s 12, 15, 19, 28, and 35	
Lee Leck	TtNUS DM	412-921-8856		Worksheet #s 12, 15, 19, 28, and 35	
Pam Doughty	Northeast Laboratory Services, Inc. Total Quality Manager	(207) 873-7711 ext. 310		Worksheet #s 12, 15, 19, 23-28	

DM	Data Manager
DVM	Data Validation Manager
FOL	Field Operations Leader
HASP	Health and Safety Plan
HSM	Health and Safety Manager
PM	Project Manager
QAM	Quality Assurance Manager
SAP	Sampling and Analysis Plan
TBD	To be determined

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



SAP Worksheet #6 -- Communication Pathways

(UFP-SAP Manual Section 2.4.2)

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or E-Mail	Procedure (timing, pathway to & from, etc.)
QAPP amendments	Navy RPM	Todd Bober	215-897-4911	Send scope change to TtNUS Program Management Office
Changes in schedule	TtNUS PM	Linda Klink	412-921-8650	Inform 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 FOL TtNUS PM	TBD Linda Klink	TBD 412-921-8650	Immediately obtain approval from TtNUS PM; Document via Field Task Modification Request (FTMR) form FOL inform PM; PM inform RPM; RPM issue scope change if warranted; Scope change to be implemented before work is executed. RPM to inform regulatory agencies (EPA and MEDEP); Concurrence is required associated with any significant changes.
Recommendations to stop work and initiate work upon corrective action	TtNUS FOL TtNUS PM TtNUS QAM TtNUS Site Safety Officer Navy RPM	TBD Linda Klink Tom Johnston TBD Todd Bober	TBD 412-921-8650 412-921-8615 TBD 215-897-4911	Responsible party immediately informs subcontractors, the Navy, and Project Team
Analytical data quality issues	Analytical Laboratory TtNUS Project Chemist	Pam Doughty Kelly Carper	207-873-7711 x 310 412-921-7273	Immediately notify TtNUS Project Chemist; Notify data validation staff and TtNUS PM if necessary
Community relations/public communication	TtNUS PM TtNUS FOL	Linda Klink TBD	412-921-8650 TBD	PM to be notified immediately if representatives of local community contact TtNUS during project. Formal project public relations through NAVFAC Mid-Atlantic.
Deliverables and data management	TtNUS PM	Linda Klink	412-921-8650	A draft version of the SAP to be presented to stakeholders; Draft versions of this SAP and Report will be prepared, and upon addressing conmments, the documents will be finalized.

FOL Field Operations Leader
FTMR Field Task Modification Request
PM Project Manager (TtNUS)
QAM Quality Assurance Manager
RPM Remedial Project Manager (Navy)

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

The personnel from TtNUS NUS and the analytical laboratory responsible for implementing the SAP are identified in the following table. Resumes are available upon request.

Name	Title/Role	Organizational Affiliation	Responsibilities	Education and/or Experience Qualifications (Optional)
Linda Klink	PM	TtNUS NUS (Pittsburgh)	Oversees project, financial, schedule, and technical day to day management of the project.	M.S. Environmental Engineering, B.S. Chemical Engineering, Professional Engineer, PA; 26 years professional engineering experience
Chuck Race	Maine Certified Geologist (C.G.)	TtNUS NUS (Boston)	Oversees geology/hydrogeology-related tasks to ensure compliance with State of Maine requirements	M.S. Geology, B.A. Geological Sciences, Maine Certified Geologist, N.H. Certified Geologist, Mass. Site Licensed Professional, 24 years geology experience
TBD	FOL	TtNUS NUS	Supervise, coordinate, and perform field sampling activities prepare all geological interpretation and text.	Information can be provided at a later date
Kelly Carper	Project Chemist and QAM	TtNUS NUS	Participate in scoping, prepare lab scope, coordinate with lab, and data quality review. Ensure Quality aspects of the CLEAN program.	B.S. Biology, 15 years environmental experience
Joseph Samchuck	DVM	TtNUS NUS	Quality assurance of data validation deliverables.	B.S. Chemistry, MBA, M.S. Finance, 24 years environmental experience
Pam Doughty	Total Quality Manager	Northeast Laboratory Service	Coordinate analyses with lab chemists, ensure the scope is followed, QA data packages, communicate with TtNUS staff.	Can be provided upon request
TBD	Site Safety Officer	TtNUS NUS	Oversees site safety	Information can be provided at a later date

DVM Data Validation Manager
FOL Field Operations Leader
PM Project Manager
QAM Quality Assurance Manager

SAP Worksheet #8 -- Special Personnel Training Requirements Table

(UFP-SAP Manual Section 2.4.4)

Project Function	Specialized Training By Title or Description of Course	Training Provider	Training Date	Personnel/ Groups Receiving Training	Personnel Titles / Organizational Affiliation	Location of Training Records/ Certificates[1]
Field and Laboratory Personnel See Below Text						
Utility Clearance and Location as per SAP SOP						

[1] If training records and/or certificates are on file elsewhere, document their location in this column. If training records and/or certificates do not exist or are not available, then this should be noted.

All field personnel will have appropriate training to conduct the field activities to which they are assigned. Additionally, 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 Code of Federal Regulations (CFR) 1910.120(b)(4). Safety requirements are addressed in greater detail in the accompanying TtNUS Health and Safety Plan (HASP) (under separate cover).

The selected analytical laboratory (Northeast Laboratory Services) has successfully completed the laboratory evaluation process required as part of the Naval Facilities Engineering Service Center (NFESC) Quality Assurance Program and described in "Department of Defense (DoD) Quality Systems Manual (QSM)" (January 2006) and has been approved by the Navy for the analysis/methods listed in the SAP.

SAP Worksheet #9- Project Scoping Session Participants Sheet
(UFP-SAP Manual Section 2.5.1

The following two worksheets are provided:

- Worksheet #9.1 concerning relevant activities from June 2007 until submittal of the initial draft UFP-SAP in October 2007.

- Worksheet #9.2 concerning scope revision and expansion during the Data Quality Objectives (DQO) meeting of February 2008 and addressing significant comments on the initial draft UFP-SAP dated October 2007.

SAP Worksheet #9.1- Project Scoping Session Participants Sheet

Project Name: 1,4-Dioxane Supplemental RI of Eastern Plume Projected Date(s) of Sampling: October/November 2007 Project Manager: Linda Klink	Site Name: Eastern Plume Site Location: Naval Air Station Brunswick, Maine

Date of Session: Not Applicable
Scoping Session Purpose: Refer to the following discussion on Comments/Decisions concerning relevant activities from June 2007 until submittal of the draft UFP-SAP in October 2007.

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Not applicable (see discussion below)					

Comments/Decisions:

Other than general discussions among the Stakeholders during technical meetings, scoping meetings specific to the 1,4-dioxane area of the Eastern Plume have not been held to date. However, the Stakeholders did agree on modifying the Eastern Plume monitoring plan to increase the number of wells sampled for 1,4-dioxane to yield data that better define its presence within the Eastern Plume. As a result, during the April 2007 long-term monitoring event, groundwater samples from seven shallow and 15 deep monitoring wells, one deep piezometer, and the four extraction wells were analyzed for 1,4-dioxane. The validated results are presented in the draft Sites 1 and 3 and Eastern Plume Monitoring Event 30 of April 2007 Report [ECC, August 2007 (draft)].

Appendix C-1 provides correspondence between the United States Environmental Protection Agency (USEPA or EPA) and the Navy that serves as the basis for the proposed activities, summarized as follows. Note that various key stakeholders are copied on the correspondence, including Claudia Sait (MEDEP), Dale Mosher (NAS Brunswick), Lisa Joy (NAS Brunswick), Carolyn LePage (LePage Environmental), Lawrence Lansdale (BRAC PMO HQ), Amy Walker (NAVFAC HQ), Kim Parker-Brown (NAVFAC HQ), Jennifer Wright (NAVFAC Atlantic), Dan Wadill (NAVFAC Atlantic), Lonnie Monaco (MidLANT), Bob Schirmer (MidLANT), Wanda Holmes (CNO N45), Bonnie Capito (NAVFAC Atlantic, Administrative Record), Al Easterday (ECC), Gina Calderone (ECC), Catherine Guido (ECC), and Linda Klink (Tetra Tech or TtNUS) The EPA correspondence, described as follows, was also in concert with and on behalf of MEDEP.

- A letter dated June 5, 2007, from the Navy (Dawn Kincaid, BRAC Environmental Coordinator) to EPA (Christine Williams, Remedial Project Manager) stated that 1,4-dioxane is an emerging contaminant, making it difficult to select defensible criteria for initiating and completing remedial action. Acknowledging the State of Maine's Maximum Exposure Guideline (MEG) of 32 µg/L for 1,4-dioxane, the Navy proposed conducting a risk assessment to support joint discussions on future actions. The Navy also proposed continued groundwater monitoring for 1,4-dioxane.
- A response letter from the Navy to EPA dated June 26, 2007, requested an extension to an EPA request (letter dated June 6, 2007) for a schedule to be provided within 30 days. The extension

was requested to allow the Navy to consider the serious policy implications raised by 1,4-dioxane and to allow further discussion on the subject at a meeting on July 10, 2007 (minutes not available).

- A letter from the Navy dated July 27, 2007, responding to EPA's June 6, 2007, letter stated that the current extraction system (treatment plant influent concentration) does not exceed the State MEG for 1,4-dioxane. Moreover, neither the interim nor final Records of Decision (RODs) include 1,4-dioxane as a contaminant of concern; therefore, the extraction wells are not properly positioned to remove 1,4-dioxane. In the letter, the Navy also proposed a schedule to address the 1,4-dioxane issue.

Note that Mike Daly replaced Christine Williams as EPA remedial project manager for the NAS Brunswick site effective October 1, 2007.

Action Item:

The Navy's task is to submit the subject Sampling and Analysis Plan (SAP) addressing the Navy detailed plan for addressing the 1,4-dioxane issue.

Consensus Decision:

As per the correspondence between the Navy and EPA, there is consensus on conducting additional Remedial Investigation (RI) activities to refine the extent of 1,4-dioxane groundwater contamination before deciding whether a remedial action plan [supported by a Feasibility Study (FS)] or an amendment to the remedial design is appropriate.

SAP Worksheet #9.2- Project Scoping Session Participants Sheet

Project Name: 1,4-Dioxane Supplemental RI of Eastern Plume and Bedrock [Day 1 DQO Meeting] Projected Date(s) of Sampling: Fall 2008 Project Manager: Linda Klink	Site Name: Eastern Plume Site Location: Naval Air Station Brunswick, Maine

Date of Session: 02/26/08 [Day 1 DQO Meeting–1,4-Dioxane Groundwater Characterization]
Scoping Session Purpose: Identify information needed to characterize the extent of 1,4-dioxane in groundwater, focusing on areas exceeding the Maine Exposure Guideline (MEG) of 32 ug/L.

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Mike Daly	EPA Remedial Project Manager (RPM)	EPA	617-918-1386	Daly.Mike@epamail.epa.gov	RPM
Claudia Sait	MEDEP Remedial Project Manager (RPM)	MEDEP	207-287-7713	claudia.b.stait@maine.gov	RPM
Chris Evans	MEDEP Geologist	MEDEP	207-287-7656	Gordon.C.Evans@maine.gov	Geologist
Victoria Boundy	MRRA Planning	MMRA	207-798-6512	victoriab@mrra.us	MRRA
Carolyn Lepage	LePage Environmental, BACSE Technical Advisor	BACSE	207-777-1049	calepage@adelphia.net	BACSE
Ed Benedikt	BACSE	BACSE	207-442-0448	Contact Carolyn LePage	BACSE
Todd Bober	NAVFAC Mid-Atlantic Remedial Project Manager (RPM)	Navy	215-897-4911	todd.bober@navy.mil	RPM
Lisa Joy	NAS Brunswick Environmental Director	NASB	207-921-1720	lisa.joy@navy.mil	NASB
Michael Fagan	NAS Brunswick IR Coordinator	NASB	207-921-1717	Michael.Fagan1@navy.mil	NASB
Tom Johnston	Tetra Tech DQO Facilitator	TtNUS	412-921-8615	tom.johnston@tetratech.com	DQOs
Linda Klink	Tetra Tech Project Manager	TtNUS	412-921-8650	linda.klink@tetratech.com	PM
Chuck Race	Tetra Tech Geologist	TtNUS	978-474-8437	charles.race@tetratech.com	Geologist
Joe Logan	Tetra Tech Engineer	TtNUS	412-921-7231	joe.logan@tetratech.com	Engineer
Al Easterday	ECC Project Manager	ECC	508-229-2270	aeasterday@ecc.net	PM
Gina Calderone	ECC Project Manager/Geologist	ECC	845-687-0427	gcalderone@ecc.net	Geologist
Jackson Kiker	ECC Chemist	ECC	508-229-2270	JKiker@ecc.net	Chemist

Project Name: 1,4-Dioxane Supplemental RI of Eastern Plume and Bedrock [Day 2 DQO Meeting] Projected Date(s) of Sampling: NA Project Manager: Linda Klink	Site Name: Eastern Plume Site Location: Naval Air Station Brunswick, Maine

Date of Session: 02/27/08 [Day 2 DQO Meeting–Remedial System Design Considerations]
Scoping Session Purpose: Begin process of establishing a listing of viable remedial alternatives, recognizing that a detailed evaluation of the remedial alternatives cannot be conducted until groundwater characterization is accomplished. Ensure that the necessary information for groundwater characterization and design are obtained in one effort.

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Mike Daly	EPA Remedial Project Manager (RPM)	EPA	617-918-1386	Daly.Mike@epamail.epa.gov	RPM
Claudia Sait	MEDEP Remedial Project Manager (RPM)	MEDEP	207-287-7713	claudia.b.stait@maine.gov	RPM
Chris Evans	MEDEP Geologist	MEDEP	207-287-7656	Gordon.C.Evans@maine.gov	Geologist
Fred Lavallee	MEDEP Engineer	MEDEP	207-287-7677	Fred.c.lavallee@maine.gov	Engineer
Victoria Boundy	MRRA Planning	MMRA	207-798-6512	victoriab@mrra.us	MRRA
Carolyn Lepage	LePage Environmental, BACSE Technical Advisor	BACSE	207-777-1049	calepage@adelphia.net	BACSE
Carol Warren	BACSE	BACSE	207-772-1262	carol@wacubu.com	BACSE
Todd Bober	NAVFAC Mid-Atlantic Remedial Project Manager (RPM)	Navy	215-897-4911	todd.bober@navy.mil	RPM
Michael Fagan	NAS Brunswick IR Coordinator	NASB	207-921-1717	Michael.Fagan1@navy.mil	NASB
Tom Johnston	Tetra Tech DQO Facilitator	TtNUS	412-921-8615	tom.johnston@tetratech.com	DQOs
Linda Klink	Tetra Tech Project Manager	TtNUS	412-921-8650	linda.klink@tetratech.com	PM
Chuck Race	Tetra Tech Geologist	TtNUS	978-474-8437	charles.race@tetratech.com	Geologist
Joe Logan	Tetra Tech Engineer	TtNUS	412-921-7231	joe.logan@tetratech.com	Engineer
Al Easterday	ECC Project Manager	ECC	508-229-2270	aeasterday@ecc.net	PM
Gina Calderone	ECC Project Manager/Geologist	ECC	845-687-0427	gcalderone@ecc.net	Geologist
Jackson Kiker	ECC Chemist	ECC	508-229-2270	JKiker@ecc.net	Chemist

Project Name: 1,4-Dioxane Supplemental RI of Eastern Plume and Bedrock [Day 3 DQO Meeting] Projected Date(s) of Sampling: NA Project Manager: Linda Klink	Site Name: Eastern Plume Site Location: Naval Air Station Brunswick, Maine

Date of Session: 02/28/08 [Day 3 Technical Meeting–Bedrock Characterization]
Scoping Session Purpose: Identify information needed to characterize bedrock and associated potential groundwater contamination considering that one bedrock well (MW-308) exhibits chlorinated VOCs that exceed Federal MCLs and/or State of Maine MEGs.

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Mike Daly	EPA Remedial Project Manager (RPM)	EPA	617-918-1386	Daly.Mike@epamail.epa.gov	RPM
Bill Brandon	EPA Technical Specialist	EPA	Contact Mike Daly	brandon.bill@epa.gov	Technical Advisor
Dave McTigue	Gannett Fleming/EPA Geologist	EPA/ Gannett Fleming	Contact Mike Daly	dmctigue@gfnet.com	Technical Advisor
Claudia Sait	MEDEP Remedial Project Manager (RPM)	MEDEP	207-287-7713	claudia.b.stait@maine.gov	RPM
Chris Evans	MEDEP Geologist	MEDEP	207-287-7656	Gordon.C.Evans@maine.gov	Geologist
Victoria Boundy	MRRA Planning	MMRA	207-798-6512	victoriab@mrra.us	MRRA
Carolyn Lepage	LePage Environmental, BACSE Technical Advisor	BACSE	207-777-1049	calepage@adelphia.net	BACSE
Carol Warren	BACSE	BACSE	207-772-1262	carol@wacubu.com	BACSE
Todd Bober	NAVFAC Mid-Atlantic Remedial Project Manager (RPM)	Navy	215-897-4911	todd.bober@navy.mil	RPM
Lisa Joy	NAS Brunswick Environmental Director	NASB	207-921-1720	lisa.joy@navy.mil	NASB
Michael Fagan	NAS Brunswick IR Coordinator	NASB	207-921-1717	Michael.Fagan1@navy.mil	NASB
Chuck Race	Tetra Tech Geologist	TtNUS	978-474-8437	charles.race@tetratech.com	Geologist
Johanna Trout	Tetra Tech notetaker	TtNUS	978-474-8442	Johanna.Traut@tetratech.com	Notetaker
Al Easterday	ECC Project Manager	ECC	508-229-2270	aeasterday@ecc.net	PM
Gina Calderone	ECC Project Manager/Geologist	ECC	845-687-0427	gcalderone@ecc.net	Geologist
James Gatherer	ECC Technical Specialist	ECC	Contact Al Easterday	jamesgatherer@hotmail.com	Technical Specialist

Comments/Decisions:

The originally identified 1,4-dioxane area was located in the southern area of the Eastern Plume, south of Mere Brook (see Appendix C-2 for figures showing locations of these features). After the draft SAP for Supplemental Remedial Investigation of 1,4-dioxane in the Eastern Plume was submitted in October 2007, results from an ECC investigation indicated that 1,4-dioxane was present in both pore water and groundwater north of Mere Brook. Furthermore, even further to the north, during ECC sampling of a new extraction well, EW-05B installed in July 2007 to address volatile organic compounds (VOCs) and sampled in late September 2008, 1,4-dioxane was detected. These events resulted in a DQO meeting.

The DQO process was split into three day-long sessions. The attendees varied each day and are identified above. Day 1 (February 26, 2008) was used to identify information needed to characterize the extent of 1,4-dioxane in groundwater, Day 2 (February 27, 2008) was used to start the process of establishing a list of viable remedial alternatives to ensure that the necessary information for groundwater characterization and design could be obtained in one effort. The focus of this investigation, however, is only to collect data to support that effort and not to resolve decisions associated with that effort. Day 3 (February 28, 2007) consisted of a technical meeting with a focus on bedrock groundwater characterization.

The DQO meeting resulted in an expanded work scope in terms of the area of investigation and associated expansion of tasks to include pore water and horizontal/vertical profiling of the plume. Additionally, it was decided to add bedrock investigation to the revised 1,4-dioxane SAP for efficiency purposes and because the overburden and bedrock contamination appear linked. The Navy agreed to procure funding for the expanded work scope.

Action Items: 1,4-DIOXANE DQO MEETING (DAYs 1 and 2)

1. Linda Klink/Chuck Race: TtNUS will work with ECC and others from the project team to establish locations and identify chemical and physical data to be collected in each area of the Eastern Plume and also outside the plume to support attainment of project objectives.

2. Linda Klink/Joe Logan: TtNUS will review existing metals data provided by ECC to evaluate whether it can be used in support of project objectives rather than collecting new metals data.

3. Claudia Sait: MEDEP will determine whether the EPA mobile laboratory can perform 1,4-dioxane analyses at concentrations low enough to support project objectives. She asked Mike Daly of EPA this question the second day of the meeting (see Item 10 below).

4. Gina Calderone: ECC will send groundwater monitored natural attenuation (MNA) data to Linda Klink (completed after the first day of the meeting).

5. Gina Calderone: ECC will send the latest base property boundary data to Linda Klink (completed after the first day of meeting) and to Stakeholders.

6. Todd Bober: Todd will decide who will sign the SAP for the Navy (either Todd or Paul Burgio).

7. Todd Bober/Vicky Boundy/Claudia Sait: Find a copy of the infiltration gallery Design Basis Memorandum, which is needed by TtNUS for evaluating remedial alternatives. If MEDEP has it and the Navy wants it, they will have to go to MEDEP offices to obtain it because Claudia has no resources to provide it any other way.

8. Todd Bober: The State was not sure they could accept 32 µg/L 1,4-dioxane concentrations for discharge to groundwater after treatment of the contaminant plume. Navy will propose discharge concentration limits for 1,4-dioxane from the groundwater treatment system to the State for their approval. This will be accomplished in a letter to the State from the Navy. The Navy will ask MEDEP for the list of discharge standards as their basis. The Navy will cite the ROD as the basis for their request to the MEDEP.

9. Mike Daly/Todd Bober: EPA and Navy to verify applicability of risk goals to decision for cleanup to ensure whether 32 µg/L is the only limit that must be achieved or whether lower limits must be achieved because of contributions to risk from more than one chemical.

10. Mike Daly: Determine whether the EPA mobile laboratory can perform field screening analysis of 1,4-dioxane at low enough detection limits (i.e., 20 ug/L or less) to support the project objectives.

11. Al Easterday: Determine whether power is available from the new extraction well vault (EW-05B) to support portable skids with treatment units on them.

Action Items: BEDROCK TECHNICAL MEETING (DAY 3)

12. Linda Klink/Chuck Race: Add the following items to the revised SAP:

Site 11 Tasks
- Review existing information on Site 11 (and Site 4 and Site 13) and removal action
- Install new bedrock well(s) based on strike/dip information
- Install transducers to monitor groundwater level at Site 11. Private wells east of plume were assumed to act as pumping wells in determining if there is a hydraulic connection with the plume. (This action item was eliminated after the DQO meeting because the private wells located east of the Eastern Plume are too distal from Site 11.)

MW-308 Area
- Install a bedrock well couplet that discretely monitors the bedrock where the overburden is properly sealed off from the bedrock. Conduct continuous soil sampling during drilling.
- Install a total of three bedrock well couplets in the vicinity of the bedrock well MW-308 in vicinity of bedrock depression and P-106. Use existing data to locate wells and discuss with project team.
- Well couplets would be monitored twice a year and at least for two years duration as part of the Long Term Monitoring Plan (LTMP) of the Eastern Plume for VOCs and 1,4 dioxane.
- Install overburden wells upgradient and cross-gradient of MW-308 at equal elevation to provide vertical gradient information.
- Install transducers to monitor groundwater levels during shut down of extraction network of bedrock wells MW-308, MW-309A, and MW-309B and any newly installed bedrock wells. Private wells to east would act as pumping wells to determine if any hydraulic connection. (This action item was eliminated after the DQO meeting because the private wells located east of the Eastern Plume are too distal from the MW-308 Area.)
- Conduct downhole geophysics at each of the couplets: caliper, heat-pulse flowmeter, natural gamma, acoustic televiewer, resistivity, and fluid temperature.
- In-well salinity testing (to consider, but not necessary if geophysics is completed at each well).
- Coring – one representative core for the three couplets but not at each well location.
- Identification of private wells east of Eastern Plume using bedrock wells within close proximity of the site (i.e., 1/2 mile).

- Continue to monitor bedrock wells at plume as part of LTMP and assess groundwater data from newly installed bedrock well couplets to determine whether additional monitoring wells are necessary to define the nature and extent of bedrock impact.

13. Todd Bober: Determine whether to include the bedrock groundwater investigation work items identified in the technical meeting in the 1,4-Dioxane SAP or a separate SAP. (After the meeting, it was determined that the bedrock groundwater investigation work would be included in the 1,4-Dioxane SAP).

14. Todd Bober: Determine whether to include groundwater sampling of the residential domestic water supply well (identified as RW1) in the LTMP.

15. Linda Klink/Jane Connet: Create a database and add to GIS. Inventory and collect info on private wells, if available, using:
    - Town Assessor Record Review
    - State of Maine Geologic Survey Well records
    - Conduct survey to determine use of private wells and any other known well information

Consensus Decisions:

The Navy and regulatory agencies (EPA and MEDEP) agreed that the significant additions to the original scope warrant a complete review of the updated version; therefore, it was agreed that the revised SAP would be submitted as a draft to reflect the current situation. It was also agreed upon that prior to Stakeholder review, the SAP will also need to be reviewed by the Navy's government chemist.

There was agreement that prior to submittal of the revised SAP, Worksheet # 17, including a map(s) of the proposed pore water locations and groundwater profiling locations, would be submitted to the regulatory agencies for review and concurrence. Permanent overburden and bedrock well locations and well screen intervals were to be generally described in Worksheet #17 but specific locations cannot be established until after the proposed pore water and groundwater profiling results are available for evaluation; again, the regulatory agencies will be involved in review and concurrence of locations.

SAP Worksheet #10 -- Problem Definition

(UFP-SAP Manual Section 2.5.2)

INTRODUCTION

History

Investigation at what was identified as the Eastern Plume began in 1987 when groundwater analytical results indicated the presence of contamination as a result of leaching from past solvent disposal practices at Site 4 (Acid/Caustic Pit), Site 11 (Former Fire Training Area), and Site 13 (Defense Reutilization and Marketing Office Area (figure provided in Appendix C-2). The dissolved-phase plume associated with these disposal activities consisted primarily of chlorinated volatile organic compounds (VOCs) including tetrachloroethene, 1,1,1-trichloroethane, and trichloroethene. The Eastern Plume was found to extend in a north-south orientation (Appendix C-2). The northern one-third area of the plume is located to the north of Gurnet Road in a wooded area and beneath a recreational and picnic area on base. There are two surface water bodies located in this area (Picnic Pond and Merriconeag Stream). The southern two-thirds of the plume lies beneath an area comprised of woodland, wetlands, and paved access roads. There are two surface water bodies located in this area (Mere Brook and Merriconeag Stream).

In June 1992, an interim Record of Decision (ROD) identifying a groundwater extraction and treatment system (GWETS) as the selected remedy was signed. The GWETS operation began in June 1995 and consisted of an extraction system, addressing the northern and southern lobes of the plume, and treatment via ultraviolet oxidation. The treated groundwater was discharged to the Brunswick Sewer District. In 1998, the final ROD was signed featuring hydraulic containment (via extraction), recovery, and treatment to remediate groundwater. The ROD identified the remedial action objectives for the Eastern Plume as follows: (1) to minimize further migration of the Eastern Plume, (2) to minimize any further negative impact to surface water resulting from discharge of contaminated groundwater, (3) to reduce the potential risk associated with ingestion of contaminated groundwater to acceptable levels, and (4) to restore the aquifer. In December 2000, an Explanation of Significant Differences (ESD) for the Eastern Plume altered the original GWETS to remove the ultraviolet oxidation system and replace it with an air stripping system with carbon polishing and to replace discharge to the Brunswick Sewer District with discharge to an infiltration gallery. The treatment system went on line in January 2001 and the infiltration gallery went on line in February 2002. The GWETS objective is to achieve hydraulic control (via extraction) and to reduce hot-spot concentrations and total concentrations of VOCs in the Eastern Plume. The Navy has been performing long-term monitoring, maintenance, and corrective measures as required by the ROD.

During the April 2004 long-term monitoring event, in addition to the standard sampling and analysis, samples from selected monitoring wells were analyzed for an emerging contaminant, 1,4-dioxane. This contaminant has been used as a solvent stabilizer associated with solutions of the solvent 1,1,1-trichloroethane (TCA). Elevated concentrations of 1,4-dioxane (180 ug/L) compared to criteria [State of Maine's Maximum Exposure Guideline (MEG) of 32 ug/L] were encountered in the southeastern portion of the plume during the April 2004 event. Moreover, in 2005, a pore water sampling event was conducted in the southern area of the plume along Mere Brook and Merriconeag Stream that indicated upwelling of 1,4-dioxane and VOCs to these surface water bodies (figure provided in Appendix C-2). Also, during the April 2005 monitoring event, the data for the GWETS combined effluent were 43 J ug/L and 20 J ug/L (duplicate), resulting in a concern about the potential for untreated discharge of 1,4-dioxane. As a result, the number of monitoring wells to be sampled for 1,4-dioxane was increased to yield more spatially comprehensive data for the Eastern Plume. More recently, the April 2007 groundwater monitoring event indicated that two monitoring wells in the southeastern portion of the Eastern Plume exceeded the MEG for 1,4-dioxane.

Also, the Navy began collecting monthly influent and effluent concentration data from the GWETS in July 2006 for 1,4-dioxane. While 1,4-dioxane in the GWETS effluent was less than its MEG, it was largely untreated in passing through the air stripping system with carbon polishing; the extraction well concentrations ranged from 1.4 to 9.7 ug/L. The GWETS would have to be expanded and modified to reduce 1,4-dioxane concentrations yet the design of a comprehensive treatment system cannot be evaluated until the plume is defined; input information such as locations, concentrations, and flow rates of contaminated groundwater that are critical to the treatment system design are not known.

In 2007, during meetings with the regulators (EPA and MEDEP), the Navy expressed that 1,4-dioxane's status as an emerging contaminant made for serious policy implications in selecting defensible criteria for initiating and completing remedial action. While acknowledging the State of Maine's MEG of 32 µg/L for 1,4-dioxane, the Navy stated that the current extraction system (treatment plant influent) does not exceed the State MEG for 1,4-dioxane and, moreover, neither the interim nor final RODs include 1,4-dioxane as a contaminant of concern. However, the Navy acknowledged that the extraction wells are not properly positioned to remove 1,4-dioxane. Ultimately, consensus with the regulators was reached on conducting a supplemental RI to refine the extent of 1,4-dioxane groundwater contamination before deciding whether a remedial action plan [supported by a Feasibility Study (FS)] or an amendment to the remedial design is appropriate. A SAP was prepared by TtNUS, draft dated October 2007, in support of the supplemental RI, which focused on the hot spot in the southeastern area of the plume, south of Mere Brook. Shortly after submittal of the draft SAP, however, new data became available that dramatically expanded the scope of the planned investigation. Specifically, results from an investigation by ECC, a Navy contractor, indicated that 1,4-dioxane was present in both pore water and groundwater north of Mere Brook. Furthermore, even further to the north within the Eastern Plume, analytical results from ECC September

2007 sampling of a new extraction well, namely EW-05B, installed to extract groundwater contaminated with VOCs showed elevated 1,4-dioxane concentrations (compared to the MEG) at that location. Finally, groundwater from one bedrock well (MW-308) located in the northern portion of the Eastern Plume was found to contain chlorinated VOCs that are similar to those found in the Eastern Plume and exceed criteria (MEGs and Maximum Contaminant Levels [MCLs]); therefore, the overburden and bedrock contamination appear to be linked. However, the contamination may not be representative of bedrock because the well was completed without a steel casing grouted into the top of bedrock and so the annular seal may have become compromised.

The expanded scope of the investigation was discussed with the Stakeholders, including the Navy and the regulators, during a DQO meeting in February 2008. Considering the complexity and urgency of the issues, in particular the implications to the existing treatment system in place, DQO discussions were split into three day-long sessions.

- Day 1 (February 26, 2008) of the meeting was used to identify information needed to characterize the extent of 1,4-dioxane in overburden groundwater, focusing on areas exceeding the MEG of 32 ug/L.

- Day 2 (February 27, 2008) of the meeting was used to establish information inputs associated with viable remedial alternatives, while recognizing that a detailed evaluation of the remedial alternatives cannot be conducted until groundwater characterization is accomplished. Considering remedial alternatives early in the project helps ensure that the necessary information for groundwater characterization and design can be obtained in one effort and advances the process by starting the next step, the Feasibility Study (FS).

- Day 3 (February 28, 2008) of the meeting focused on bedrock groundwater characterization, considering that VOC contamination was encountered in one bedrock well (MW-308) and so the overburden and bedrock contamination appear to be linked.

It was agreed at the DQO meeting that the significant additions to the original project scope warrant a complete replacement of the draft October 2007 SAP and a complete review of the updated version. Much of the previously presented material in the draft SAP has been superseded by the recent update of the ECC Conceptual Site Model (CSM) that reflects current-day conditions. Also, the Day 2 DQOs, while important, are incidental to the subject field effort and so have been moved to Appendix C-4. These DQOs were developed only to ensure that data critical to evaluating treatment system design are not likely to be overlooked during this investigation, thus increasing the overall cost-effectiveness of this effort.

During the DQO meeting, a phased sequential approach to the field work was agreed upon to address the Day 1 and Day 3 meeting DQOs, with the regulators (EPA and MEDEP) involved in the decision-making process. The phases of field work consist of the following:

- Pore water sampling along Merriconeag Stream to aid in establishing transects for groundwater profiling. Note this task is being conducted by the EPA and MEDEP with requirements addressed by MEDEP under separate cover from the subject SAP.

- Groundwater profiling of the overburden via temporary wells plus bedrock investigation via permanent monitoring wells to delineate the Eastern Plume, refine geology/hydrogeology, and aid in determining migration potential. Slug tests and water-level measurements from the new wells will aid in the hydrogeology investigation.

- Installation and sampling of permanent overburden monitoring wells plus comprehensive evaluation including most recent data from existing Eastern Plume wells that are part of the long-term monitoring program. Slug tests and water-level measurements from the new wells will aid in the hydrogeology evaluation.

As a result of the phased approach, specific locations for permanent overburden and bedrock wells (and well screen intervals) cannot be established until after the proposed pore water and groundwater profiling results are available for evaluation; again, the regulatory agencies will be involved in review and concurrence of locations.

Conceptual Site Model

A Conceptual Site Model (CSM) was generated with input from the Navy, EPA, MEDEP, and BACSE. The CSM was originally developed in 2000, using the data collected during the Remedial Investigation (RI) (E.C. Jordan, 1990 and 1991), and typically is updated in December of each year and when new information becomes available on the Eastern Plume. Data collected during the long-term monitoring program have also been used to develop an understanding of site conditions and to refine the CSM. The most recent version of the CSM (ECC, July 2008) is provided in Appendix B. Supporting geology/hydrogeology and analytical historical information, such as geologic cross sections and tag maps, are included in Appendices C-2 and C-3, respectively.

In summary, CSM describes the stratigraphy in the area of the Eastern Plume (recent depiction of the Eastern Plume is provided in Appendix C-2) as comprised of interbedded sand, silt, and clay units that overlie the undulating bedrock surface and some occasional discontinuous till. Three major overburden layers are present: upper sand, transition which contains a sandy interval often referred to as the lower

sand, and clay. The upper sand unit consists of fine sands that readily transmit groundwater; the unit is approximately 10 to 20 feet thick across the Eastern Plume area and has the highest conductivity of the overburden units. The transition unit lies between the upper sand and the clay, and is composed on interbedded sands, silts, and clay; the transition unit ranges in thickness from 0 to 30 feet and forms a wedge that pinches out just east of the Eastern Plume. The transition unit contains relatively thin and possibly continuous sand layers that may act as preferential pathways for groundwater movement within this unit, including a mappable sandy interval near the base of the transition unit referred to as the lower sand. The relatively impermeable Presumpscot clay underlies the transition unit and is draped over the bedrock in a nearly continuous layer; the clay thickness has been measured up to 80 feet and in one area the clay appears to be very thin or not present. The bedrock underlying the site is comprised of a micaceous schist, with pegmatite veins up to several feet thick. Major fractures were not typically encountered; however, bedrock test wells do yield water.

The CSM also describes the hydrogeology and contaminant transport. Based on available data, the direction of bulk groundwater flow is generally to the southeast except in the vicinity of Merriconeag Stream and Mere Brook, where the groundwater flow direction may shift so that is perpendicular to the drainage channel. In particular, it appears the 1,4-dioxane is migrating through fine-to medium-sand lenses within the transition unit; however, the concentration distribution or the migration pathways on both sides of Mere Brook are currently unknown. Furthermore, buried coarser stream lenses may act as preferential contaminant migration pathways. The Eastern Plume has been slowly migrating to the south and southeast. Potential receptors for the Eastern Plume are residential and ecological receptors (aquatic- benthic and fish).

1,4-DIOXANE GROUNDWATER CHARACTERIZATION
(DAY 1 DQOs)

DQO Step 1: State the Problem
(1,4-Dioxane Groundwater Characterization)

Background

The original intent of the SAP issued by the Navy in October 2007 was to focus investigation in the southern area of the Eastern Plume, where 1,4-dioxane, at a maximum concentration in groundwater of 180 ug/L in April 2005, in excess of the MEG of 32 ug/L. Also, during the April 2005 monitoring event, the data for the GWETS combined effluent were 43 J ug/L and 20 J ug/L (duplicate), resulting in a concern about the potential for untreated discharge of 1,4-dioxane. A presentation was conducted by ECC to explain current site conditions. The 1,4-dioxane plume area was originally identified in the southern area of the Eastern Plume, south of Mere Brook. Recently, 1,4-dioxane was detected north of Mere Brook at maximum concentrations of 190 ug/L in pore water (PW-81) and 133 ug/L in groundwater (PZ-MB-C3) as part of another investigation conducted by the Navy's contractor, ECC. In addition, even further to the north, a new extraction well, EW-05B that was installed in July 2007 to address VOCs had a 1,4-dioxane concentration of 179 ug/L when sampled on September 28, 2008. This well is not currently connected to the extraction system.

A revised conceptual site model (CSM) (draft) was developed and presented during the meeting. Note that the CSM has since been updated and is provided in Appendix B. Highlights from the DQO meeting are as follows:

- Three known source areas of groundwater VOC contamination were reaffirmed:
  - Site 4 (Acid/Caustic Pit)
  - Site 11 (Former Fire Fighting Training Area)
  - Site 13 (Defense Reutilization and Marketing Office)

- It is unknown whether additional VOC sources exist off base, although none is expected based on evaluation of current data.

- The Eastern Plume, including 1,4-dioxane, appears no longer to be attached to the source(s) of contamination. A primary contaminant source in soil was remediated by removal at Site 11, and underground tanks used for diesel fuel, waste fuel and oils, and degreasing solvents, were removed and replaced at Site 13. However, the remediation reports need to be reviewed to evaluate whether it is possible that a secondary source of contamination exists at these sites. At Site 4, soil located

under the corner of Building 584 has not been removed because of accessibility issues; therefore, it still remains a potential source of VOC contamination. Also at Site 4, VOCs were detected in groundwater from shallow wells MW-209 (2002) and MW-224 (2004). Overall, a continuing source of significant 1,4-dioxane contamination is not expected to exist.

- The Weapons Compound is located west and upgradient to the southern plume area. There is no current strong evidence to support the Weapons Compound as a source of VOCs considering that the Weapons Compound is primarily used for weapons storage. There is no actual manufacturing done in the compound; however, pre-manufactured components and parts may be assembled within the compound to produce finished weapons. Two lobes pointing westward from the main plume extend about halfway to the Weapons Compound, along east-west lines slightly north and slightly south of the complex. Available data from four wells located between the Weapons Compound and the Eastern Plume (MW-310, MW-206A, MW-206B, and MW-219) suggest that if it is a source, it is not a significant source. In particular, the Weapons Compound known history does not suggest use of 1,1,1-TCA (the presumed source of 1,4-dioxane).

- The vertical and lateral extent of 1,4-dioxane contamination in groundwater have not been adequately delineated to date.

- Regional groundwater flow is west to east and north to south, but Merriconeag Stream and Mere Brook influence groundwater flow to create local deviations from these regional flow directions. Groundwater near the streams is diverted toward the streams, which drain the area. Upwelling of groundwater toward Mere Brook occurs, as evidenced by the recent Navy/ECC investigation.

- Groundwater velocities have not been estimated.

Problem Definition

Based on the new CSM developed by Navy/ECC, a primary concern is groundwater contamination migrating into Mere Brook and Merriconeag Stream and potentially having adverse effects on receptors exposed to the surface water. The remedy in place for the Eastern Plume may no longer be protective since most recent data shows that 1,4-dioxane in groundwater is discharging to Mere Brook.

The delineation of the lateral and vertical extent of 1,4-dioxane in groundwater to at least 32 ug/L, which is the MEG, must be completed. Additionally, because chlorinated VOCs and in particular 1,1,1-TCA and trichloroethene (TCE) are also present in the groundwater, VOCs must be quantified across the plume to support the development of an overall treatment strategy. Lastly, the hydrogeological and geological

conditions at the site need to be studied to determine the future migration of 1,4-dioxane and other VOCs in groundwater.

This knowledge will be used later to support the design and implementation of remedial alternatives, potentially including land use controls. A design goal would be to reduce groundwater 1,4-dioxane concentrations in the aquifer to less than 32 ug/L and to protect the streams from significant 1,4-dioxane contamination. Detection limits during the investigation must be low enough to allow detection of concentrations as low as the 6.1 ug/L, which is the EPA screening criterion.

BEDROCK CHARACTERIZATION
(DAY 3 DQOs)

Day 3 was conducted as a technical meeting and not as a formal DQO meeting, and so DQOs were expanded upon herein following the February 2008 DQO meeting to complete this UFP-SAP.

DQO Step 1: State the Problem
(Bedrock Characterization)

Background

A presentation was made by Navy/ECC to explain current information on bedrock and bedrock contamination at NAS Brunswick. A notebook titled "Handouts for Bedrock Technical Evaluation Group Meeting" was provided to the participants. As part of that presentation, detections of 1,4-dioxane (and VOCs) in the overburden and bedrock were discussed. Highlights are summarized below.

General Bedrock Character. The Eastern Plume is underlain by 5 to 60 feet of clay with the greatest thicknesses in the central area of the plume in the vicinity of a bedrock surface depression. The clay thickness decreases to the east toward Picnic Pond and also to the west near MW-323. The top of clay mimics the top of bedrock surface and noted areas of more elevated concentrations correlate with low lying depressions of clay (e.g., EW-05B). The closest bedrock wells to the southern end of the plume include MW-316, MW-317, and Sites 1 and 3 borings (201, 211, etc). The extraction well (EW-05B) is in a depression underlain by clay.

Underlying the clay are two bedrock formations mapped by the Maine Geological Survey beneath NAS Brunswick. The Cushing Formation underlies the western portion of the Base, and the Cape Elizabeth Formation underlies the eastern portion, including the Eastern Plume. The Flying Point Fault is located northwest of Sites 4, 11, 13 and the Eastern Plume. Pertinent information is as follows:

- The Cushing Formation consists of massive gray gneiss that consists of metamorphosed volcanic and sedimentary rocks. Minerals include plagioclase, quartz, and the micas (biotite and muscovite). Micas are aligned with foliation roughly parallel to the north-northeast regional fault zone.

- The Cape Elizabeth Formation consists of metamorphic foliated and massive gray phyllite and schist with quartz inclusions. Again, foliation is roughly parallel to the north-northeast regional fault zone.

Chronology of Bedrock Investigations. A chronology of previous bedrock investigations conducted at NAS Brunswick is summarized as follows:

- 1990 - Bedrock wells installed as part of the RI
- 2000 - Data gaps identified at Site 11
- 2001 - Site 11 direct-push investigation near MW-323-bedrock knoll
- 2003 - Bedrock investigation by EPA
- 2004 – Geophysica l investigation at Site 11 and downgradient areas
- 2005 - Site 11 bedrock well installation sampling using passive diffusion techniques
- 2006 and 2007 - Monitoring that identified VOCs in excess of drinking water standards in bedrock well MW-308, but no VOCs or 1,4-dioxane were detected in groundwater from a residential well east of the Merriconeag Stream

Long-Term Monitoring Program. Long-term groundwater monitoring is being conducted in compliance with the ROD. Groundwater monitoring data have been collected from eight NAS Brunswick bedrock wells in the Eastern Plume (MW-308, -309A, -309B, 316A, -316B, 317A, -317B, and -323) that were sampled from August 1995 through September 2007. VOC concentrations exceeded criteria in 7 of the 79 groundwater samples collected from monitoring wells MW-308 and MW-323, with six of the seven exceedances associated with MW-308 from more recent samples collected in 2006 and 2007. The exceedance at MW-323, associated with Site 11, occurred in 2003. It was clarified that although there have only been seven exceedances, a greater number of detections were reported in these samples that did not exceed the screening criteria. 1,4-Dioxane was reported as an estimated value in bedrock well MW-308. This is the only bedrock well with detected VOCs exceeding regulatory criteria. According to a Navy Chemist review, the quality of the data is in question. There is sufficient evidence that shows 1) the reported sensitivity is not as low as reported and 2) the data are biased high. These data indicate that bedrock well MW-308 is connected in some way with the Eastern Plume that has historically been associated with the lower sand unit of the overburden.

The monitored natural attenuation (MNA) results suggested that the population of biota in the overburden is not sufficient; although there has been breakdown and chemical reduction of trichloroethene (TCE), the biota is neither biologically degrading all chlorinated VOCs in the entire plume nor continuing to degrade the TCE. Also, degradation of fuel products has occurred but the process may have stalled at this point. It was suggested that bioaugmentation may be needed to stimulate natural biodegradation.

Focus on Known Bedrock Issues.

MW-308: Pertinent information regarding bedrock monitoring well MW-308 follows:

- TCE and 1,1-dichloroethene (1,1-DCE) concentrations exceeded drinking water standards in April 2006, April 2007 and September 2007. The contaminants and concentrations in MW-308 correspond

with Eastern Plume contaminants and concentrations. The highest detections of TCE and 1,1-DCE at MW-308 occurred in April 2006, and although detections still exceed MEGs, they have been decreasing. The most recent groundwater sample was collected using low-flow procedures, but samples from this well have also been collected using passive diffusion bags. The higher detections were associated with low-flow groundwater sampling, and the lower detections were taken using passive diffusion bags.

- This shallow bedrock well MW-308 (70 feet depth below ground surface [bgs]) was completed in 1990 with a 10-foot-long well screen from 60 to 70 feet bgs. The well is screened in the top 30 feet of bedrock, with the top of rock at 40.5 feet bgs. The well was completed without a steel casing grouted into the top of bedrock, and so this well is not discretely monitoring the bedrock zone. It is possible that due to the age of the well (17 years), the annular seal has become compromised.

- According to the boring log for MW-308, poorly graded, stratified silt with a little fine sand and a little clay is present from 18 to 40.5 feet bgs directly overlying fractured bedrock. The soil description is interpreted as the Transition Unit. Sandy strata are present in the Transition Unit. Because the soil sampling was not continuous, and only one description is provided for four samples collected at 5-foot intervals (3 feet missing between five successive samples between 18 and 40 feet bgs), it is possible that clay strata and/or sandy strata are present. Other investigation techniques (cone penetrometer and surface geophysics) suggested the presence of clay in the vicinity of MW-308; however, the soil boring log is expected to be the most reliable because it is based on actual soil samples.

- MW-308 has a very low rock quality designation (RQD) (i.e., high degree of fracturing) in comparison to the other bedrock wells in the area. There was some discussion as to whether the RQD was a function of drilling, based on a table from the RI report, but the work was performed by others so the significance of drilling impacts on the coring cannot be clearly defined. The RQD is more likely a function of the rock in this area, considering that the top 30 feet of bedrock is on a knoll and would be subject to weathering, so is most likely more fractured than bedrock in the surrounding area.

- MW-308 also has the highest hydraulic conductivity (K) value of all the bedrock wells and its K value falls within the range of K values reported in overburden units. Based water levels at on PZ-108 (which has since been destroyed) and MW-308 water levels, there was an upward gradient in this area at the time of the RI report. An overburden well located beside MW-308 is not longer usable, so current vertical hydraulic gradients cannot be determined; therefore, it is uncertain whether groundwater currently flows from the overburden into the bedrock or vice versa in this area.

- Bedrock may be exhibiting dissolved-phase contamination from the overburden at the MW-308 location. MW-308 is located southwest of a former hot spot area of the Eastern Plume near P-106,

east of extraction well EW-4 and directly southwest of new extraction well (and current hot spot) EW-5B. Contaminant travel time has not been estimated; of note, the reported hydraulic conductivity for MW-308 is 2.13 feet per day (7.50E-04 cm per sec), which is higher than other bedrock locations at NAS Brunswick. Concentrations of contaminants at deep overburden well P-106, located northeast of MW-308, have been decreasing steadily. Chlorinated VOC and 1,4 dioxane concentrations in this area decreased sharply between 1995 and 2002, and have been declining since 2002. There is no extraction well near the P-106 area. Decreases in concentrations may be due to movement of the plume core toward the southwest. A phased investigation approach in 2005, led to identification of clay depressions near P-106 and MW-331. Direct-push probe techniques were attempted first, but due to difficult drilling conditions, the investigation was completed with rotosonic drilling methods. Based on the data from the rotosonic logs, direct-push groundwater sample results, and surface geophysical survey results, the new extraction well location (EW-5B) was chosen.

In summary, MW-308 is a shallow bedrock well, extending 30 feet into bedrock on top of a highly fractured bedrock knoll. Although the lower sand unit is present in areas surrounding the well, this unit pinches out in the direct vicinity of MW-308, where there is little to no clay in the overburden above the bedrock and the well is not discretely monitoring the bedrock. MW-308 is downgradient of a former hot spot area, and the contaminants and concentrations in MW-308 correspond with the hot spot contaminants and concentrations. Contaminant concentrations have been decreasing at MW-308 since the highest detection in the spring of 2006, although they still exceed MEGs.

The team discussed why MW-308 has recently become a concern, when historically there have not been exceedances at this location and the following was suggested:

- This well is downgradient of a hot spot
- There is little or no clay in the overburden above the bedrock
- The well is not discretely monitoring the bedrock
- VOC concentrations were lower with the diffusion bag samples relative to the low-flow samples
- Overburden contamination may have migrated into the area around this well where bedrock hydraulic conductivity is high
- The well was not installed with a steel casing sealing the overburden from the monitored zone

- If there were downward gradients in fractured bedrock, it could have resulted in a groundwater migration pathway

- Because of higher bedrock elevation and similar hydraulic conductivity with the Transition Unit sediments, part of the plume could be migrating through the shallow bedrock

- There are no extraction wells near MW-308 to influence gradients

The bedrock surface depressions seem to be important areas and correlate with high concentrations of VOCs, so obtaining more information, especially surrounding the depression to the south of MW-308 and further south of the Weapons Compound, is important. The team agreed that conducting additional surface geophysics is not necessary at this time.

*MW-323 (and MW-NASB-11BR)***:** Pertinent information regarding bedrock monitoring wells associated with Site 11 is as follows:

- The 1,1-dichloroethene (1,1-DCE) concentration exceeded the MEG in only one sample (October 2003) at MW-323 at a concentration of 2 ug/L compared to the MEG of 0.6 ug/L.

- Six passive diffusion samples were collected from varying depth intervals from MW-NASB-11BR, and the VOC concentrations detected were significantly less than MEGs. At the time of the well installation and passive diffusion sampling, the infiltration gallery had been in operation for a few years.

- An important note from the EPA consultant's "bedrock study report" (May 2003) was that the rock in this area is highly variable. There are places where it is highly fractured and places where the fractures have been replaced with quartz veins. The stressed pegmatite has been reworked. There was a second metamorphic stress, which is significant because of the variability in the rock that can create potential pathways in some areas versus quartz-filled veins in other areas. The west-facing slopes tend to be steeper and dip to the east. This influences contaminant migration, which tends to be from high points to low points and may be the reason the Eastern Plume is located in a low-lying area. There was no association noted between the steeper (western- facing) slopes and pegmatite. The bedrock is low lying in this area but is at a higher elevation on the other side of Mere Brook. The Site 11 bedrock well is at the edge of this depression.

- The location of the Site 11 bedrock well (MW-NASB-11BR) was based on geophysical investigations, which provided a better understanding of the pathways for contaminant migration. The well was placed near MW-323; rock was encountered at 30 feet bgs and cored to 94 feet bgs.

Connection between Site 11 and MW-308:

The infiltration gallery was located atop Site 11 and had been operating since 2003 (more than 2 years) at the time that the Site 11 bedrock well (MW-NASB-11BR) was sampled. Several detections (no exceedances) were noted in monitoring wells, but that does not necessarily dismiss Site 11 as the source area for contamination at MW-308. TCA was detected at Site 11, and the Site 11 contaminants correspond with the plume contaminants. At Site 13, 1,2-DCE was detected at 700 ug/L (MW-1303), and at Site 4, low levels of TCE were detected at MW-405. These areas could also have some effect on MW-308.

Bedrock Contamination Potential Sources and Extent: The team discussed whether the contaminant source for the Eastern Plume is at Site 11 and whether background information (e.g., soil contaminants and concentrations) on the removal actions from Site 11 is documented. The Drum Investigation Report submitted by Halliburton NUS in August 1995 stated that 15 test pits were dug around Site 11 prior to the removal action. Low concentrations of TCE and tetrachloroethene (PCE), and elevated xylene and toluene concentrations were encountered. There were also trace semivolatile organic compounds (SVOCs) and low concentrations of the pesticides 4,4'-DDE, 4,4'-DDD, and 4,4'-DDT. More information may be needed to determine if Site 11 is a source of VOC contamination in the Eastern Plume. In the future, additional bedrock wells may be warranted in an effort to pinpoint the source at Site 11 based on strike and dip information.

The team discussed whether is reasonable that contamination is present over a significant area in bedrock. Not enough information is available for this determination. Specifically, the following information is lacking or needs to be researched:

- Information such as COCs for potential source areas (Sites 4, 11, and 13)
- Estimated groundwater velocities
- If a bedrock plume exists or if contamination is only localized near MW-308. No analytical data from bedrock in the immediate vicinity of well MW-308 exist; MW-308 is located in the northern portion of the Eastern Plume and is the only bedrock that has historically had exceedances of regulatory criteria. MW-309A and B, located approximately 300 feet downgradient of MW-308, have no historical exceedances.

Also, pathways and potential future impacts to off-base residential wells are uncertain based on the following information to date:

- A limited number of monitoring wells are located on the eastern side of the Merriconeag stream.

- Groundwater contamination crossing Merriconeag Stream appears unlikely, considering that it should act as a hydraulic barrier and groundwater appears to be upwelling.

- Domestic water-supply wells are located on the eastern side of Merriconeag Stream, and it should be demonstrated that these wells will not be impacted by the Eastern Plume from NAS Brunswick. Contaminant migration in the bedrock aquifer appears unlikely considering that there is a bedrock ridge to the east of the stream and that there is some evidence for upwelling to surface water. Both the MEDEP and Carolyn Lepage stressed that some monitoring should be conducted at the residential well (closest well identified as RW1) to ensure protection of the local residents. This residential well (RW1) has been sampled at least seven times and results have been nondetect for all COCs. Other residential wells have been sampled over the past two years and were nondetecct with the exception of one, which may have been attributed to exhaust during sampling.

Problem Definition

Based on the new CSM, one part of the problem is that the migration pathways for established, overburden contamination entering the bedrock groundwater appear to be unknown. Three areas of interest were identified: (1) the vicinity of MW-308 where 1,1-DCE and TCE were detected in excess of regulatory criteria, although the data may not be representative considering the monitoring well MW-308 well installation methods and, (2) the vicinity of Site 11 where 1,1-DCE was detected albeit at low concentrations (2 ug/L compared to the MCL of 7 ug/L and MEG of 0.6 ug/L), and (3) the Site 11 area is of interest because the infiltration gallery for the Eastern Plume GWETS is located directly over the former source area and close to an area of previously identified shallow bedrock.

A second part of the problem is that private bedrock water-supply wells located east of Merriconeag Stream may be drawing from the shallow bedrock aquifer. The most recent data show that 1,4-dioxane and VOCs have not been detected in bedrock wells east or immediately west (MW-309 A and MW-309B) of Merriconeag Stream. Existing bedrock hydrogeology information, such as potential fractures and water-bearing zones, is not adequate for characterization purposes. However, the off-base private water wells within close proximity of the site have been inventoried and some private wells have been sampled periodically. The inventory of private water wells is to include total depth of the well and water bearing zone information.

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

DATA USERS AND USES

The Navy and regulatory agencies (EPA and MEDEP) will use the data collected from this effort to:

- Determine if MW-308 is acting as a preferential pathway for groundwater flow within the Transition Unit to flow into the shallow bedrock aquifer. If so, immediately abandon MW-308 to eliminate this pathway and replace monitoring of MW-308 in the LTMP with monitoring of at least one of the new bedrock wells to be installed.

- Define site geologic and hydrogeologic conditions in both overburden and bedrock, including groundwater and contaminant (1,4-dioxane and other VOCs) flow paths and velocities.

- Determine if groundwater contamination within bedrock is localized or regional and, if regional, determine the contaminant migration pathway(s) into the bedrock in the vicinity of MW-308 and at Site 11.

- Evaluate if there is a bedrock hydraulic connection between the MW-308 vicinity and the private water-supply wells on the eastern side of Merriconeag Stream and monitor increases/decreases via long-term monitoring.

- Refine the delineation of the extent VOCs, including 1,4-dioxane, in groundwater sufficiently to support a risk assessment (if necessary) and the design of a remediation system that addresses the VOCs including 1,4 dioxane.

- Collect data to support the design of a groundwater remediation system for reducing 1,4-dioxane and other VOC concentrations to acceptable levels to meet MEGs.

The remediation system design will occur later as part of a different project. The DQO planning was split into three day-long sessions. Day 1 focused on characterization of the site, Day 2 included consideration of potential remedial alternatives, and Day 3 focused on bedrock characterization. The Day 2 DQOs, while important, are incidental to the subject field effort and so have been moved to Appendix C-4. These DQOs were developed only to ensure that data critical to evaluating treatment system design are not likely to be overlooked during this investigation, thus increasing the overall cost-effectiveness of this effort.

The data to be collected for supplemental RI of the Eastern Plume must be of sufficient quality, quantity, and spatial resolution to support risk assessment and future remedial design. As previously established, detection limits must be sufficiently low to measure 1,4-dioxane concentrations to 6.1 µg/L or less, which is the EPA Region 9 Preliminary Remediation Goal (PRG) screening level. Since the time of the February 2008 DQO meeting, the EPA has replaced Region 9 PRGs with Regional Screening Levels (RSLs); note that the RSL for 1,4-dioxane is the same value as the PRG. It was agreed upon that project action limits (PALs) and laboratory method detection limits (MDLs) for other VOCs would be provided to the MEDEP and EPA for approval as SAP Worksheet #15, and the VOCs of concern would be those previously detected during groundwater monitoring of the Eastern Plume.

At the time of the DQO meeting, the extent of EPA support for laboratory analyses was unknown. Based on decisions since that time:

- MEDEP will provide the Work Plan for pore water sampling and will lead the sampling effort (August 2008).

- EPA will analyze pore water samples on site for VOCs using the EPA Region 1 mobile laboratory. Additionally, EPA will analyze pore water samples for 1,4-dioxane at a fixed-base laboratory.

- TtNUS will aid in the sample collection effort and will prepare the sample location map.

Data will be reported, managed, and archived as described throughout the remainder of this SAP. The amount of data and other pertinent Project Quality Objectives (PQOs) are described in the remainder of this worksheet and on other worksheets in this SAP.

1,4-DIOXANE GROUNDWATER CHARACTERIZATION
(DAY 1 DQOs)

Data Quality Objectives Step 2: Identify the Goals of the Study
(1,4-Dioxane Groundwater Characterization)

Based on the CSM and issues presented in Worksheet #10, the following decision statements were developed:

Decision Statement #1

Determine whether 1,4-dioxane concentrations in groundwater across the Eastern Plume and pore water in streams in the eastern and northern areas of the Eastern Plume are adequately delineated in the lateral and vertical directions to support remedial decisions for groundwater. If so, stop delineation; otherwise, continue with delineation.

Decision Statement #2

Determine whether the CSM has been sufficiently refined to understand significant groundwater and 1,4-dioxane flow paths (current and future) and migration rates. If these are sufficiently refined, stop data collection; otherwise, continue refining the CSM.

Decision Statement #3

Determine whether chlorinated VOCs are adequately characterized within the 1,4-dioxane plume to support remedial decisions. If they are, stop chlorinated VOC data collection; otherwise, continue chlorinated VOC data collection.

At this point, the project team recognized that several interacting qualitative and quantitative factors would be operational in determining whether enough data of the correct type and quality are obtained to support project objectives. The team agreed that previously used data interpretation methods (e.g., isoconcentration mapping) would suffice to make this determination.

It is assumed that 1,4-dioxane is diluted within the streams; however, pore water contaminant concentrations might have an adverse effect on stream dwellers such as benthic organisms.

Data Quality Objectives Step 3: Identify Information Inputs
(1,4-Dioxane Groundwater Characterization)

The following data inputs are required to support the decisions identified in DQO Step 2:

- 1,4-Dioxane concentrations in groundwater and select pore water samples using a gas chromatographic/mass spectrometric method agreed on by the EPA and Navy. At the time of the February 2008 DQO meeting, TtNUS had planned to use a modified Method 8270 semivolatile organic compound analysis featuring sodium chloride (NaCl) addition that had been approved by the MEDEP chemist. During a July 2008 conference call, however, the analytical method was changed to VOC Method 8260B selective ion monitoring (SIM) to be consistent with the analytical method used by ECC during groundwater monitoring of the Eastern Plume.

- Chlorinated VOC concentrations in groundwater using analytical methods in accordance with TtNUS Standard Operating Procedures (SOPs). The specific compounds are described in Worksheet #15 and analytical methods are listed in WS #23.

- Lithology across the site obtained using standard geological boring log methods in accordance with TtNUS SOPs.

- Groundwater and stream water-level elevations. Groundwater levels must be obtained across the site from any new and existing wells and piezometers using methodology in accordance with TtNUS SOPs.

- Hydraulic conductivity (e.g., from slug testing) in accordance with TtNUS SOPs.

- Well stabilization parameters collected in the field such as pH and turbidity (in accordance with TtNUS SOPs) to ensure that sampling conditions are stable prior to sample collection.

- Pump test data for EW-5B, both hydraulic and chemical data, which will be useful in evaluating the plume and will support any remedial design.

- Alkalinity, hardness (calcium, magnesium, etc.), sulfate, sulfide, and total organic carbon (TOC) data to support treatment design and to estimate aquifer conditions and the potential for natural degradation. Existing data from previously collected MNA and RI data at the facility can be used to meet project needs.

- Dissolved and total iron, plus Fe^{+2} and Fe^{+3}, to estimate aquifer conditions and potential for natural degradation. Existing iron data from previously collected MNA and RI data at the facility can be used to meet project needs.

- TOC concentrations in select uncontaminated soil areas to estimate aquifer conditions and the potential for natural degradation, obtained using a standard fixed-base laboratory analytical method. This information may be obtained from previous investigations.

- Literature distribution coefficients (K_d) for 1,4-dioxane and chlorinated VOCs in soil.

- Fraction organic carbon obtained from K_d and TOC data.

- Total contaminated groundwater volume.

- Other data inputs as identified in Day 2 and Day 3 DQOs.

Based on existing data and site conditions, no significant seasonal variations in contaminant concentrations or water levels are known or expected. Furthermore, such variations would not be expected to have a significant influence on remedial design; therefore, seasonal variations are not a concern. Historical variations in water levels seem to be caused by operation of the existing extraction system.

The team discussed the need to determine the total mass of VOCs in groundwater and concluded that it might not be necessary from a technical point of view to support attainment of project objectives, but it could be useful as a means to communicate to the public the magnitude of the contamination problem on an “order of magnitude” scale.

DQO Step 4: Define the Boundaries of the Study
(1,4-Dioxane Groundwater Characterization)

In addition to the overburden investigation discussed during the Day 1 DQOs, bedrock groundwater will also be investigated, as summarized from the notes of a technical meeting held on Day 3.

The following areas must be investigated to support the decision statements identified in DQO Step 2:

- Central and northern portion of the Eastern Plume to fill data gaps with respect to groundwater 1,4-dioxane (and VOC concentrations) across the area

- The immediate vicinity of MW-308 where bedrock contamination was previously identified
- East of Mere Brook and Merriconeag Stream to delineate known contamination because 1,4-dioxane migrates with groundwater (more quickly than chlorinated VOCs) and is likely to be at the leading edge of any contamination
- Between the Eastern Plume as currently delineated and Picnic Pond (northeastern portion of the site)
- Just north of Old Gurnett Road to bound contamination in the southern area of the Eastern Plume
- South of MW-313 to further delineate potential contamination south of the previously investigated area (southeastern edge of site in the floodplain)
- West of the Eastern Plume to further delineate known contamination
- The core of the plume representing shallow (average 20 feet bgs), intermediate (average 40 feet bgs), and deep (average 60 feet bgs) zones previously investigated to improve understanding of vertical groundwater gradients and migration under Mere Brook, and with sample intervals of up to 10 feet depending on the thickness of the given monitored unit
- Upland area between existing wells MW-308 and MW-331 where intermediate and deep groundwater data are especially important in determining if there is a connection between the overburden and bedrock
- Existing wells 204, 205, 206A, 206B, 208, etc. that are located in the vicinity of the western side of the plume to aid in understanding potential sources of the Eastern Plume
- In the floodplain where shallow, intermediate, and deep groundwater data are all important because upwelling of contaminants to Mere Brook and Merriconeag Stream are of concern.

Slug test locations were not established during the February 2008 DQO meeting. However, they have since been later established and are discussed in Worksheet #17.

DQO Step 5: Develop the Analytic Approach
(1,4-Dioxane Groundwater Characterization)

The previous DQO outputs were assimilated into decision rules that govern data use. It is assumed for the purpose of delineating the extent of contamination that concentrations are greatest within the Eastern

Plume and decrease in the downgradient direction. The decision rules are as follows; individual sample results will be compared to criteria rather than an average of samples in one area:

Decision Rule #1

If the 1,4-dioxane concentration in any overburden groundwater sample on the three-dimensional perimeter of the 1,4-dioxane plume exceeds 32 ug/L then step outward and collect additional samples until 1,4-dioxane concentrations are delineated to less than 32 µg/L. When 1,4-dioxane concentrations in samples on the outer edge of the plume are less than 32 ug/L, stop delineation. An effort will be made to characterize to a concentration of 6.1 ug/L for the regions with 1,4-dioxane concentrations less than 32 µg/L, but this will not drive additional sample collection. Bedrock investigation is discussed later in this worksheet (Day 3).

Data needed to support this decision rule are as follows:

- Well stabilization parameters
- 1,4-Dioxane and chlorinated VOC concentrations in groundwater (and select pore water samples)

Decision Rule #2

If, based on hydrogeological and geological data, the project team agrees that the groundwater and 1,4-dioxane flow paths and velocities are sufficiently determined to support remediation, stop data collection; otherwise, continue data collection.

Factors to be considered include the following:

- Groundwater flow directions from water-level measurements across the plume
- K values of contaminated geologic units from slug testing of new wells (and existing wells as necessary)
- Vertical groundwater gradients
- Subsurface stratigraphy based on standard geological correlation techniques (indicating locations of highest VOC and 1,4-dioxane concentrations and associated relative permeabilities and thicknesses of geologic units)
- Estimated contaminated groundwater volume

Decision Rule #3

If chlorinated VOC concentrations are sufficiently characterized throughout the 1,4-dioxane plume, bounded by 32 ug/L, to support remedial decisions, stop chlorinated VOC data collection; otherwise, continue data collection until concentrations are less than MEGs. Continued characterization may be deferred to another study (the remedial design stage) at the discretion of the project team. The necessary data are identified in DQO Step 3.

DQO Step 6: Specify Performance or Acceptance Criteria
(1,4-Dioxane Groundwater Characterization)

Several interrelated qualitative and quantitative factors make calculation of the minimum number of samples and identification of well locations difficult at this time. The project team agreed that the rationale for well locations in the previous October 2007 version of the SAP prior to the updating of the CSM were satisfactory; therefore, selection of new data collection locations and depths was to continue to be based on similar logic, including selection of pore water sampling locations. The location of new permanent monitoring wells will be based on evaluation of existing available hydrogeologic and geochemical data and the results of the groundwater profiling. The well locations will be determined considering nearby groundwater and pore water data point results. Wells will be located midway between nearby existing wells in the core of the 1.4-dioxane plume. There will be a preference to locate wells across the core of the plume where no data currently exists. Other permanent locations will be considered downgradient of the core of the plume for the purpose of monitoring future potential contaminant migration. In addition, one overburden well will be located beside each of the proposed nested bedrock wells (MW-EP-308R, MW-EP-340, and MW-EP-341). It was agreed during the DQO meeting that each member of the team would have an opportunity to review and concur with the proposed locations. This has since been facilitated by submittal of Worksheet #17, including a draft map(s) showing the proposed locations, which was distributed to the team prior to submittal of the subject version of the SAP. During the DQO meeting, the team agreed that the significant addition to the original scope of work in the October 2007 version of the SAP warranted a complete review of the updated version of the SAP by the regulators. It was also agreed upon that prior to regulator review, the SAP would also be reviewed anew by the NAVFAC government chemist.

As discussed in conference calls and e-mails following the February 2008 DQO meeting, MEDEP will be responsible for preparation of the planning documents for the pore water sampling event. Moreover, MEDEP will be the lead for the field effort (August 2008) and EPA will conduct pore water analyses.

Because the data collected from this groundwater sampling effort could be used for risk assessment (if necessary), full validation of the fixed-base laboratory data will be conducted in accordance with the method-specific criteria for inorganic and organic data review presented in Worksheets 12, 15, and 28

and the logic outlined in Region 1 data validation guidance to apply data qualifiers. The focus will be on organic data review because any inorganic data are generally being collected only to support remedial designs. Field-collected data (e.g., pore water samples, well purging stabilization parameters) will not be validated.

DQO Step 7: Develop the Plan for Obtaining Data
(1,4-Dioxane Groundwater Characterization)

During the February 2008 DQO meeting, the project team agreed that a statistical design for groundwater well installation is not appropriate because factors such as known groundwater flow paths and velocities cannot be addressed with simple statistics. Even if geostatistics was to be used, a significant amount of professional judgment by a Certified Geologist would be necessary during hydrogeologic model development and in data interpretation. Because the team agreed that previously proposed well locations were reasonable, the team agreed that the selection of additional sampling locations could be approached in a manner similar to the original well selection process.

The following specifications were developed by the team to help guide sampling plan development, which was the basis for the design now detailed in Worksheet #17:

- Access to the floodplain (wetlands) and working within the wetlands could be problematic because of wet and soft surface conditions. Alternatively, collection of pore water samples in the wetlands area would not only provide direct concentration estimates in pore water, but it might provide a way to more readily characterize groundwater at the sediment level without having to install wells or direct-push technology (DPT) borings at the risk of getting the drill or DPT rigs trapped in the mud.

- Pore water analyses, especially in the northern area of the Eastern Plume, could help to determine well or DPT locations for groundwater sampling. Pore water sampling might best aid the process if conducted before a groundwater profiling effort. Pore water data might also provide information on areas of groundwater upwelling to surface water. Pore water contaminant concentration results (1,4-dioxane and total VOCs) will be posted beside the associated sample locations and the results contoured, if possible. If there are one or more reaches along Merriconeag Stream that are associated with elevated contaminant concentrations, then these segments will be compared with similar data from nearby monitoring wells to decide if a monitoring well should be located along or upgradient of the reach. In making these decisions, not only should the pore water results be considered, but also vertical and horizontal hydraulic gradients, and well construction/historical groundwater results from near by data points.

- Groundwater profiling (e.g., via electrical conductivity and discrete-interval groundwater sampling) using DPT and with quick-turnaround analyses may be used to help determine well locations that will best support delineation of contamination and characterization of groundwater flow paths, flow velocities, and overall chemistry in support of remedial design. If DPT is unable to penetrate the top of the Presumpscot Clay unit, conventional drilling techniques will be considered.

- During groundwater profiling, transects will be established to characterize various cross sections of the Eastern Plume. One of the profiling transects should characterize the area near MW-308 since contamination has been detected in bedrock at this location. Another transect should characterize the area near new extraction well EW-05B because groundwater from this well had a 1,4-dioxane concentration of 169 ug/L.

- The project team was to pre-approve the pore water and groundwater profiling locations based on figures to be distributed pending Navy funding of TtNUS to conduct this task. The figures have since been provided to the EPA and MEDEP. As discussed in conference calls and e-mails following the February 2008 DQO meeting, MEDEP will be responsible for preparation of the planning documents for the pore water sampling event. Moreover, MEDEP will be the lead for the field effort (August 2008), and EPA will conduct pore water analyses.

- The Supplemental RI permanent monitoring wells for groundwater will be added to LTMP for the Eastern Plume; also, currently existing Mere Brook wells will also be considered for inclusion. Both DPT and conventional drilling techniques are needed for installation of permanent monitoring wells based on varying depths anticipated.

The team agreed on the following general sequence of characterization:

- Pore water sampling with quick-turnaround analyses.

- Groundwater profiling of cross-sectional transects with quick-turnaround analyses.

- Permanent well installation (DPT boring or conventional methods) for groundwater sample collection and aquifer characterization. Lithology data will be collected during drilling activities as a natural part of logging holes.

- Other data collection needs as identified in previous DQO steps will be addressed.

BEDROCK CHARACTERIZATION
(DAY 3 DQOs)

The Day 3 meeting was conducted as a technical meeting and not as a formal DQO meeting and so DQOs were elaborated upon following the February 2008 DQO meeting to complete this UFP-SAP.

Data Quality Objectives Step 2: Identify the Goals of the Study (Bedrock Characterization)

Based on the CSM and issues presented on Worksheet #10, the following decision statements were developed:

Decision Statement #1

Determine if bedrock groundwater is contaminated in the vicinity of MW-308. If the bedrock is contaminated, then continue bedrock investigation. If the bedrock is not contaminated, then terminate bedrock investigation. The team will decide if and when to abandon MW-308; this decision will be made by the team prior to initiation of the bedrock investigation field work. Abandonment of monitoring well MW-308 may be delayed until the team evaluates data from the new well couplets (i.e., paired wells at a given location monitoring different depth intervals). The new overburden and bedrock wells will be located approximately 10 feet northwest (upgradient) of MW-308 to avoid potential impacts from MW-308 that may have compromised well integrity. Conversely, MW-308 may first be abandoned before proceeding with installation of the replacement well primarily because if the structural integrity of MW-308 is compromised, contaminated groundwater may be pulled in via fractures by the replacement well.

Decision Statement #2

Determine if contaminant concentrations in bedrock are from the overburden immediately upgradient of the MW-308. If contaminant concentrations are higher in newly installed upgradient wells (along potential migration pathways), relative to MW-308, or evaluation of vertical gradients indicates that there is no pathway to the bedrock, then stop investigating contaminant migration pathways; otherwise, continue investigating potential migration pathways.

Decision Statement #3

Decision Statement 3 is intended to determine if Site 11 is a source of contamination to downgradient well MW-308. If Site 11 is a source of contamination to MW-308, then the same contaminants would need to

be present at Site 11. If similar contaminants are not present in bedrock, then stop investigating potential sources. If the contaminants are present, then discuss options with the project team.

Decision Statement #4

Determine if the bedrock aquifer in the vicinity of MW-308 is hydraulically connected to private water-supply wells east of Merriconeag Stream. If there is no response attributable to water-supply well operation, then stop. If a hydraulic response may be attributed to water supply well operation, then discuss other options with the project team.

Data Quality Objectives Step 3: Identify Information Inputs
(Bedrock Characterization)

The following data inputs are required to support the decisions identified in DQO Step 2:

Site 11 Tasks

- Any pertinent information from review of existing information on Site 11 (to include Site 4 and Site 13) and removal actions.

- Discrete data from one bedrock borehole location with one or two monitoring well screens installed in the same borehole depending on borehole geophysics based on strike and dip information downgradient of Site 11.

- Downhole geophysics data (caliper, heat-pulse flowmeter, natural gamma, acoustic televiewer, resistivity, and fluid temperature) at each of the couplets to determine bedrock hydraulic characteristics, fracture orientations, and depth for completing bedrock wells by determining monitoring well screen intervals.

MW-308 Area

- Discrete data from three new bedrock well couplets near MW-308 to allow for triangulation in determining hydraulic gradients and VOCs concentrations, including 1,4-dioxane. One couplet is required to address the vicinity of MW-308 (and the team will make a decision as to if and when existing monitoring well MW-308 will be abandoned). The other two couplets will address the vicinity west and north of MW-308 in the bedrock depression area and P-106 areas; a direct-push transect positioned to pass near the vicinity of MW-308 would provide stratigraphic information that would help

position these two couplets (as discussed in Day 1 DQOs DQO Step 3). The LTMP for the Eastern Plume will need to be revised to address monitoring requirements for the new wells; it is anticipated that the well couplets will be monitored at least semi-annually (twice a year) for 2 years for VOCs, including 1,4-dioxane.

- Discrete data from new overburden wells adjacent to and properly sealed off from the bedrock couplets to determine hydraulic gradients, VOC concentrations (including 1,4-dioxane), and lithology using standard geological boring log methods. To properly determine hydraulic gradients, the overburden wells will be screened at the same elevations as each other to bracket the water table. The specific compounds and analytical methods are described in Worksheet #15, developed following the February 2008 DQO meeting.

- Representative bedrock characteristics from rock coring at one location.

- Downhole geophysics data (caliper, heat-pulse flowmeter, natural gamma, acoustic televiewer, resistivity, and fluid temperature) at each of the couplets to determine bedrock hydraulic characteristics, fracture orientations, and depth for completing bedrock wells by determining monitoring well screen intervals.

- Well stabilization parameters to ensure sampling conditions are stable prior to sample collection.

- Although it was originally planned during the February 2008 technical meeting to conduct transducer work at MW-308/MW-309A-B to determine if there is a hydraulic connection with private wells to the east, the duration and rate of pumping at a residence is fairly low, and any effects may be difficult to measure at a fairly distal location; therefore, transducer work is not included in this SAP. However, for any new wells, hydraulic head data will be collected at surveyed Merriconeag Stream stream gauges, and other accessible wells east of Merriconeag Stream to evaluate groundwater flow directions. When the report for the groundwater model is issued, the Aquifer Recovery Test data for MW-308 and MW-309B may be able to provide an initial indication of whether there are any pumping effects influencing groundwater elevations in bedrock near MW-308; these results will be referenced and evaluated as part of this RI.

General Information

- A complete list of potential receptors using private bedrock wells east of Eastern Plume within close proximity of the site (i.e., 1/2 mile) as well as a database and GIS. Among other uses, this information can be used in decision making if Eastern Plume contaminants are detected in any

nearby private wells; however, this task is beyond the scope of the subject UFP-SAP. Inventory and information, if available, will be obtained from the following sources:

- Town Assessor Record Review
- State of Maine Geologic Survey Well records
- Survey to determine use of private well and any other known well information
- Incorporation of previous research of private wells performed by ECC

- Assessment of continued monitoring of bedrock wells as part of LTMP and assessment of groundwater data from newly installed bedrock well couplets to determine whether additional monitoring wells are necessary to define the nature and extent of contamination of bedrock.

DQO Step 4: Define the Boundaries of the Study
(Bedrock Characterization)

Note that the Day 3 meeting was conducted as a technical meeting and not as a formal DQO meeting and so DQO Step 4 was elaborated on following the February 2008 DQO meeting to complete this UFP-SAP.

In addition to bedrock investigation, overburden groundwater will also be investigated, as summarized from the notes of the Day 1 DQO meeting.

The study boundaries are general areas where the bedrock investigation is planned as part of this UFP-SAP. The following areas must be investigated to support the decision statements identified in DQO Step 2:

- The area north and west of MW-308
- The area south of Site 11 and west of existing bedrock well MW-323
- The area in the vicinity of MW-308 and the eastern side of Merriconeag Stream

Vertically, each borehole will be advanced through the overburden and approximately 2 feet into the bedrock. Each borehole will be advanced approximately 50 feet into bedrock. Additionally, at one location, the upper 20 feet of rock will be cored. A rationale table will be prepared justifying each recommended new permanent monitoring well, well location, and well screen interval. Well screens will be 5 or 10 feet in length (recommendations will be made to the project team).

The rationale for locating the new bedrock wells follows:

- MW-EP-308R – Replacement well. The purpose of this well is to obtain vertical hydraulic head and groundwater quality data within two intervals in the upper 50 feet of fractured bedrock. These data

will be used to evaluate whether the upper bedrock is in fact contaminated and if contaminated, obtain data to estimate the vertical extent. MW-308 is the only bedrock well that has contained VOCs and 1,4-dioxane. The contamination may be migrating down the borehole annulus from the overburden into the bedrock because the overburden is not sealed off with a 6-inch steel casing grouted in place. MW-EP-308R will be located approximately 10 feet in the inferred upgradient direction (northeast) of MW-308 to avoid intercepting potential groundwater plume migrating through the borehole annulus downgradient of this well. After grouting steel casing 2 to 3 feet into bedrock, the well will be cored 30 feet to confirm the rock type and characterize bedrock fractures. After the core is collected, the borehole will be reamed out and advanced a total of 50 feet into bedrock, and the open borehole cleaned out. Next, borehole geophysical logging (including caliper, natural gamma, heat pulse flow meter [ambient and stressed] conditions, optical televiewer, temperature and resistivity) will be performed to identify transmissive fractures. The borehole geophysical results will be used to target two discrete vertical intervals for completion of two nested 1-inch inside diameter, Schedule 40 PVC wells.

- MW-EP-340 – New well located approximately 150 feet upgradient (northeast) of MW-EP-308R. The purpose of this well is to evaluate the horizontal and vertical extent of VOCs and migration pathway in an upgradient direction toward a former contaminant source for the Eastern Plume. This well will be constructed the same way as MW-EP-308R so the data is comparable from all three bedrock wells.

- MW-EP-341 – New well located approximately 180 feet north of MW-EP-308R. The purpose of this well is to evaluate the horizontal and vertical extent of VOCs and migration pathway in a potential upgradient direction and in the vicinity of relatively high VOC and 1,4-dioxane overburden groundwater concentrations. This well will be constructed the same way as MW-EP-308R so the data is comparable from all three bedrock wells.

DQO Step 5: Develop the Analytic Approach
(Bedrock Characterization)

The Day 3 meeting was conducted as a technical meeting and not as a formal DQO meeting and so was DQO Step 5 was elaborated on following the February 2008 DQO meeting to complete this UFP-SAP. DQOs were beyond the scope of the technical meeting; but were required to complete this UFP-SAP. Previous DQO outputs were assimilated into decision rules that govern data use:

Decision Rule #1

If the bedrock is contaminated in the vicinity of MW-308, then continue bedrock investigation. If the bedrock is not contaminated, then terminate bedrock investigation. However, if any other new or existing bedrock well is contaminated, then the investigation must be continued.

Data needed to support this decision rule are as follows:

- Lithology and structure (overburden and bedrock)
- Paired upgradient overburden and bedrock wells
- Groundwater VOC concentrations, including 1,4-dioxane
- Vertical and horizontal hydraulic gradients

Decision Rule #2

During the February 2008 technical meeting, it was assumed that concentrations are greatest near the contaminant source and decrease in the downgradient direction. However, this assumption may not be correct because the primary known source of the Eastern Plume, the former fire training area (Site 11), has been remediated through soil removal, and the overburden plume has in large part detached from the source area such that the plume has migrated beyond the limits of the source area. The operation of the GWETS directly over the former source area may have flushed any potential dense non-aqueous phase liquid (DNAPL) or other residual VOCs deeper into the bedrock aquifer than would be typical. In this scenario the highest VOCs concentrations in bedrock may no longer be contiguous with the source area. However, it can be assumed that the concentrations are greatest within the Eastern Plume and decrease in the downgradient direction.

Regardless of the concentrations, however, the contaminants themselves would need to be similar to show a connection between the source and the contamination. If the contaminants in the bedrock are not similar to those in the overburden in the MW-308 area (contaminants are not a result of plume migration from the overburden aquifer), stop investigating contaminant migration pathways to the bedrock near MW-308; otherwise, continue investigation of potential migration pathways.

Data needed to support this decision rule are:

- Lithology and structure (overburden and bedrock)
- Data from paired upgradient overburden and bedrock wells to concurrently evaluate bedrock and overburden north and west of MW-308
- Groundwater VOC concentrations, including 1,4-dioxane
- Vertical and horizontal hydraulic gradients

Decision Rule #3

If the contaminants in the bedrock wells or bedrock well clusters are not present or are present at concentrations that are similar or less then those in the vicinity of Site 11 (using the only data for bedrock

near Site 11 consisting of MW-323 and MW-NASB-11BR), stop investigation of Site 11 bedrock; otherwise, discuss options with the project team.

Data needed to support this decision rule are:

- Lithology and structure (overburden and bedrock)
- Data from paired upgradient overburden and bedrock wells to concurrently evaluate bedrock and overburden north and west of MW-308
- Groundwater VOC concentrations, including 1,4-dioxane
- Vertical and horizontal hydraulic gradients

DQO Step 6: Specify Performance or Acceptance Criteria
(Bedrock Characterization)

Several interrelated qualitative and quantitative factors make calculation of the minimum number of samples and identification of well locations difficult at this time. During the February 2008 technical meeting, it was agreed that the entire team would have an opportunity to review and concur with the proposed locations. This was to be facilitated by preparation of a draft figure(s) showing the proposed locations and distributed to the team prior to finalization of the SAP, which has since been accomplished. The team agreed that the significant addition to the original scope warranted a complete review of the updated version by the regulators and, prior to regulator review, the SAP would also be reviewed anew by the NAVFAC government chemist.

Following the February 2008 DQO meeting, bedrock well locations in the vicinity of MW-308 were provided via e-mail on July 2, 2008 to the project team and discussed in a conference call on July 23, 2008. It was decided during the conference call that the bedrock drilling locations would be recommended to the project team after the results from the pore water task is reviewed and groundwater profiling transects are refined. Historical information from the Sites 4, 11, and 13 source areas will be considered in evaluating results of the bedrock investigation. Rationale for bedrock well installation is included in DQO Step 4.

In accordance with the DQOs established during Day 1, because the data collected from this effort could be used for risk assessment, full validation of the fixed-base laboratory data will occur in accordance with the method-specific criteria for inorganic and organic data review presented in Worksheets 12, 15, and 28 and the logic outlined in Region 1 data validation guidance to apply data qualifiers. The focus will be on higher tier validation of organic data review because any inorganic data are generally being collected only to support remedial designs.

DQO Step 7: Develop the Plan for Obtaining Data
(Bedrock Characterization)

The project team agreed that a statistical design for groundwater well installation is not appropriate because factors such as groundwater flow paths and velocities cannot be addressed with simple statistics. Even if geostatistics was to be used, a significant amount of professional judgment is necessary both during hydrogeologic model development and data interpretation.

The following specifications were developed by the team to help guide the sampling plan development:

- Groundwater profiling results from the 1,4-dioxane study (Day 1 DQOs) will be used to determine permanent well locations. Inputs to the choice of locations will also include the Eastern Plume long-term monitoring data, pore water data, conceptual model for groundwater migration in bedrock, fracture and foliation data, and geophysical and lithologic data.

- Conventional drilling techniques will be used for installation of permanent monitoring wells in overburden and bedrock.

- Bedrock geophysical logging will be conducted at all bedrock well locations. At one select location, rock coring data will additionally be collected for the top 30 feet.

- Lithology data will be collected during drilling activities as a natural part of logging holes.

- Groundwater sampling for VOCs, including 1,4-dioxane.

- Groundwater head data will be collected in new monitoring wells. Stabilized groundwater head data will be converted to elevations to support generation of a bedrock groundwater contour map and to calculated vertical and horizontal hydraulic gradients. Refer to the sampling design of Worksheet #17.

SAP Worksheet #12 -- Measurement Performance Criteria Table Field Quality Control Sample – Volatiles

(UFP-QAPP Manual Section 2.6.2)

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Field Blank	Volatile Organic Compounds (VOCs) (including 1,4-dioxane and 1,1-DCE selective ion monitoring [SIM] analyses)	One per source water	Bias/Contamination	Detections ≤ ½ the quantitation limit (QL). (> QL for common laboratory contaminants).	S&A
Rinsate Blanks	VOCs (including 1,4-dioxane and 1,1-DCE SIM analyses)	One per 20 samples per matrix per days samples are collected	Bias/Contamination	Detections ≤ ½ QL. (>QL for common laboratory contaminants), unless target analytes in field samples are > 10x those in rinsate blank).	S&A
Trip Blanks	VOCs (including 1,4-dioxane and 1,1-DCE SIM analyses)	One per cooler containing VOC samples	Bias/Contamination	Detections ≤ ½ QL. (>QL for common laboratory contaminants), unless target analytes in field samples are > 10x those in trip blank).	S&A
Cooler Temperature Blank	VOCs (including 1,4-dioxane and 1,1-DCE SIM analyses), and TCLP organics and inorganics	One per cooler	Accuracy / Representativeness	Between 2 and 6 ° C	S&A
Field Duplicates	VOCs (including 1,4-dioxane and 1,1-DCE SIM analyses), and TCLP organics and inorganics	One per 20 samples	Precision/ comparability	Soils: RPD ≤ 50% Waters: RPD ≤ 30%. If samples results are < 2x QL, professional judgment is used.	S & A
Data Completeness Check	VOCs (including 1,4-dioxane and 1,1-DCE SIM analyses) and TCLP organics and inorganics	Calculated for the data set	Data Completeness	≥ 95% Overall	S&A
Sensitivity Check	VOCs (including 1,4-dioxane and 1,1-DCE SIM analyses) and TCLP organics and inorganics	Each compound	Sensitivity	QLs less than project action limits listed in Worksheet #15	S&A

Note: TtNUS has reviewed the analytical laboratory QC detection limits and finds them to be acceptable for the project.

SAP Worksheet #13 -- Secondary Data Criteria and Limitations Table

(UFP-SAP 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
April 2007 and September 2007 and April 2008 groundwater monitoring data for 1,4-dioxane	*Draft* SITES 1 AND 3 AND EASTERN PLUME MONITORING EVENT 30 REPORT APRIL 2007 (ECC, August 2007) *Draft* SITES 1 AND 3 AND EASTERN PLUME MONITORING EVENT 31 REPORT SEPTEMBER 2007 (ECC, February 2008) *Draft* EASTERN PLUME MONITORING EVENT 32 REPORT APRIL 2008 (ECC, August 2008)	Originating Organization: ECC Data Types: 1,4-dioxane and other VOC data for groundwater Data Collection Dates: April 2007 Event: 4/20/07 to 4/28/07 September 2007 Event: 09/20/07 to 10/10/07	The data will be used as a baseline for current-day conditions and in determining data gaps. Both analytical data and water-level measurements will be used.	Although the analytical data are fairly recent, resampling is warranted at key existing monitoring wells because 1,4-dioxane migrates readily and concentrations may have changed. It is important to collect synoptic water-level data during each event because variable with season and precipitation events. Water-level data will be recollected.

February through September 2007 groundwater and pore water data for 1,4-dioxane and updated geological cross sections, and revised Site Conceptual Model (CSM)	*Final* SUMMARY REPORT FOR THE MERE BROOK AND MERRICONEAG STREAM FLOODPLAIN AND UPLAND AREA INVESTIGATION, EASTERN PLUME (ECC, July 2008)	Originating Organization: ECC Data Types: 1,4-dioxane and other VOC data for pore water and groundwater, lithology Data Collection Dates: February to September 2007	The data will be used as a baseline for current-day conditions and in determining data gaps. Analytical data and water-level measurements will be used as well as geologic cross sections. The CSM was used in planning the subject supplemental RI and is included herein as Appendix B. The CSM will be used as a baseline.	Although the analytical data are fairly recent, resampling is warranted at key existing monitoring wells because 1,4-dioxane migrates readily and concentrations may have changed. It is important to collect synoptic water-level data during each event because variable with season and precipitation events. Water-level data will be recollected. Existing geological cross sections may require revision because interpretations may need to be updated after results from the subject supplemental RI are evaluated. The CSM will be revised in the future to incorporate results from the subject supplemental RI.

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

DATA MANAGEMENT TASKS

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

Summary of Project Tasks

- Mobilization/demobilization
- Site-specific health and safety training
- Utility clearance
- Grubbing and clearing for site access
- Monitoring equipment calibration
- Pore water sampling (MEDEP and EPA lead)
- Groundwater profiling via temporary wells along transects
- Drilling and installation of permanent 1-inch polyvinyl chloride (PVC) monitoring wells (overburden and bedrock)
- Subsurface soil sampling for lithology
- Rock coring for lithology
- Decontamination pad construction
- Equipment decontamination
- Well development
- Investigation-derived waste (IDW) sampling and management tasks
- Staff gauge/stream piezometer installation
- Collection of groundwater samples
- Field QA/QC management tasks
- Synoptic water-level measurements
- Land surveying
- Field documentation QA/QC and review tasks
- Sample custody and shipment tasks
- Data validation

Analysis Tasks:
Pore Water: Mobile field laboratory chemical analysis of VOCs (except 1,4-dioxane) and fixed-based laboratory analysis for 1,4-dioxane (both by EPA).
Groundwater: Fixed-based laboratory chemical analysis for VOCs, including 1,4-dioxane (by TtNUS).
Field instrumentation for VOC gas analysis.

Quality Control Tasks: QA/QC field samples including duplicates, rinsate blanks, field blanks, and trip blanks. Lab matrix spike/matrix spike duplicate (MS/MSD) samples. Chain-of-custody procedures implemented.

Secondary Data: See Worksheet #13.
Data Management Tasks: Assign data qualifiers, verify sample IDs, run statistical calculations, ensure data are transferred to the project database for safekeeping.
Documentation and Records: Record all field data in logbook and on associated field forms. Data validation reporting of groundwater data. Pore water analytical results from EPA represent screening-level data and so data validation is not warranted.
Assessment/Audit Tasks: Routine audits of field personnel, field procedures, 100 percent data validation for laboratory analytical data.
Data Review Tasks: Data validation, database QA, calculation/data input technical review.

Project Documentation and Records

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

Data Package Deliverables

Sample Collection and Field Measurements Data Package Deliverables

Sample collection data are as required by the sample collection SOPs of Appendix D (TtNUS SOP SA-1.1). In addition, calibration data will also be provided in accordance with the SOPs of Appendix D (EPA SOP S2).

On-Site Analysis Data Package Deliverables

When available, EPA will provide the pore water screening analytical results for VOCs, including 1,4-dioxane, to the team. Otherwise, this project does not involve on-site analyses beyond the field screening associated with well stabilization and personnel protection.

Off-Site Laboratory Data Package Deliverables

Data package deliverable requirements are detailed in the table at the end of this worksheet, entitled Analytical Data Deliverable Elements and Worksheet #30, Analytical Services.

Data packages will require all the elements specified in the table at the end of this worksheet. Data packages will be provided as both hardcopy and portable document format (PDF). All laboratories will provide a Naval Installation Restoration Information Solutions (NIRIS)-compatible electronic data deliverable (EDD). Data packages will be contract laboratory program (CLP)-equivalent (i.e., they will contain CLP-equivalent summary forms and raw data). The standard turnaround time for analytical services is 21 calendar days. Turnaround time will be measured from the laboratory receipt of the last samples in a sample delivery group (SDG). SDGs must contain 20 samples (no more than 20 and only less if the entire sampling event was comprised of less than 20 samples). PDF data will be stored by the analytical laboratory for 5 years.

The NIRIS deliverable does contain the necessary data for MEDEP's EDD (required by 10 MRSA section 9418(1) and 9418(2)(A)) but is not in the required format. Therefore, Tetra Tech will export the NIRIS data into the format required for the MEDEP's EDD.

Data Reporting Formats

Field data will be recorded in field logbooks and on field forms. All logbook and log sheet entries must be made in indelible ink (black pen is preferred). No erasures or liquid paper or white out are permitted. If an incorrect entry is made, the data will be crossed out with a single strike mark, initialed, and dated. The field personnel will sign and date the logbook pages and field forms. Examples of the forms to be used in the field are presented in Appendix E of this SAP.

Data Handling and Management

The data-handling procedures to be followed by the laboratory 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 chain-of-custody forms, sample log forms, sample location maps, and documentation of quality assurance of data manipulation. These forms are included in the applicable SOPs in Appendix D of this SAP.

The overall field data flow is as follows:

1. Field personnel calibrate sampling equipment and document calibrations in accordance with the SOPs in Appendix D (EPA SOP S2).
2. Field personnel use the figures and tables of Worksheets #17 through #22 and surveying results to identify sampling locations.
3. As applicable, sampling locations are evaluated to ensure they are safe for personnel to collect samples and the evaluations are documented in accordance with SOPs in Appendix D and the Health and Safety Plan (HASP) (provided under separate cover).
4. If applicable, sampling conditions are monitored with field instruments to ensure that they are representative of the intended populations. These measurements are made and documented in accordance with SOPs in Appendix D.
5. Samples are collected and documented and sampling equipment is decontaminated in accordance with SOPs in Appendix D.
6. Samples are shipped to the laboratory for analysis, and shipping documentation, including chain-of-custody records, are compiled for later transfer to the Project Manager.
7. Sampling locations are marked for land survey and are later surveyed to establish sample locations within the desired precision and accuracy.
8. Field conditions are recorded throughout field work.
9. During demobilization, field records are collected, double checked for completeness, and forwarded to the TtNUS Project Manager for inclusion in the project database and the appropriate site report.

The overall laboratory data flow is as follows:

1. Samples are received and inspected and the condition of the samples is logged.
2. Analytical equipment is calibrated for analysis and calibrations are documented.
3. Samples are analyzed and the analyses are documented.
4. The resulting data are reviewed and incorporated into the laboratory information management system (LIMS).

5. Hardcopy data packages are assembled and electronic data deliverables (EDDs) are prepared to match the hardcopy data packages.

6. The hardcopy and electronic data are transmitted to TtNUS.

The overall TtNUS data flow is as follows:

1. EDDs and hardcopy data packages are received from the laboratory. These data are reviewed for accuracy and are validated.

2. A validation report is prepared to document the data quality.

3. Data qualifiers are assigned to electronic data and the data are transferred, with qualifiers, to the TtNUS project database.

4. Corresponding field data are transferred to the project database.

5. Data are now available for use by TtNUS project personnel. A data usability assessment is performed to ensure the data will be useful as intended. Data usability is assessed continually as various data users have the opportunity to work with the data.

6. Validated data are made available to the GIS for plotting and other manipulations.

Additional details are provided in the subsections below.

Data Tracking and Control

A "cradle-to-grave" sample tracking system will be used from the beginning to the end of the investigation. Before field mobilization, the FOL will coordinate/initiate the sample tracking process. The Project Manager (PM) will ensure that sample jar labels are printed before field sampling, as necessary. The FOL and PM or PM designee will review the labels for completeness of information and adherence to SAP requirements, as well as for accuracy. The PM will coordinate with the analytical laboratory to ensure that they are aware of the number and type of samples and analyses that are about to be requested.

When field sampling is underway, the FOL will forward the chain-of-custody forms to the TtNUS PM or designee via facsimile at the end of each day. The PM or designee will compare the entries on the chain-of-custody forms with the sample tracking database and enter the sample date and other sample information as appropriate. The PM or designee will also confirm that the chain-of-custody 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 crew is mobilized. After successful completion of all requested analyses, the laboratory will submit an electronic deliverable for every SDG. As EDDs are received from the laboratory, the PM will ensure that the laboratory 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 directly in field notebooks or on sample logs. 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 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.

Before electronic files are received from the laboratory, all sample-specific information will be entered into the data management system. The sample information file will allow the analytical results to be grouped together properly for statistical purposes. The data will be managed in one data structure. For field data, the FOL will coordinate with the GIS lead to ensure that all survey technical specifications are consistent with the underlying coordinate system in the GIS.

Electronic data arriving from the laboratory 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 laboratory subcontractor will submit a hard copy and a PDF file of the analytical data packages, as well as EDDs. The electronic database will be checked against the hard copy/PDF file provided by the laboratory and updated as required, based on data qualifier flags applied during the data validation process. The data generated during the implementation of this SAP will be incorporated into the project 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 this project. The relational database stores chemical, geological, hydrogeological, 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.

DATA REVIEW ELEMENTS

Data review is the process by which data are examined and evaluated by various personnel at various levels of detail. This process includes data verification, data validation, and data usability assessment. Data verification is a process of evaluating the completeness, correctness, and contractual compliance of a data set against the method standard, SOP, or contract requirements documented in this SAP. Data validation is an analyte- and sample-specific process that extends the qualification of data beyond data verification to determine the quality of the specific data set relative to analytical QC requirements. Data usability assessment extends these other review processes to examine the data in the context of the project objectives to determine whether the data are suitable for supporting the attainment of these objectives.

The internal data verification requirements for this project include the maintenance and periodic review of field documentation (site logbooks, instrument calibration logs, chain-of-custody forms, field summary reports, and field modification records) and laboratory analytical data packages.

Data validation is a systematic review of the analytical data package with respect to sample receipt and handling, compliance with required analytical methods, data reporting and deliverables, and document control. A qualified chemist will review the analytical data packages using EPA procedures.

Usability assessment will be performed by project-level personnel who understand the intended use of the data. These personnel include but are not necessarily limited to project chemists, geologists, and risk assessors.

Data Review Steps

Upon receipt of analytical laboratory results, TtNUS database personnel will evaluate electronic and hardcopy deliverables for conformance to data format specifications and for completeness. TtNUS validation chemists also perform a completeness verification and will then perform data validation according to the most current data validation guidelines to ensure that the analytical results meet the DQOs for data usability. The validation guidance documents that will be used include:

- Region 1 Inorganic Data Validation Functional Guidelines (February 1989)
- Region 1 Organic Data Validation Functional Guidelines (Volatile/Semivolatile Part 2) (December 1996), to the extent practicable.
- Region 1 Organic Data Validation Functional Guidelines [Pesticide/PCB part 3] (February 2004)
- DoD QSM (January 2006).
- Test Methods for Evaluating Solid Waste; Physical/Chemical Methods (SW-846), 3rd edition, up to and including Update III. Office of Solid Waste and Emergency Response, Washington, DC. 1986.

The TtNUS DVM stays current with available guidelines to ensure that the appropriate validation guidelines are used.

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 QC 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. 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 requirements described above. 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.

Step I: Verification

Verification includes field data verification and laboratory data verification. Verification inputs as per Worksheet #34 will be checked.

Step II: Validation

Validation of field measurements and laboratory analytical data is discussed in this section. Validation of field data will be limited to real-time checks in the field as data are generated, whereas laboratory analytical data will be validated in accordance with current applicable EPA guidance.

Step IIa: Validation Activities

One hundred percent of the laboratory data from chemical analyses conducted by TtNUS will be validated (screening-level pore water data from EPA will not be validated). Validation of analytical data will be conducted by the TtNUS or data validation subcontractor. Groundwater data (VOCs including 1,4-dioxane) will undergo Tier III data validation, investigation-derived waste (IDW) data to determine disposal requirements will undergo Tier I data validation, and soil data (TOC) will undergo Tier I data validation. Final review and approval of validation deliverables will be completed by the DVM. All parameters will be reviewed using applicable sections of the aforementioned guidelines and laboratory SOPs. Each analysis will be validated in accordance with Worksheet #36.

As part of the validation process, the validator will check that the laboratory has provided all the documentation required to support the reported analytical results. If any documentation is missing from the data package, the data validator will contact the laboratory to request a resubmittal. If the laboratory fails to resubmit the requested information, the data validator will note this on the data validation cover letter. The usability of such data will then be determined by the PM and the Navy.

Data validation will be completed to ensure that the data are of evidentiary quality. Particular emphasis will be placed on holding time compliance, equipment calibration, spike recoveries, and blank results although all required elements of the validation process will be considered.

Step IIb: Validation Activities

Worksheets #35 and #36 summarizes the Step IIb data validation activities.

Step III: Usability Assessment

After completion of data validation and an overall review of data quality indicators (DQIs), the data will be reconciled with Measurement Performance Criteria (MPC) to determine whether sufficient data of acceptable quality are available for decision making. A series of inspections and analyses will be performed to estimate several of the data set characteristics. The evaluations will include simple summary statistics for target analytes, such as maximum and minimum concentrations, frequency of samples exhibiting detectable analytes, and proportion of samples with elevated results. Worksheet #37 summarizes the Step III Usability Assessment. The data will be presented in a tabular format. These inspections and statistical analyses will be designed to:

- Identify deviations, if any, from the field sampling SOPs.
- Identify deviations, if any, from the laboratory analytical methods.

- Identify deviations, if any, from the SAP.
- Identify deviations, if any, from the data validation process.
- Evaluate effects of the above-listed deviations from planned procedures and processes on the interpretation and utility of the data.
- Identify elevated detection limits and explain their impacts on the attainment of project objectives.
- Identify unusable data (i.e., data qualified as "R").
- Evaluate project assumptions.
- Identify unanticipated data set characteristics such as a laboratory variance greater than the sampling variance (i.e., ANOVA, *t*-test) if enough data are available.
- Identify and evaluate potential data outliers (95 percent confidence goodness-of-fit test on probability plot data). The plotted data will be transformed, if necessary, depending on the observed distribution.
- Evaluate adherence to investigation objectives and decision rules.
- Ensure completion of corrective actions.
- Identify the existence of remaining data gaps.

For statistical comparisons and mathematical manipulations, non-detect values will be represented by a concentration equal to one-half the sample-specific reporting limit. Duplicate results (original and duplicate) will not be averaged for the purpose of representing the range of concentrations. However, the average of the original and duplicate will be used to represent the concentration at that sample location.

Statistical tests for outliers will be conducted using standard statistical techniques appropriate for this task. Potential outliers will be removed if a review of field and laboratory documents indicates that the results are true outliers. If no physical cause for a statistical outlier can be identified, the data point will not be removed from the data set. However, if the data point is found to truly represent a physical quantity that is different from the rest of the data set, it will be removed.

The suitability of any given statistical test will be assessed based on the completeness of the data sets and the conditions observed at the site. For example, when a single data value is available for soils or water samples at a given sampling location, statistical tests cannot be conducted for that individual sampling location. However, pooling of data across sampling locations may be possible and, if logical to do so, may be implemented at the discretion of the TtNUS PM. Statistical testing will generally be conducted at the five percent significance level. Statistical testing at other significance levels may also be warranted to provide perspective on the results of testing at 5 percent significance. If other significance levels are used, they will be supported with rationales for their use and must be reviewed/approved by MEDEP.

Data Limitations and Actions from Usability Assessment

After all data evaluations are completed, any limitations on the use of data will be known and will be considered during decision making. If necessary, investigation objectives may be revised in anticipation of additional data collection to meet PQOs for the site.

Because analytical performance is generally similar for groundwater and surface water and for soils and sediments, two matrix groups (solids and aqueous) may be used when discussing bias and precision. These groupings also cause the number of samples within a group to be greater than when the matrices are discussed separately. The increased number of samples in a group better supports statistical analyses of the data.

Precision

Field and laboratory precision indicators will be reviewed. In general, laboratory precision should be greater than field precision because laboratory replicate samples have had an additional opportunity to be homogenized beyond the homogenization applied to field samples. If laboratory precision is determined to be less than field precision, and the difference is judged to be significant, the cause of the difference will be investigated and an appropriate corrective action will be devised. In general, Relative Percent Difference (RPD) values from laboratory and field duplicates (Worksheet #28 and #14, respectively) will be used for this evaluation.

Accuracy/Bias

Overall contamination (both field and laboratory) will be reviewed and assessed for impact to the project. In general, this will be reflected in the assignment of data validation qualifiers that have accounted for the effect of blank contamination.

Laboratory Control Sample (LCS), Laboratory Control Sample Duplicate (LCSD), MS, and MSD percent recovery results will also be used to assess the degree of bias to be expected for various analyte groups and, to the extent that it is warranted, for individual analytes. Whether review and discussion to the analyte level is warranted depends on which analytes exhibit unacceptable bias and whether they are of importance to this project. In general, MS and MSD data will be used as a more accurate indication of bias in site samples than LCS and LCSD data.

If enough data are available and multiple field events are conducted, precision may be compared for the multiple field events at the discretion of the TtNUS PM. The usability assessment will strive to indicate limitations of the data caused by positive or negative bias and will attempt to do so based on logical groupings such as individual or multiple SDGs, matrices, etc.

Representativeness

Representativeness (see Worksheet #37) will be evaluated by reviewing field logs and analytical results to verify that project requirements were satisfied for these operations. Special emphasis will be placed on determining whether all intended samples were collected from the intended locations. If they were not, an assessment on the attainment of project objectives will be made. Based on these assessments, a qualitative value judgment will be made concerning the representativeness of the samples and associated data. Representativness may also be assessed during the evaluation of precision and accuracy and will be summarized in the precision and accuracy assessments as applicable. An inclusion of representativeness of data for risk assessment may also be made when, for example, chemicals may be partitioned between various physical phases of a sample such as the dissolved and suspended solids of a groundwater or surface water sample.

Comparability

Comparability will be assessed by first ensuring that SAP requirements were satisfied. In addition, the comparability of data from one sample group to another may be assessed if the precision, accuracy or sensitivity indicate that the data are not or may not be comparable in terms of those quality indicators. If multiple sampling rounds are undertaken, the comparability of multiple sets of data for the same chemical parameters and matrices will be assessed in terms of precision, accuracy, sensitivity, and representativeness.

In the case of upgradient data, comparability of the upgradient and site data sets will be described in terms of their representativeness for the intended populations, and any other factors such as precision, accuracy, and sensitivity of analytical results that either support or inhibit comparisons of the two types of data.

Sensitivity and Quantitation Limits
The range of non-detect values, which represents the sensitivity of the analytical measurements, will be compared to SAP target detection and QLs. This comparison will identify if actual sensitivity was sufficient to meet the SAP targets. If the targets were not met, a discussion will be presented in the final report to establish the impact of not meeting the targets on the attainment of project objectives.

Completeness
Completeness will be evaluated on the following levels:

- Number of samples collected compared to number of samples scheduled for collection, by matrix
- Number of valid results compared to number of planned results, by matrix, by analytical group

In addition, a tally of the rejected values and reasons for rejection will be presented. Where rejected data are identified, the impact on attainment of project objectives will be described.

Special emphasis will be placed on discussing completeness deficiencies that have the greatest impact on the project. Completeness deficiencies that are inconsequential to the attainment of project objectives will be identified but will receive little discussion.

Activities
Several activities to be undertaken by the project team to assess data usability are described above. When the usability assessment is completed the project team will be able to:

- Establish whether intended samples were collected for each matrix and whether the samples and associated data are of sufficient quality to support the attainment of project objectives.
- Identify the impact of the missing data on the project if data for any scheduled samples are not available (e.g., because the samples were not analyzed or the data were rejected for use).
- Identify all Precision, Accuracy, Representativeness, Completeness, and Comparability (PARCC) parameters that were not satisfied and in which samples, matrices, analytical groups, etc., they were not satisfied.
- Identify data use restrictions for any data that did not meet PQOs or otherwise are insufficient to support the attainment of project objectives.
- Identify corrective actions necessary to recover from missing data or data of insufficient quality.

STREAMLINING DATA REVIEW

Steamlining data review is not an applicable task for this project. Full 100 percent data validation is planned.

DATA PACKAGE ELEMENTS	VOCs	1,4-dioxane	TCLP Metals	TCLP Organic
♦ **NARRATIVE (Org. Narrative, Inorg. Cover Page)**	X	X	X	X
♦ **SHIPPING/RECEIVING DOCUMENTS AND INTERNAL LABORATORY COC RECORDS:**				
- Airbills	X	X	X	X
- Chain-of-Custody Records/Forms (Traffic Report)	X	X	X	X
- Sample Log-In Sheet (Org. and Inorg. DC-1 Form)	X	X	X	X
- Miscellaneous Shipping/Receiving Records	X	X	X	X
- Internal Lab. Sample Transfer Records and Tracking Sheets	X	X	X	X
♦ **SAMPLE DATA:**				
- Tabulated Summary Form for Field Sample, Method Blanks, and PE Sample Results (Org. and Inorg. Form I)	X	X	X	X
- Tentatively Identified Compounds Tabulate Summary Form (Org. Form I TIC)				
- Reconstructed Total Ion Chromatogram (RIC) for each sample	X	X		X
- Raw spectra of target compound and background subtracted spectrum of target compound for each sample	X	X		X
- Mass spectra of all reported TICs/three best library matches for each sample				
- Chromatograms from both columns for each sample				
- Pesticide/PCB Identification Tabulated Summary Form (Org. Form X)				
- For Pest/PCB or Dioxin/Furan results confirmed by GC/MS, copies of raw spectra and background subtracted spectrum of target compounds				
- GPC sample chromatograms (if necessary)		X		X
- Sample preparation/extraction/digestion log (Inorg. Form XIII) and logbook pages	X	X	X	X
- Sample analysis run log (Inorg. Form XIV) and logbook pages	X	X	X	X
- ICP Raw Data			X	
- Furnace AA Raw Data				
- Mercury Raw Data			X	
- Cyanide Raw Data				
- Other Analytical Raw Data	X	X	X	X
♦ **STANDARDS DATA:**				
- Method Detection Limit Study Tabulated Summary Form	X	X	X	X
- Initial Calibration Tabulated Summary Form (Org. Form VI, Inorg. Form IIA)	X	X	X	X
- Continuing Calibration Tabulated Summary Form (Org. Form VII, Inorg. Form IIA)	X	X	X	X

DATA PACKAGE ELEMENTS	VOCs	1,4-dioxane	TCLP Metals	TCLP Organic
- RICs and quantitation reports for all GC/MS Standards	X	X		X
- Pesticides Analyte Resolution tabulated Summary Form (Org. Form VI, Pest-4)				X
- Pesticides Calibration Verification Tabulated Summary Form (Org. Form VII, Pest-1 and Pest-2)				X
- Analytical Sequence Tabulated Summary Form (e.g. Org. Form VIII-Pest)				X
- GC Chromatograms and data system printouts for all GC standards				X
- For Pesticides/Aroclors confirmed by GC/MS, copies of spectra for standards data				
- GPC Calibration Tabulated Summary Form (Org. Form IX, Pest-2)				
- Florisil Cartridge Check Tabulated Summary Form (Org. Form IX, Pest-1)				
- Instrument Detection Limits Tabulated Summary Form (Inorg. Form X)			X	
- ICP Interelement Correction Factors Tabulated Summary Form (Inorg. Form XIA and XIB)			X	
- ICP Linear Ranges Tabulated Summary Form (Inorg. Form XII)			X	
- CRDL Standards for AA and ICP Tabulated Summary Form (Inorg. Form IIB)			X	
- Standards preparation logbook pages	X	X	X	X
♦ **QC DATA:**				
- Tuning and Mass Calibration Tabulated Summary Form (Org. Form V)	X	X		X
- Window defining mixture				
- Chromatographic resolution				
- Surrogate Percent Recovery Tabulated Summary Form (Org. Form II)	X	X		X
- MS/MSD Recovery Tabulated Summary Form (Org. Form III)	X	X		X
- Method Blank Tabulated Summary Form (Org. Form IV and Inorg. Form III)	X	X	X	X
- Internal Standard Area and retention time (RT) Tabulated Summary Form (Org. Form VIII)	X	X		X
- Labeled Compound Recovery Summary Form				
- QC Raw Data – RICs, Chromatograms, Quan Reports, Integration Reports, Mass Spectra, etc.	X	X		X
- Spike Sample Recovery Tabulated Summary Form (Inorg. Form IV)			X	
- Duplicates Tabulated Summary Form (Inorg. Form VI)			X	
- Internal Laboratory Control Sample Tabulated Summary Form (Org Form III and Inorg. Form VII)		X	X	X
- Standard Addition Results Tabulated Summary Form (Inorg. Form VIII)			X	
- ICP Interference Check Sample Tabulated Summary Form (Inorg. Form IV)			X	
- Post Digestion Spike Tabulated Summary Form (Inorg. Form VB)			X	

DATA PACKAGE ELEMENTS	VOCs	1,4-dioxane	TCLP Metals	TCLP Organic
- ICP Serial Dilutions Tabulated Summary Form (Inorg. Form IV)			X	
- QC Raw Data – ICP, Furnace, Mercury computer printouts, etc.			X	
- QC sample preparation logbook pages	X	X	X	X
- Toxicity Equivalence Factor				
- Compound Identification Criteria				X
♦ **MISCELLANEOUS DATA:**				
- Original preparation and analysis forms or copies of preparation and analysis logbook pages	X	X	X	X
- Screening records	X	X	X	X
- All instrument output, including strip charts from screening activities	X	X	X	X
- Instrument stability				
- Preparation Logs Raw Data	X	X	X	X
- Percent Solids Determination Log	X	X	X	X
- Other Records (ex. Telephone Communication Log)	X	X	X	X

(1) Miscellaneous data package will include information as applicable to the method.
VOC - Volatile organic compounds.
TCLP – Toxicity Characteristic Leaching Procedure.

SAP Worksheet #15 -- Reference Limits and Evaluation Table

(UFP-SAP Manual Section 2.8.1)

Matrix: Groundwater
Analytical Group: TCL Chlorinated VOCs Method 8260 (Chlorinated Aliphatic Solvents Detected in Eastern Plume)

Analyte [1]	CAS Number	Project Action Limit [2,3] (µg/L)	Project Action Limit Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-specific	
					Quantitation Limit (µg/L)	Method Detection Limit (µg/L)
1,1- DICHLOROETHANE	75-34-3	70	MEG	1.0	1.0	0.18
1,1,1-TRICHLOROETHANE	71-55-6	200	MEG	1.0	1.0	0.34
1,1,2- TRICHLOROETHANE	79-00-5	5	MCL	1.0	1.0	0.39
1,1-DICHLOROETHENE	75-35-4	0.6	MEG	0.20	0.20	0.06 (SIM)
1,2- DICHLOROETHANE	107-06-2	4	MEG	1.0	1.0	0.12
CHLOROFORM	67-66-3	70	MEG	1.0	1.0	0.36
CIS-1,2- DICHLOROETHENE	156-59-2	70	MEG	1.0	1.0	0.38
METHYLENE CHLORIDE	75-09-2	5	MCL	2.0	2.0	0.51
TETRACHLOROETHENE	127-18-4	5	MCL	1.0	1.0	0.36
TRANS-1,2- DICHLOROETHENE	156-60-5	100	MCL	1.0	1.0	0.41
TRICHLOROETHENE	79-01-6	5	MCL	1.0	1.0	0.31
VINYL CHLORIDE	75-01-4	1	[3]	1	1	0.35

Matrix: Groundwater
Analytical Group: TCL VOCs Method 8260, 1,4-Dioxane

Analyte [1]	CAS Number	Project Action Limit [2,4] (µg/L)	Project Action Limit Reference	Project Quantitation Limit Goal (µg/L)	Laboratory-specific	
					Quantitation Limit (µg/L)	Method Detection Limit (µg/L)
1,4-DIOXANE	123-91-1	6.1	RSL	2.0	2.0	0.35 (SIM)

Matrix: IDW
Analytical Group: TCLP Regulatory List Organics and Inorganics

Analyte	CAS Number	Project Action Limit (mg/L)	Project Action Limit Reference	Project Quantitation Limit Goal (mg/L)	Laboratory-specific	
					Quantitation Limit	Method Detection Limit
BENZENE	71-43-2	0.5	REG	0.1	100 µg/L	0.37 µg/L
CARBON TETRACHLORIDE	56-23-5	0.5	REG	0.1	100 µg/L	0.39 µg/L
CHLOROBENZENE	108-90-7	100	REG	5.0	5000 µg/L	0.28 µg/L
CHLOROFORM	67-66-3	6	REG	2.0	2000 µg/L	0.36 µg/L
1,2-DICHLOROETHANE	107-06-2	0.5	REG	0.1	100 µg/L	0.12 µg/L
1,1-DICHLOROETHENE	75-35-4	0.7	REG	0.1	100 µg/L	0.21 µg/L
2-BUTANONE	78-93-3	200	REG	0.5	500 µg/L	0.79 µg/L
TETRACHLOROETHENE	127-18-4	0.7	REG	0.1	100 µg/L	1.0 µg/L
TRICHLOROETHENE	79-01-6	0.5	REG	0.1	100 µg/L	1.0 µg/L
VINYL CHLORIDE	75-01-4	0.2	REG	0.040	40 µg/L	2.0 µg/L
O-CRESOL	95-48-7	200	REG	0.5	500 µg/L	0.36 µg/L
M-CRESOL	95-48-7	200	REG	0.5	500 µg/L	0.36 µg/L
P-CRESOL	106-44-5	200	REG	0.5	500 µg/L	0.36 µg/L
1,4-DICHLOROBENZENE	106-46-7	7.5	REG	0.2	200 µg/L	0.62 µg/L
2,4-DINITROTOLUENE	121-14-2	0.13	REG	0.020	20 µg/L	0.34 µg/L

Analyte	CAS Number	Project Action Limit (mg/L)	Project Action Limit Reference	Project Quantitation Limit Goal (mg/L)	Laboratory-specific	
					Quantitation Limit	Method Detection Limit
HEXACHLOROBENZENE	118-74-1	0.13	REG	0.020	20 µg/L	0.36 µg/L
HEXACHLORO-1,3-BUTADIENE	87-68-3	0.5	REG	0.1	100 µg/L	0.64 µg/L
HEXACHLOROETHANE	67-72-1	3	REG	0.2	200 µg/L	0.64 µg/L
NITROBENZENE	98-95-3	2	REG	0.2	200 µg/L	0.43 µg/L
PENTACHLOROPHENOL	87-86-5	100	REG	0.5	500 µg/L	0.53 µg/L
PYRIDINE	110-86-1	5	REG	0.2	200 µg/L	0.93 µg/L
2,4,5-TRICHLOROPHENOL	95-95-4	400	REG	0.5	500 µg/L	0.43 µg/L
2,4,6-TRICHLOROPHENOL	88-06-2	2	REG	0.2	200 µg/L	0.22 µg/L
CHLORDANE	57-74-9	0.03	REG	0.001	1.0 µg/L	0.012 µg/L
ENDRIN	72-20-8	0.02	REG	0.001	1.0 µg/L	0.0062 µg/L
HEPTACHLOR	76-44-8	0.008	REG	0.001	1.0 µg/L	0.0089 µg/L
HEPTACHLOR EPOXIDE	1024-57-3	0.008	REG	0.001	1.0 µg/L	0.0083 µg/L
LINDANE	58-89-9	0.4	REG	0.050	50 µg/L	0.007 µg/L
METHOXYCHLOR	72-43-5	10	REG	0.5	500 µg/L	0.0079 µg/L
TOXAPHENE	8001-35-2	0.5	REG	0.1	100 µg/L	0.012 µg/L
2,4-D	94-75-7	10	REG	0.5	500 µg/L	0.75 µg/L
2,4,5-TP (SILVEX)	93-72-1	1	REG	0.1	100 µg/L	0.274 µg/L

Analyte	CAS Number	Project Action Limit (mg/L)	Project Action Limit Reference	Project Quantitation Limit Goal (mg/L)	Laboratory-specific	
					Quantitation Limit	Method Detection Limit
ARSENIC	7440-38-2	5	REG	1.0	1.0 mg/L	0.0087mg/L
BARIUM	7440-39-3	100	REG	20	20 mg/L	0.0065 mg/L
CADMIUM	7440-43-9	1	REG	0.2	0.2 mg/L	0.0054 mg/L
CHROMIUM	7440-47-3	5	REG	1.0	1.0 mg/L	0.0063 mg/L
LEAD	7439-92-1	5	REG	1.0	1.0 mg/L	0.010 mg/L
MERCURY	7439-97-6	0.2	REG	0.05	0.05 mg/L	0.047 µg/L
SELENIUM	7782-49-2	1	REG	0.2	0.2 mg/L	0.022 mg/L
SILVER	7440-22-4	5	REG	1.0	1.0 mg/L	0.0035 mg/L

RSL = Regional Screening Level, Update to Oak Ridge National Laboratory Regional Screening Criteria (EPA, July 7, 2008 and adopted by EPA Regions 3, 6, and 9).
MCL = Maximum Contaminant Level (EPA, 2004 Edition of the Drinking Water Standards and Health Advisories).
MEG = Maine CDC Maximum Exposure Guidelines (MEG) for Drinking Water (September 2007).
NA = Not available.
REG = The project action limit is the TCLP regulatory level.

1 Analytes included are chlorinated aliphatic solvent compounds and 1,4-dioxane previously detected in the Eastern Plume as part of the Long-Term Monitoring Program.
2 With the exception of 1,4-dioxane and vinyl chloride, the more conservative of the MEG and MCL was selected as the project action limit. The purpose of the investigation is further definition of the plume for remediation purposes rather than risk assessment, and so lower values will be achieved by the analytical laboratory only if readily attainable. The project limits shown in this table are acceptable to EPA and MEDEP for this project.
3 For vinyl chloride, the Maine MEG of 0.2 ug/L will not be achieved. MEDEP, in a conference call with EPA and the Navy on July 23, 2008, determined that a project action limit of 1 ug/L is acceptably low for this analyte. Of note, the EPA MCL of 2 ug/L will be achieved. Any vinyl chloride detections will be reported to the method detection limit.
4 For 1,4-dioxane the Maine MEG of 32 µg/L will be utilized for remedial action. Defining the extent of the 1,4-dioxane plume that exceeds the Maine MEG of 32 µg/L will help focus evaluation of remedial options, but understanding the migration routes will require defining the extent of the plume at lower concentrations than the MEG. Therefore, the more conservative EPA RSL for tap water of 6.1 ug/L is shown on the table.

Note: Method detection limit (MDL) is the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix type containing the analyte.

SAP Worksheet #16 -- Project Schedule / Timeline Table (optional format)

(UFP-SAP Manual Section 2.8.2)

The following is the anticipated schedule for the 1,4 Dioxane and Bedrock Groundwater Assessment:

Activities	Organization	Dates (MM/DD/YY)		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
Discussion with MEDEP/EPA on "Pre-RI SAP summary"	Navy/EPA/MEDEP	07/16/08	07/16/08	---	---
MEDEP/EPA suggestions for Sampling and Analysis Program	Navy/EPA/MEDEP	07/16/08	07/23/08	---	---
Draft RI SAP to Navy (Navy project team/Navy chemist)	TtNUS	07/24/08	08/12/08	Draft RI SAP	08/12/08
EPA/MEDEP initiate pore water sampling	EPA/MEDEP	08/12/08	08/14/08	---	---
Navy comments due	Navy	08/12/08	08/26/08	---	---
Navy comments resolved	Navy/TtNUS	08/27/08	09/03/08	---	---
Draft Final RI SAP to EPA/MEDEP	TtNUS	09/04/08	09/08/08	Draft Final RI SAP	09/08/08
EPA/MEDEP comments due	EPA/MEDEP	09/08/08	09/25/08	Comment Letter	09/25/08
Resolution of Comments from MEDEP/EPA	EPA/MEDEP/Navy/TtNUS	09/26/08	10/02/08	---	---
Final RI SAP issued	TtNUS	10/03/08	10/13/08	Final RI SAP	10/08/08
Sites 4, 11, and 13 Source Summary Tech Memo	TtNUS	09/09/08	10/31/08	Tech Memo	09/30/08
Fall RI Field work (groundwater profiling and bedrock)	TtNUS	10/20/08	11/26/08	---	---
Raw Data provided to EPA/MEDEP (1)	TtNUS	12/31/08	01/27/09	Raw Data Package	01/20/09

Activities	Organization	Dates (MM/DD/YY)		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
Rough Draft RI Report to Navy	TtNUS	12/01/08	02/23/09	Rough Draft RI Report	02/23/09
Navy comments due	Navy	02/24/09	03/05/09	---	---
Navy comments resolved	Navy/TtNUS	03/06/09	03/13/09	---	---
Draft RI Report to EPA/MEDEP	TtNUS	03/16/09	03/25/09	Draft RI Report	03/25/09
EPA/MEDEP Comments Due on Draft Report	EPA/MEDEP	03/25/09	05/08/09	Comment Letter	05/08/09
Draft Final RI Report Issued	TtNUS	05/11/09	06/25/09	Draft Final RI Report	06/25/09
Draft Tech Memo (Permanent Wells) Work Plan to EPA/MEDEP	TtNUS	05/11/09	07/01/09	Draft Tech Memo Work Plan	07/01/09
EPA/MEDEP Comments on Draft Final RI Report	EPA/MEDEP	06/25/09	07/24/09	Comment Letter	07/24/09
Comments Due on Draft Tech Memo Work Plan	EPA/MEDEP	07/02/09	07/31/09	Comment Letter	07/31/09
Final RI Report Issued	TtNUS	07/27/09	08/15/09	Final RI Report	08/15/09
Final Tech Memo Work Plan to EPA/MEDEP	TtNUS	08/03/09	09/10/09	Final Tech Memo work Plan	09/10/09
Spring RI Field Work (permanent wells)	TtNUS	09/24/09	11/06/09	---	---
Raw data from Permanent Wells to EPA/MEDEP (1)	TtNUS	01/24/10	01/24/10	Raw Data Package	01/24/10
Rough Draft Tech Memo Report to Navy	TtNUS	11/07/09	02/22/10	Rough Draft Tech Memo Report	02/22/10
Navy comments due	Navy	02/22/10	03/05/10	---	---

Activities	Organization	Dates (MM/DD/YY)		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
Navy comments resolved	Navy/TtNUS	03/06/10	03/15/10	---	---
Draft Tech Memo Report to EPA/MEDEP	TtNUS	03/16/10	03/25/10	Draft Tech Memo Report	03/25/10
EPA/MEDEP Comments Due	EPA/MEDEP	03/25/10	05/10/10	Comment Letter	05/10/10
EPA/MEDEP Comments Resolved	EPA/MEDEP/Navy/TtNUS	05/11/10	06/11/10	---	---
Final Tech Memo Report	TtNUS	06/12/10	06/30/10	Final Tech Memo Report	06/30/10
Meeting to discuss design concepts for 1,4-dioxane Treatment	EPA/MEDEP/Navy/TtNUS	05/25/10	05/25/10	---	---
Proposed path Forward for Design/FS	EPA/MEDEP/Navy/TtNUS	06/15/10	06/15/10	---	---

1. The raw data package provided to MEDEP and EPA will be provided electronically in the MEDEP's Environmental Data Deliverable (EDD) format for the Environmental and Groundwater Database (EGAD). The contents of the data package will include field parameter data, water level data, and laboratory analytical data. Specific EDD formats and additional information can be found on the MEDEP's EGAD web page at http://www.maine.gov/dep/rwm/egad/.

SAP Worksheet #17 -- Sampling Design and Rationale

(UFP-QAPP Manual Section 3.1.1)

Sampling Design and Rationale

The sampling design is a multi-task approach, in which the information from initial tasks is used to guide sample collection in later tasks. Groundwater is the only medium of concern. 1,4-Dioxane and other volatile organic compounds (VOCs) are the contaminants of concern (COCs). Additionally, available contaminant source and hydrogeologic information for Sites 4, 11, and 13 will be summarized to evaluate if there is a connection with the 1,4-dioxane and VOC contamination present in the Eastern Plume; this information will be summarized and provided to the Project Team prior to proceeding with the Task 3 bedrock investigation.

Based on available data, the direction of bulk groundwater flow is generally to the southeast except in the vicinity of Merriconeag Stream and Mere Brook, where the groundwater flow direction may shift so that is perpendicular to the drainage channel (see Figure 17-1). Low to moderate concentrations are anticipated based on historical data. (See Worksheet #11 for supporting information). In particular, it appears the 1,4-dioxane is migrating through fine to medium sand lenses within the Transition Unit; however, the concentration distribution and migration pathways on both sides of Mere Brook are currently unknown. Furthermore, buried coarser stream lenses may act as preferential contaminant migration pathways. The COCs are not naturally occurring or anthropogenic; therefore no samples will be collected to determine background conditions.

Tasks 1 through 3 will be performed in the fall of 2008; Tasks 4 through 5 will be performed in the spring of 2009. The U.S. Environmental Protection Agency (EPA) and Maine Department of Environmental Protection (MEDEP) are responsible for implementing Task 1; the Navy is responsible for implementing Tasks 2 through 5. The results of Tasks 1 and 2 will be distributed among the project team for review and discussion on optimizing sample locations in subsequent tasks. Refer to Figure 17-1 for preliminary groundwater profiling locations; refer to Figure 17-2 for preliminary bedrock well locations and pore water sample locations. See Worksheet #18 for sampling location rationale.

Task 1 – Pore Water Sampling

This task consists of pore water sampling for 1,4-dioxane, and other VOC analysis. Real-time VOC data will be used to help define potential groundwater contaminant discharge areas. MEDEP and EPA are responsible for the planning document for this task, as well as the sampling and analysis. Tetra Tech provided the mapping (Figure 17-2 with PW-82 provided for reference to start taping PW-83, PW-84, etc.); Tetra Tech/Navy will also have a Trimble GPS with the locations preloaded to navigate to if that is found to be more expeditious with the taping approach as the backup. EPA will analyze pore water samples for select chlorinated VOCs using their mobile laboratory and 1,4-dioxane using their fixed-base laboratory in Chelmsford, Massachusetts. The pore water sampling results will be provided to the project team for review. The pore water results will be used to identify potential contamination migration pathways to Merriconeag Stream and results will be considered for refining groundwater profiling transect locations.

Task 2 – Groundwater Profiling

This task includes brush clearing and utility clearance and groundwater profiling. The proposed profiling point locations will be staked and flagged in the field using a Global Positioning System (GPS). Brush/tree cutting will be required to access the drilling locations. Utility clearances will be obtained before drilling. Direct-push technology (DPT) drilling and brush clearing will be subcontracted by TtNUS.

A total of 30 temporary monitoring wells of nominal 1-inch inside diameter (ID) (using a Geoprobe groundwater profiler) will be used to collect discrete-interval groundwater samples at each of the 30 locations. Refer to Figure 17-1 for 22 preliminary profiling locations; 8 contingency profiling locations are to be determined based on pore water (Figure 17-2) results. Also, the transect locations may be modified based on the pore water results. The purpose of the profiling is to better define 1,4-dioxane concentrations exceeding the MEDEP criterion of 32 ug/L in the Upper Sand Unit, Lower Sand Unit, and in one additional relatively thick sand unit that may be present within the Transition Unit. Electrical conductivity (EC) profiling, using a DPT rig, will be performed initially to identify the discrete intervals for sampling (i.e., Upper Sand Unit, Lower Sand Unit, and sand unit within the Transition Unit). Discrete-interval groundwater samples will be analyzed for 1,4-dioxane and other select VOCs (see Worksheet #15); these results will be used, together with available information, to locate up to 25 permanent overburden monitoring wells.

Also, TOC samples will be collected from select uncontaminated soil areas (recommendations will be made to the project team) to estimate aquifer conditions and to estimate the potential for natural degradation.

Task 3 - Bedrock Investigation near MW-308

This task consists of a bedrock investigation to evaluate potential groundwater contaminant migration pathways into the bedrock in the vicinity of bedrock well MW-308 and this task includes bedrock drilling, rock coring, borehole geophysics, and bedrock well construction. The migration pathway of groundwater at bedrock well MW-308 will be investigated. This investigation includes completion of three well clusters that consist of a deep overburden well, and a shallow bedrock well. More specifically, open-hole bedrock wells (with associated deep overburden wells) will be completed at three locations in the vicinity of MW-308 (Figure 17-2). TtNUS will subcontract drilling and borehole geophysical services.

- Three open-hole bedrock wells will be completed in the inferred upgradient and cross-gradient directions of MW-308. The purpose of these bedrock wells is to evaluate the migration pathway for 1,4-dioxane (and other VOCs) into bedrock well MW-308. Borehole geophysics will be performed in each bedrock borehole to identify rock characteristics, including lithology, fracture orientation, fracture spacing, fracture aperture width, transmissive zones and vertical gradients. In addition, rock cores will be collected at one of the bedrock drilling locations for documentation of the rock lithology, degree of weathering, and fracture spacing. Each well cluster will consist of one (or possibly two) bedrock wells screened in a single bedrock borehole and an overburden well screened immediately above bedrock, the Presumpscot Clay, or basal till, if present.

The three bedrock wells will be constructed as follows:

- The borehole will be advanced through the overburden and approximately 2 feet into the bedrock, and permanent steel casing will be grouted in place. After the grout is allowed to congeal for a minimum of 72 hours, or a minimum of 48 hours if high early strength Portland cement is used, each borehole will be advanced approximately 50 feet into bedrock and water evacuated until the discharge water is free of sediment.
- At the MW-EP-308R location, the upper 30 feet of rock will be cored.
- Borehole geophysics will be performed in each open-hole-bedrock well. Borehole geophysics will consist of caliper, heat-pulse, flowmeter (ambient and stressed conditions), natural gamma, acoustical or optical televiewer, resistivity, and fluid temperature. The borehole geophysical logs will be reviewed by the TtNUS Maine C.G. and recommendations made to the project team. A rationale table will be prepared justifying each recommended new permanent monitoring well, well location, and well screen interval.
- Each bedrock well will be constructed of 1-inch ID, Schedule 40, flush-joint, polyvinyl chloride (PVC) riser pipe and flush-joint, factory-slotted well screen.
- Each section of casing and screen will be National Sanitation Foundation (NSF) approved.
- Well screens will be 5 or 10 feet in length (recommendations will be made to the project team), with an approximate 0.01-inch slot size, surrounded by 20-40 grade filter sand.
- Filter sand will be placed 1 foot (minimum) below the bottom of each well and will extend to 2 feet (minimum) above the top of the well screen.
- A bentonite seal will separate each nested well screen, and the borehole annulus will be sealed above the uppermost filter sand.
- Each well located in an undeveloped area will be protected by a 4-inch (minimum) diameter by 5-foot-long steel protective casing. Protective steel casing will stick up approximately 3 feet above existing grade elevation and will be painted orange with rust-preventative paint. In the vicinity of buildings (and other cultural features), traffic-rated well covers will be cemented flush slightly below grade, with Navy approval. All wells will be locked with keyed-alike locks either provided by the Navy or the Navy's contractor.
- After a minimum of 48 hours has elapsed to allow time for the grout to setup, each new well will be developed by pumping and surging.
- Investigation-derived waste (IDW) soils will be containerized for off-site disposal, if field PID readings exceed 2 parts per million; otherwise, drill cuttings will be spread on the ground.
- IDW liquids will be transported to the Eastern Plume treatment plant for treatment and disposal unless 1,4-dioxane concentrations exceed the MEG of 6.1 ug/L, in which case the IDW liquids will be transported offsite by an IDW subcontractor to TtNUS.

Task 4 – Overburden Monitoring Well Construction and Well Survey

This task includes soil lithology sampling, overburden well construction, development of new monitoring wells, and well surveying. All new monitoring wells completed in this scope will be sampled using bladder pumps following EPA's low-flow sampling protocols and all groundwater samples will be analyzed for 1,4-dioxane and other select VOCs (see Worksheet #15). Horizontal control will be referenced to the State of Maine State Plane coordinate system; vertical control will be to the National Geodetic Vertical Datum of 1983. TtNUS will subcontract survey and drilling services.

Up to 25 permanent overburden wells will be installed as individual shallow, intermediate, or deep wells, where needed, in areas of elevated 1,4-dioxane (and/or other VOCs) concentrations, based on the results of the groundwater profiling. Three of the new overburden wells will be located beside new bedrock wells (as described in Task 3 above); the locations of the remaining overburden wells are to

be determined based on the results of the groundwater profiling task. Justification for permanent monitoring well locations, and well screen intervals will be presented to the project team for approval or modification.

Overburden wells will be constructed as follows:

- The overburden wells will be installed using DPT to the extent possible. If DPT encounters shallow refusal or difficult drilling conditions are encountered that prohibit reaching the target zone for well installation with DPT, then conventional drive and wash drilling will be used to install 1-inch ID monitoring wells.
- Soil samples will be collected at select discrete-interval groundwater sample locations to confirm the presence of sandy strata or zones targeted for well installation. Soil samples will be collected continuously using 4-foot-long sampler using DPT or a 2-foot-long split spoon sampler using drive-and-wash drilling if DPT is not sufficient to install 1-inch ID monitoring wells.
- The wells will be constructed of 1-inch ID, Schedule 40, flush-joint, PVC riser pipe and flush-joint, factory-slotted well screen.
- Each section of casing and screen will be NSF approved.
- Well screens will be 5 or 10 feet in length (recommendations will be made to the project team), with an approximate 0.01-inch slot size, and 20-40 grade silica filter sand will be installed. DPT- installed 1-inch ID wells will be equipped with a pre-packed filter sand.
- Drive-and-wash installed 1-inch ID wells will be completed inside a temporary steel casing and 20-40 grade silica filter sand pack, installed 2 feet above (minimum) the well screen.
- Filter sand will be placed 1 foot (minimum) below the bottom of each well and extend to 2 feet (minimum) above the top of the well screen.
- A bentonite seal will seal the borehole annulus above the top of the filter sand.
- Each well located in an undeveloped area will be protected by a 4-inch (minimum) diameter by 5-foot-long steel protective casing. Protective steel casing will stick-up approximately 3 feet above existing grade elevation and will be painted orange with rust-preventative paint. In the vicinity of buildings (and other cultural features), traffic-rated well covers will be cemented flush slightly below grade, with Navy approval. All wells will be locked with keyed-alike locks either provided by the Navy or the Navy's contractor.
- After a minimum of 48 hours has elapsed to allow time for the grout to set up, each new well will be developed by pumping and surging.
- IDW solids will be containerized for off-site disposal, if PID readings exceed 2 parts per million; otherwise, drill cuttings will be spread on the ground.
- IDW liquids will be transported to the Eastern Plume treatment plant for treatment and disposal unless 1,4-dioxane concentrations exceed the screening criteria of 6.1 ug/L, in which case the IDW liquids will be transported offsite by an IDW subcontractor to TtNUS.

Task 5 –Stream Gauges, Water Level Gauging, and Groundwater Sampling

This task includes installation of stream gauges, a round of groundwater level and surface water-level gauging, and groundwater sampling, as follows:

Staff gauges will be installed at up to four locations along Merriconeag Stream in areas where the 1,4-dioxane plume appears to be discharging based on the pore water results or in areas where no staff gauges exist.

Water level gauging will be performed prior to implementing the groundwater sampling event identified in this SAP. The list of groundwater and surface water gauging stations include ECC's *Final Long-Term Monitoring Plan, Eastern Plume, Naval Air Station Brunswick, Maine*, dated February 2008 (Table 4), and all new monitoring wells and new stream gauges completed as part of this SAP. In addition, existing monitoring wells and surface water gauges may be added based on the results of the pore water survey and groundwater profiling.

Water levels will be manually collected from residential wells. Small-diameter PVC drop pipes will be temporarily inserted into the well and the water level probe lowered into the well through the drop tube, to minimize the potential for the cable to become entangled with the in-place pump piping and wiring. Should the construction of the well be such that a water level measurement cannot be obtained unless the pump is removed, the Tetra FOL will inform the Tetra Tech C.G., who will then consult MEDEP. A water level from a residential well will not be obtained unless the well is deemed critical to the effort (i.e. there are no other nearby wells that can be used instead). If pump removal is required, the pump will be reinstalled (after water level collection) as per the pre-existing conditions and the well will be checked to ensure that it is fully operational.

The 25 new wells and 9 existing monitoring wells selected by the MEDEP will be sampled after well development using a bladder pump following EPA low-flow sampling techniques. The 9 existing wells to be sampled include MW-MB-03C, PZ-MB-B4B, MW-MB-01C, MW-MB-02C, MW-MB-04B, PZ-MB-B6B, PZ-MB-C4B, MW-MB-06A, and MW-MB-06C. All groundwater samples will be analyzed for 1,4-dioxane and select other VOCs. The latest round of long-term monitoring results from existing wells will support preparation of comprehensive isoconcentration maps; the 9 existing

monitoring wells selected by the MEDEP are not included in the long-term monitoring program and are therefore included herein to support the mapping effort.

Quality assurance/quality control (QA/QC) samples will be collected to aid the data validation process (see Worksheet # 20 for a summary of field QA/QC including sampling identifications):

- Field duplicates will be collected at a frequency of 1 per 20 samples.
- Trip blanks will be collected at a frequency of 1 per cooler for VOCs only.
- Field blanks will be collected (at the beginning of the sampling effort) representing the source water used for decontamination (one sample) and steam cleaning (one sample).
- An equipment rinsate blank (one sample) will be collected (at the beginning of the sampling effort) representing the batch of dedicated tubing for groundwater sampling. Dedicated tubing will be used for sampling of each well.

IDW will be containerized for off-site disposal.

Task 6 – Hydraulic Conductivity Testing

This task consists of hydraulic conductivity (slug) testing. Slug tests will be conducted at up to 25 monitoring wells to estimate hydraulic conductivities. Slug tests will be conducted at all newly installed bedrock well and selected monitoring wells (recommendations will be made to the project team). Wells for slug testing will be distributed across the core of the 1,4-dioxane plume and along downgradient migration pathways identified from the profiling.

Slug tests will be performed with sensitive (30 psi or less) transducers. Two slug tests will be performed at each well; the time-recovery data will be analyzed using Hvorslev and Bouwer and Rice analytical solutions to estimate hydraulic conductivity. The hydraulic conductivity results, along with hydraulic gradient and effective porosity estimates, will be used to estimate the 1,4-dioxane and VOC migration rates between the plume core and the likely discharge zone.

SAP Worksheet #18 -- Sampling Locations and Methods/SOP Requirements Table

(UFP-QAPP Manual Section 3.1.1)

Sampling Location/ ID Number[2]	Mat-rix	Synoptic Water level	Depth interval[4] (feet) (bottom screen from TPVC)	Analytical Group	Concen-tration Level	Number of Samples (identify field duplicates)	Sampling SOP Reference	Rationale for Sampling Location
PL-01/ EPGWPL01S-xxxx EPGWxxxxxx Duplicate	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1 plus 1 duplicate	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 1, to delineate the vertical, horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-01/ EPGWPL01I-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 1, to delineate the vertical, horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-01/ EPGWPL01D-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 1, to delineate the vertical, horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-02/ EPGWPL01S-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 1, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-02/ EPGWPL01I-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 1, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-02/ EPGWPL01D-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 1, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.

Sampling Location/ ID Number[2]	Mat-rix	Synoptic Water level	Depth interval[4] (feet) (bottom screen from TPVC)	Analytical Group	Concen-tration Level	Number of Samples (identify field duplicates)	Sampling SOP Reference	Rationale for Sampling Location
PL-03/ EPGWPL03S-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 2, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-03/ EPGWPL03I-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 2, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-03/ EPGWPL03D-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 2, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-04/ EPGWPL04S-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 2, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-04/ EPGWPL04I-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 2, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-04/ EPGWPL04D-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 2, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-05/ EPGWPL05S-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 2, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.

Sampling Location/ ID Number[2]	Mat-rix	Synoptic Water level	Depth interval[4] (feet) (bottom screen from TPVC)	Analytical Group	Concen-tration Level	Number of Samples (identify field duplicates)	Sampling SOP Reference	Rationale for Sampling Location
PL-05/ EPGWPL05I-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 2, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-05/ EPGWPL05D-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 2, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-06/ EPGWPL06S-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 3, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-06/ EPGWPL06I-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 3, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-06/ EPGWPL06D-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 3, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-07/ EPGWPL07S-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 3, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-07/ EPGWPL07I-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 3, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.

Sampling Location/ ID Number[2]	Mat-rix	Synoptic Water level	Depth interval[4] (feet) (bottom screen from TPVC)	Analytical Group	Concen-tration Level	Number of Samples (identify field duplicates)	Sampling SOP Reference	Rationale for Sampling Location
PL-07/ EPGWPL07D-xxxx EPGWxxxxxx Duplicate	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1 plus 1 duplicate	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 3, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-08/ EPGWPL08S-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 3, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-08/ EPGWPL08I-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 3, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-08/ EPGWPL08D-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 3, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-09/ EPGWPL09S-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 4, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-09/ EPGWPL09I-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 4, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-09/ EPGWPL09D-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 4, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.

Sampling Location/ ID Number[2]	Mat-rix	Synoptic Water level	Depth interval[4] (feet) (bottom screen from TPVC)	Analytical Group	Concen-tration Level	Number of Samples (identify field duplicates)	Sampling SOP Reference	Rationale for Sampling Location
PL-10/ EPGWPL10S-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 4, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-10/ EPGWPL10I-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 4, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-10/ EPGWPL10D-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 4, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-11/ EPGWPL11S-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 4, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-11/ EPGWPL11I-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 4, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-11/ EPGWPL11D-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 4, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-12/ EPGWPL12S-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 5, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.

Sampling Location/ ID Number[2]	Mat-rix	Synoptic Water level	Depth interval[4] (feet) (bottom screen from TPVC)	Analytical Group	Concen-tration Level	Number of Samples (identify field duplicates)	Sampling SOP Reference	Rationale for Sampling Location
PL-12/ EPGWPL12I-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 5, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-12/ EPGWPL12D-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 5, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-13/ EPGWPL13S-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 5, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-13/ EPGWPL13I-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 5, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-13/ EPGWPL13D-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 5, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-14/ EPGWPL14S-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 5, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-14/ EPGWPL14I-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 5, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.

Sampling Location/ ID Number[2]	Mat-rix	Synoptic Water level	Depth interval[4] (feet) (bottom screen from TPVC)	Analytical Group	Concen-tration Level	Number of Samples (identify field duplicates)	Sampling SOP Reference	Rationale for Sampling Location
PL-14/ EPGWPL14D-xxxx EPGWxxxxxx Duplicate	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1 plus 1 duplicate	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 5, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-15/ EPGWPL15S-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 6, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-15/ EPGWPL15I-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 6, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-15/ EPGWPL15D-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 6, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-16/ EPGWPL16S-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 6, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-16/ EPGWPL16I-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 6, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-16/ EPGWPL16D-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 6, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.

Sampling Location/ ID Number[2]	Mat-rix	Synoptic Water level	Depth interval[4] (feet) (bottom screen from TPVC)	Analytical Group	Concen-tration Level	Number of Samples (identify field duplicates)	Sampling SOP Reference	Rationale for Sampling Location
PL-17/ EPGWPL17S-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 6, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-17/ EPGWPL17I-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 6, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-17/ EPGWPL17D-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 6, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-18/ EPGWPL18S-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 6, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-18/ EPGWPL18I-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 6, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-18/ EPGWPL18D-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 6, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-19/ EPGWPL19S-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 6, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.

Sampling Location/ ID Number[2]	Mat-rix	Synoptic Water level	Depth interval[4] (feet) (bottom screen from TPVC)	Analytical Group	Concen-tration Level	Number of Samples (identify field duplicates)	Sampling SOP Reference	Rationale for Sampling Location
PL-19/ EPGWPL19I-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 6, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-19/ EPGWPL19D-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 6, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-20/ EPGWPL20S-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 7, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-20/ EPGWPL20I-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 7, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-20/ EPGWPL20D-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 7, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-21/ EPGWPL21S-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 7, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-21/ EPGWPL21I-xxxx EPGWxxxxxx Duplicate	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1 plus 1 duplicate	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 7, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.

Sampling Location/ ID Number[2]	Mat-rix	Synoptic Water level	Depth interval[4] (feet) (bottom screen from TPVC)	Analytical Group	Concen-tration Level	Number of Samples (identify field duplicates)	Sampling SOP Reference	Rationale for Sampling Location
PL-21/ EPGWPL21D-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 7, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-22/ EPGWPL22S-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 7, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-22/ EPGWPL22I-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 7, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-22/ EPGWPL22D-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New temporary overburden well, part of transect line 7, to delineate the vertical and horizontal extent of contamination and determine where to place permanent monitoring wells.
PL-23/ EPGWPL23S-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	To be determined based on pore water sample results.
PL-23/ EPGWPL23I-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	To be determined based on pore water sample results.
PL-23/ EPGWPL23D-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	To be determined based on pore water sample results.
PL-24/ EPGWPL24S-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	To be determined based on pore water sample results.

Sampling Location/ ID Number[2]	Mat-rix	Synoptic Water level	Depth interval[4] (feet) (bottom screen from TPVC)	Analytical Group	Concen-tration Level	Number of Samples (identify field duplicates)	Sampling SOP Reference	Rationale for Sampling Location
PL-24/ EPGWPL24I-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	To be determined based on pore water sample results.
PL-24/ EPGWPL24D-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	To be determined based on pore water sample results.
PL-25/ EPGWPL25S-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	To be determined based on pore water sample results.
PL-25/ EPGWPL25I-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	To be determined based on pore water sample results.
PL-25/ EPGWPL25D-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	To be determined based on pore water sample results.
PL-26/ EPGWPL26S-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	To be determined based on pore water sample results.
PL-26/ EPGWPL26I-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	To be determined based on pore water sample results.
PL-26/ EPGWPL26D-xxxx EPGWxxxxxx Duplicate	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1 plus duplicate	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	To be determined based on pore water sample results.

Sampling Location/ ID Number[2]	Mat-rix	Synoptic Water level	Depth interval[4] (feet) (bottom screen from TPVC)	Analytical Group	Concen-tration Level	Number of Samples (identify field duplicates)	Sampling SOP Reference	Rationale for Sampling Location
PL-27/ EPGWPL27S-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	To be determined based on pore water sample results.
PL-27/ EPGWPL27I-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	To be determined based on pore water sample results.
PL-27/ EPGWPL27D-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	To be determined based on pore water sample results.
PL-28/ EPGWPL28S-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	To be determined based on pore water sample results.
PL-28/ EPGWPL28I-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	To be determined based on pore water sample results.
PL-28/ EPGWPL28D-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	To be determined based on pore water sample results.
PL-29/ EPGWPL29S-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	To be determined based on pore water sample results.
PL-29/ EPGWPL29I-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	To be determined based on pore water sample results.
PL-29/ EPGWPL29D-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	To be determined based on pore water sample results.

Sampling Location/ ID Number[2]	Mat-rix	Synoptic Water level	Depth interval[4] (feet) (bottom screen from TPVC)	Analytical Group	Concen-tration Level	Number of Samples (identify field duplicates)	Sampling SOP Reference	Rationale for Sampling Location
PL-30/ EPGWPL30S-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	To be determined based on pore water sample results.
PL-30/ EPGWPL30I-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	To be determined based on pore water sample results.
PL-30/ EPGWPL30D-xxxx	Ground water	Yes*	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	To be determined based on pore water sample results.
MW-EP-340B1 EPGW340B1-xxxx MW-EP-340B2 EPGW340B2-xxxx	Ground water	Yes	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	2	CT-04, CT-05, GH-1.2, GH-1.3, GH-1.5, GH-2.4, GH-2.5, GH-2.8, ME-12, S1, S2, SA-1.1, SA-6.1, SA-6.3 and SA-7.1	Two nested or single bedrock wells completed in a single open-hole bedrock borehole, located northwest and upgradient at MW-308 to determine vertical gradients, and evaluate potential sources, vertical extent of contamination, and migration pathways into the bedrock near MW-308. See footnote 4. Final well location and well screen interval may be changed based on borehole geophysical results. Significant changes in well location/screen depths are subject to prior concurrence of MEDEP and EPA.
MW-EP-340S/ EPGW340S-xxxx	Ground water	Yes	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New overburden well of monitoring well couplet north and cross-gradient of MW-308 to determine if overburden aquifer cross-gradient of MW-308 is contaminated.

Sampling Location/ ID Number[2]	Mat-rix	Synoptic Water level	Depth interval[4] (feet) (bottom screen from TPVC)	Analytical Group	Concen-tration Level	Number of Samples (identify field duplicates)	Sampling SOP Reference	Rationale for Sampling Location
MW-EP-341B1 EPGW340B1-xxxx MW-EP-341B2 EPGW340B2-xxxx	Ground water	Yes	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	2	CT-04, CT-05, GH-1.2, GH-1.3, GH-1.5, GH-2.4, GH-2.5, GH-2.8, ME-12, S1, S2, SA-1.1, SA-6.1, SA-6.3 and SA-7.1	Two nested or single bedrock wells completed in a single open-hole bedrock borehole, located northwest and upgradient of MW-308 to determine vertical gradients, and evaluate potential sources, vertical extent of contamination, and migration pathways into the bedrock near MW-308. See footnote 4. Final well location and well screen interval may be changed based on borehole geophysical results. Significant changes in well location/screen depth are subject to prior concurrence of MEDEP and EPA.
MW-EP-341S/ EPGW341S-xxxx	Ground water	Yes	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New overburden well of well couplet northwest and upgradient of MW-308 to determine if overburden aquifer upgradient of MW-308is contaminated.
MW-EP-308B1 EPGW308B1-xxxx MW-EP-308B2 EPGW308B2-xxxx	Ground water	Yes	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	2	CT-04, CT-05, GH-1.2, GH-1.3, GH-1.5, GH-2.4, GH-2.5, GH-2.8, ME-12, S1, S2, SA-1.1, SA-6.1, SA-6.3 and SA-7.1	Two nested bedrock wells completed in a single open-hole bedrock borehole, located northwest and upgradient of MW-308 to determine vertical gradients and evaluate vertical extent of contamination near MW-308. See footnote 4. Final well location and well screen interval may be changed based on borehole geophysical results. Significant changes in well location/ screen depth are subject to prior concurrence of MEDEP and EPA.
MW-EP-308S/ EPGW308S-xxxx	Ground water	Yes	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-1.5, GH-2.4, GH-2.5, GH-2.8, GH-3.5, ME-12, S1, S2, SA-1.1, SA-2.5, SA-6.1, SA-6.3 and SA-7.1	New overburden well of monitoring well couplet adjacent to MW-308 to determine vertical extent of contamination at MW-308.

Sampling Location/ ID Number[2]	Mat-rix	Synoptic Water level	Depth interval[4] (feet) (bottom screen from TPVC)	Analytical Group	Concen-tration Level	Number of Samples (identify field duplicates)	Sampling SOP Reference	Rationale for Sampling Location
MW-EP-MB-03C/ EPGMW-MB-03C-xxxx	Ground water	Yes	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1 plus 1 duplicate	CT-04, CT-05, GH-1.2, GH-2.4, S1, S2, SA-1.1, SA-6.1, SA-6.3 and SA-7.1	Well screened in Lower Sand Unit; high concentrations of VOCs /1,4-dioxane discovered during Mere Brook investigation
PZ-EP-MB-B4B/ EPGMW-MB-B4B-xxxx	Ground water	Yes	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-2.4, S1, S2, SA-1.1, SA-6.1, SA-6.3 and SA-7.1	Well screened in Upper Sand Unit, VOCs /1,4-dioxane concentrations high during Mere Brook investigation; well has low turbidity/drawdown.
MW-EP-MB-01C/ EPGMW-MB-01C-xxxx	Ground water	Yes	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-2.4, S1, S2, SA-1.1, SA-6.1, SA-6.3 and SA-7.1	VOCs /1,4-dioxane concentrations elevated during Mere Brook investigation; represents pathway to floodplain.
MW-EP-MB-02C/ EPGMW-MB-02C-xxxx	Ground water	Yes	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-2.4, S1, S2, SA-1.1, SA-6.1, SA-6.3 and SA-7.1	VOCs /1,4-dioxane concentrations elevated during Mere Brook investigation; represents pathway to floodplain.
MW-EP-MB-04B/ EPGMW-MB-04B-xxxx	Ground water	Yes	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-2.4, S1, S2, SA-1.1, SA-6.1, SA-6.3 and SA-7.1	VOCs /1,4-dioxane concentrations elevated during Mere Brook investigation; represents pathway to floodplain.
PZ-EP-MB-B6B/ EPGMW-MB-B6B-xxxx	Ground water	Yes	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-2.4, S1, S2, SA-1.1, SA-6.1, SA-6.3 and SA-7.1	VOCs /1,4-dioxane concentrations elevated during Mere Brook investigation; downgradient of seep discharge.
PZ-EP-MB-C4B/ EPGMW-PZ-C4B-xxxx	Ground water	Yes	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-2.4, S1, S2, SA-1.1, SA-6.1, SA-6.3 and SA-7.1	VOCs/1,4-dioxane results non-detect during Mere Brook investigation; only location available east of Merriconeag Stream.
MW-EP-MB-06A/ EPGMW-MB-06A-xxxx	Ground water	Yes	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-2.4, S1, S2, SA-1.1, SA-6.1, SA-6.3 and SA-7.1	Well is screened in Upper Sand Unit; VOCs/1,4-dioxane detected during Mere Brook investigation; could be separate source because contamination detected in Lower Sand Unit.

Sampling Location/ ID Number[2]	Mat-rix	Synoptic Water level	Depth interval[4] (feet) (bottom screen from TPVC)	Analytical Group	Concen-tration Level	Number of Samples (identify field duplicates)	Sampling SOP Reference	Rationale for Sampling Location
MW-EP-MB-06C/ EPGMW-MB-06C-xxxx	Ground water	Yes	To Be Determined	VOCs, 1,4-Dioxane	Low to Moderate	1	CT-04, CT-05, GH-1.2, GH-2.4, S1, S2, SA-1.1, SA-6.1, SA-6.3 and SA-7.1	Well is screened in Lower Sand / Transitional Unit; OCs/1,4-dioxane detected during Mere Brook investigation;could be separate source of contamination detected in Upper Sand Unit.
Up to 25 additional wells	Ground water	Yes	To be determined	VOCs, 1,4-Dioxane	Low to Moderate	Up to 25 plus 2 duplicates	CT-04, CT-05, GH-1.2, GH-2.4, S1, S2, SA-1.1, SA-6.1, SA-6.3 and SA-7.1	To be determined[4]

[1] EPA and MEDEP will be conducting the pore ater sampling program and are responsible for sampling and analysis.

[2] The ID number consists of, in order, EP for Eastern Plume, GW for groundwater, well number, depth interval for overburden wells PL-1 through PL-21 (S –shallow, I – intermediate, D – deep), and month xx and year xx of sampling event. For example, a groundwater sample collected from intermediate depth in new discrete groundwater sampling location PL-21 in November 2008 would be numbered EPGWPL21I-1108. A duplicate(s) sample from this location would be identified as EPGW followed by the month, date, and year; for example, a duplicate sample collected on November 3, 2008 would be identified as EPGW110308 and a matrix spike/matrix spike duplicate at this location would be identified as EPGW-MSMSD-110308.

[3] Refer to the Analytical SOP References Table (Worksheet_#21).

[4] Depth intervals for the new monitoring wells will be determined based on results of groundwater profiling and confirmed by soil samples. **The TtNUS Maine Certified Geologist and MEDEP Geologist (Chris Evans) will be consulted by the TtNUS Field Operations Leader to establish the screen length and interval for each new monitoring well. The TtNUS Project Manager will be included in the discussion.**

* Although synoptic water levels cannot be collected for these discrete groundwater sampling locations, snapshot water-levels can be obtained through the drill rods and these water levels will be documented in the Supplemental RI Report.

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)
Aqueous	TCL Volatile Organic Compounds (VOCs)	SW-846 8260B/Northeast Laboratory (NEL) SOP # 3.3.13	3-40ml glass volatile vials	2-40ml glass volatile vials	Hydrochloric acid to a pH <2; Cool to 4° C	14 days to analysis
Aqueous	1,4-Dioxane	SW-846 8260B SIM/NEL SOP # 3.3.50	3-40ml glass volatile vials	2-40ml glass volatile vials	Hydrochloric acid to a pH <2; Cool to 4° C	14 days to analysis
Aqueous	1,1-dichloroethene	SW-846 8260B SIMNEL SOP # 3.3.52	3-40ml glass volatile vials	2-40ml glass volatile vials	Hydrochloric acid to a pH <2; Cool to 4° C	14 days to analysis
Solid	TOC	Lloyd Kahn/NEL SOP # 3.2.83,	1-4oz soil jar	1-4oz soil jar	Cool to 4° C	14 days to analysis
Solid	TCLP Regulated Organic Compounds	SW-846 1311 8260B, 8270C, 8081A, 8151A/NEL SOP # 3.3.15, 3.3.14, 3.3.29, 3.3.30, 3.3.5,3.2.8	2-8oz soil jars	2-8oz soil jars	Cool to 4° C	14 days until TCLP 1311 extraction/7 days from TCLP extraction to preparative extraction, then 40 days until analysis
Solid	TCLP Regulated Inorganic Compounds	SW-846 1311 6010B, 7470A/NEL SOP # 3.2.8, 3.2.1, 3.2.3, 3.2.5	1-8oz soil jar	2-8oz soil jars	Cool to 4° C	180 days from sampling to TCLP extraction, then 180 days until analysis

[1] Specify 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.

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

Matrix	Analytical Group	Concentration Level	No. of Sampling Locations	No. of Field Duplicates [1]	No. of MS/MSDs [2]	No. of Field Blanks [3]	No. of Equip. Blanks [4]	No. of PT Samples [5]	No. of VOC Trip Blanks[6]	Total No. of Samples to Lab
Groundwater	TCL VOCs	Low to Moderate	133	7	7	4	7	0	18	169
Groundwater	1,4-Dioxane	Low to Moderate	133	7	7	4	7	0	18	169
Groundwater	1,1-DCE	Low to Moderate	133	7	7	4	7	0	18	169

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

[1] Field Duplicates will be collected at a frequency of 1 per 20 samples.

[2] One MS/MSD is assigned every 20 samples. Triple volume of sample is collected for the sample assigned as Ms/MSD. The MS/MSD samples are not included in the column depicting the total # of samples collected.

[3].Equipment blanks will be collected at a frequency of 1 per 20 samples.

[4]Field blanks will be collected at a frequency of 1 per water source.

[5]No proficiency testing (PT) samples are warranted. Given that an appropriate level of quality control (including laboratory control samples, matrix spikes and replicate samples) is included in the analytical scheme, PT samples are not deemed necessary at this time. If, however, analytical problems arise that indicate a laboratory deficiency, PT sample use will be considered in concert with the state of Maine and EPA.

[6]The number of trip blanks is one per ice chest shipped to the fixed-base laboratory for VOC analysis. Assumed 3 shipments (ice chests) per week x 6 weeks = 18 trip blanks

Notes:
- The ID number will consists of, in order, EP for Eastern Plume, GW for groundwater, type of sample, and month/day/year the sample is collected.
- "T" will be used for trip blank, "R" will be used for rinsate blank, "F" will be used for field blank, and "W" will be used for IDW sample.
  For example, a trip blank collected on November 3, 2008 would be identified as EPGW-T-110308.
- A duplicate(s) sample will be identified by EPGW followed by the month, date, and year; for example, a duplicate sample collected on November 3, 2008 would be identified as EPGW110308 and a matrix spike/matrix spike duplicate at this location would be identified as EPGW-MSMSD-110308.

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
HS-1.0	Utility Locating and Excavation Clearance	TtNUS	Remote subsurface sensing, magnetometer, GPR etc.	N	Refer to Appendix D for field SOPs
CT-04	Sample Nomenclature	TtNUS	NA	N	
CT-05	Data Base Records and Quality Assurance	TtNUS	NA	N	
GH-1.2	Evaluation of Existing Monitoring Wells and Water Level Measurement	TtNUS	Water Level indicator	N	
SA-2.5	Direct Push Technology	TtNUS	Drilling Rig and accessories	N	
GH-1.3	Soil and Rock Drilling Methods	TtNUS	Rotary Drill Rig and accessories	N	
GH-1.5	Borehole and Sample Logging	TtNUS	Geological Logging	N	
GH-2.4	In-Situ hydraulic Conductivity Testing	TtNUS	Pump, Tubing, DI Water, Water Level Indicator	N	
GH-2.5	Groundwater Contour Maps and Flow Determinations	TtNUS	NA		
GH-2.8	Groundwater Monitoring Well Installation	TtNUS	Drill rig, accessories and well supplies	N	
GH-3.5	Borehole Geophysical Surveys	TtNUS	Caliper, heat pulse flow meter (ambient and pumping conditions), acoustical televiewer, conductivity, resistivity, natural gamma, spontaneous potential, and temperature sensors.	N	
SA-1.1	Groundwater Sample Acquisition and Onsite Water Quality Testing	TtNUS	Pump, tubing, water quality meter and accessories	N	
SA-1.3	Soil Sampling	TtNUS	Soil Sample Collection	N	
SA-6.1	Non-Radiological Sample Handling	TtNUS	Sample Bottleware, Packaging Material, Shipping Materials	N	

Reference Number	Title, Revision Date and/or Number	Originating Organization of Sampling SOP	Equipment Type	Modified for Project Work? (Y/N)	Comments
SA-6.3	Field Documentation	TtNUS	Field Logbook, Field Sample Forms, Boring Logs	N	
SA-7.1	Decontamination of Field Equipment	TtNUS	Decon Equipment, scrub brushes, phosphate free detergent, DI water	N	
ME-12	Photovac 2020 Photoionization Air Monitor	TtNUS	Photoionization Air Monitor	N	
ME-15	Photovac Microfid Handheld Flame Ionization Detector	TtNUS	Flame Ionization Detector Air Monitor	N	
S1	USEPA Region 1 Low-Flow Purging and Sampling Methods	USEPA	Pump, tubing, water quality meter and accessories	N	
S2	USEPA Region 1 Draft Calibration of Field Equipment	USEPA	Water Quality meters and accessories	N	
S3	FC4000 Field Instrument MP3500 MIP Controller	Geoprobe Systems	Electrical Conductivity Logging	N	
S4	Geoprobe® Dual Tube (DT21) Soil Sampling System	Geoprobe Systems	Dual Tube Groundwater Profiler	N	
S5	Standard Operating Procedures for the Installation of Non-recording Gages and Measurement of Stage in Streams	OWRB	Staff Gages	N	

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

(UFP-QAPP Manual Section 3.1.2.4)

Field Equipment	Calibration Activity	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[1]
Photo-Ionization Detector	Calibration Gas	Field checks as per manufacture	Calibrated in accordance with manufacture specifications	Visual Inspection	Daily	Manufacture's Guidance	Operator correction or Replacement	FOL	ME-12
Flame-Ionization Detector	Calibration gas	Field checks as per manufacture	Calibrated in accordance with manufacture specifications	Visual Inspection	Daily	Manufacture's Guidance	Operator correction or Replacement	FOL	ME-15
Water Quality Meter	Prepared Standards	Field checks as per manufacture	Calibrated in accordance with manufacture specifications	Visual Inspection	Daily	Manufacture's Guidance	Operator correction or Replacement	FOL	SA-1.1, S1, and S11
Turbidity Meter	Prepared Standards	Field checks as per manufacture	Calibrated in accordance with manufacture specifications	Visual Inspection	Daily	Manufacture's Guidance	Operator correction or Replacement	FOL	SA-1.1, S1
Water Level Indicator	Field checks as per manufacture	Field checks as per manufacture	Field check	Visual Inspection	Once upon receiving from vendor	0.01 foot Accuracy	Operator correction or Replacement	FOL	GH-1.2
Remote subsurface sensing instruments	Field checks as per manufacture	Field checks as per manufacture	Calibrated in accordance with manufacture specifications	Visual Inspection	As per manufacture	Manufacture's Guidance	Operator correction or Replacement	Subcontractor with FOL over sight	HS-1.0
Multi-Parameter TROLL	Field checks as per manufacture	Field checks as per manufacture	Field Check	Visual Inspection	Once upon receiving from vendor	Manufacture's Guidance	Operator correction or recplacement	FOL	GH-2.4
Rugged Reader	Field checks as per manufacture	Field checks as per manufacture	Field Check	Visual Inspection	Once upon receiving from vendor	Manufacture's Guidance	Operator correction or recplacement	FOL	GH-2.4
Geoprobe Electrical Conductivity Profiler	Field checks as per manufacture	Field checks as per manufacture	Field Check	Visual Inspection	Once upon receiving from vendor	Manufacture's Guidance	Operator correction or recplacement	Subcontractor with FOL over sight	GH-3.5

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

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? (Y/N)
3.3.13	Volatile Organic Compounds EPA 8260B-J-6/16/07	Definitive Method	Aqueous- VOCs	Archon 5100/Hewlett Packard 5890II/5971	Northeast Laboratory Services, Inc.	N
3.3.50	1,4-Dioxane 8260B (SIM)-B-1/23/08	Definitive Method	Aqueous-1,4-Dioxane	Archon 5100/Hewlett Packard 5890II/5971	Northeast Laboratory Services, Inc.	N
3.3.52	1,1-Dichloroethene 8260B-SIM 08/04/08	Definitive Method	Aqueous-1,1-dichloroethene	Archon 5100/Hewlett Packard 5890II/5971	Northeast Laboratory Services, Inc.	N
3.3.29	Separatory Funnel Liquid-Semi-Volatile Organics-D-10/20/06	Preparation Method	Aqueous-TCLP extract-Semi-volatile organics	Preparation	Northeast Laboratory Services, Inc.	N
3.3.30	Separatory Funnel Liquid- SW-846 Method 3510C-Pesticides and PCBs C-1/16/06	Preparation Method	Aqueous-TCLP extract-Pesticides	Preparation	Northeast Laboratory Services, Inc.	N
3.3.5	Determination of Chlorinated Acids in Water(8151A)-E-10/24/06	Preparation Method	Aqueous-TCLP extract-Herbicides	Hewlett Packard 5890 Series II Plus	Northeast Laboratory Services, Inc.	N
3.2.8	Toxicity Characteristic Leaching Procedure Method 1311-C-2/6/07	Preparation Method	Solid-TCLP extraction	Preparation	Northeast Laboratory Services, Inc.	N

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? (Y/N)
3.2.1	Metals Analysis EPA 6010B-I-12/14/06	Determinative Method	Aqueous-TCLP extract for inductively coupled plasma metals	Perkin Elmer Optima 3000XL ICP	Northeast Laboratory Services, Inc.	N
3.2.3	Mercury in Liquid Waste-EPA 7470A-F-4/25/07	Determinative Method	Aqueous-TCLP extract for Mercury	Perkin Elmer-FIMS 100	Northeast Laboratory Services, Inc.	N
3.3.15	Semivolatile Organic Compounds EPA 8270C-K-2/14/07	Determinative Method	Aqueous-TCLP extract for Sem-volatile organics	Hewlett Packard 5890/5970/7673A	Northeast Laboratory Services, Inc.	N
3.3.14	Organochlorine Pesticides (8081)-H-2/14/07	Determinative Method	Aqueous-TCLP extract for Pesticides	Hewlett Packard 5890 Series II Plus	Northeast Laboratory Services, Inc.	N
3.2.5	Acid Digestion of Aqueous Sample and Extracts for Total Metals for Analysis by FLAA or Inductively Coupled Plasma (ICP) Spectroscopy EPA 3010A-D-2/6/07	Preparation Method	Aqueous-TCLP extract for inductively coupled plasma metals	Preparation	Northeast Laboratory Services, Inc.	N
3.2.83	Total Organic Carbon in Solid Matrices-Method 9060 (modified) and Lloyd Kahn (1988)	Determinative Method	Sediment-wet chemistry	Hach IL500 TOC	Northeast Laboratory Services, Inc.	N

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]
Volatile Organic Analysis (VOA) - Archon 5100/HP 5890 II/HP 5971	Initial - minimum of five points	As needed, prior to sample analysis	Minimum RF for System performance check compounds (SPCC), maximum 30% relative standard deviation (RSD) for Continuing calibration compound (CCC). All analytes ≤ 15% or Correlation Coeffiecient (r^2) > 0.990	Maintenance (check for leaks, change trap, etc.) followed by recalibration	Analyst/supervisor	Northeast Laboratory Services, Inc. (NEL) SOP 3.3.13
VOA	Continuing calibration verification (CCV)	Beginning of each 12 hour window, after tune, or as part of a curve	Minimum RF for SPCCs, %D for each CCC ≤ 20%	System check and recalibrate	Analyst/supervisor	NEL SOP 3.3.13
1,1-Dichloroethene- Archon 5100/HP 5890 II/HP 5971	Initial - minimum of five points	As needed, prior to sample analysis	%RSD ≤ 30% for 1,1-DCE	Repeat calibration after system check/maintenance	Analyst/supervisor	NEL SOP 3.3.52
1,1-Dichloroethene	CCV	Beginning of each 12 hour window, after tune, or as part of a curve	%D ≤ 30% for analysis to proceed	System check and recalibrate	Analyst/supervisor	NEL SOP 3.3.52
1,4-Dioxane- Archon 5100/HP 5890 II/HP 5971	Initial-minimum of five points	As needed, prior to sample analysis	%RSD ≤ 30% for 1,4-dioxane	Repeat calibration after system check/maintenance	Analyst/supervisor	NEL SOP 3.3.50
1,4-Dioxane	CCV	Beginning of each 12 hour window, after tune, or as part of a curve	%D ≤ 30% for analysis to proceed	System check and recalibrate	Analyst/supervisor	NEL SOP 3.3.50

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA[2]	SOP Reference[1]
HP 5890 Series II Plus- pesticides/ Polychlorinated Biphenyls (PCBs)/ Herbicides	Initial-minimum of 6 points	As needed, prior to sample analysis	Each correlation coefficient > 0.990	Repeat calibration after system check/maintenance	Analyst/supervisor	NEL SOPs 3.3.15, 3.3.14, 3.3.29, 3.3.30, 3.3.5, 3.2.8
HP 5890 Series II Plus- pesticides/PCBs/ Herbicides	CCV	Opening of 12 hour sequence and every 10 samples	Each %D ≤ 15%	System check and recalibrate	Analyst/supervisor	NEL SOPs 3.3.15, 3.3.14, 3.3.29, 3.3.30, 3.3.5, 3.2.8
Perkin Elmer Optima 3000XL ICP-Metals Analysis	Initial- minimum of four points	Daily	Correlation coefficient ≥ 0.995	Repeat calibration after system check/maintenance	Analyst/supervisor	NEL SOPs 3.2.8, 3.2.1, 3.2.3, 3.2.5
Metals	CCV	Beginning of sequence and every 10 samples	%D ≤ 10% for each metals	System check and recalibrate	Analyst/supervisor	NEL SOPs 3.2.8, 3.2.1, 3.2.3, 3.2.5
Mercury- Perkin Elmer-FIMS 100	Initial-six points	Daily	Correlation coefficient ≥ 0.995	Repeat calibration after system check/maintenance	Analyst/supervisor	NEL SOPs 3.2.8, 3.2.1, 3.2.3, 3.2.5
Hg (mercury)	CCV	Beginning of sequence and every 10 samples	%D ≤ 10% for Hg	System check and recalibrate	Analyst/supervisor	NEL SOPs 3.2.8, 3.2.1, 3.2.3, 3.2.5
TOC	Initial-five points	As needed, prior to sample analysis	Each correlation coefficient >0.995	1) Investigate source of non-linearity. 2) Re-prepare and reanalyze	Analyst/supervisor	NEL SOP 3.2.83
TOC	CCV	Every 20 samples, opening and closing	Percent recovery + 10%	System check and recalibrate	Analyst/supervisor	NEL SOP 3.2.83

[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.

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]
TCL Volatile Organic Analysis (VOA) - Archon 5100/HP 5890 II/HP 5971	Check gas supply, head pressures, oil levels before tuning	Volatile	Visual	Daily	No leaks, gas tanks ≥ 500 psi, oil changes quarterly	Change gas tank and/or oil, repair leaks if found	Analyst/ supervisor	NEL SOP 3.3.13
1,4-dioxane and 1,1-DCE Hewlett Packard 5890/5970/7673A	Check gas supply, head pressures, oil levels before tuning	Volatile	Visual	Daily	No leaks, gas tanks ≥ 500 psi, oil changes quarterly	Change gas tank and/or oil, repair leaks if found	Analyst/ supervisor	NEL SOP 3.3.50 and 3.3.52
HP 5890 Series II Plus - pesticides/Polychlorinated Biphenyls (PCBs)/ herbicides	Check gas supply, head pressures, before calibration	Pesticides PCBs Herbicides	Visual	Daily	No leaks, gas tanks ≥ 500 psi	Change gas tank, repair leaks if found	Analyst/ supervisor	NEL SOPs 3.3.15, 3.3.14, 3.3.29, 3.3.30, 3.3.5, 3.2.8
Perkin Elmer Optima 3000XL ICP - Metals Analysis	Check all fluids, chiller water, internal std and rinse solutions, change peristaltic pump tubing	Metals	Visual	Daily	Internal Standard (IS) and rinse solutions at least ½ full- depends on the number of samples to be analyzed	Fill solutions	Analyst/ supervisor	NEL SOPs 3.2.8, 3.2.1, 3.2.3, 3.2.5

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person2	SOP Reference[1]
Mercury-Perkin Elmer-FIMS 100	Check all fluids, internal std and rinse solutions, change peristaltic pump tubing	Metals	Visual	Daily	IS and rinse solutions at least ½ full-depends on the number of samples to be analyzed	Fill solutions	Analyst/ supervisor	NEL SOPs 3.2.8, 3.2.1, 3.2.3, 3.2.5
Hach IL500 TOC-TN	Check all fluids and rinse solutions.	Wet Chemistry, TOC	Visual	Daily	Rinse bottle, sodium persulate, and phosphoric acid containers at least ½ full	Fill solutions	Analyst/ supervisor	NEL SOP 3.2.83

[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.

SAP Worksheet #26 -- Sample Handling System

(UFP-QAPP Manual Appendix A)

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): Field Operations Leader/TtNUS NUS, Inc.
Sample Packaging (Personnel/Organization): Field Operations Leader/TtNUS NUS, Inc.
Coordination of Shipment (Personnel/Organization): Field Operations Leader/ TtNUS NUS, Inc.
Type of Shipment/Carrier: Federal Express
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Sample Receipt Personnel/Northeast Laboratory Services, Inc.-See NEL SOP # 3.4.0 Procedure for proper sample receipt and log-in of analytical samples.
Sample Custody and Storage (Personnel/Organization): See above
Sample Preparation (Personnel/Organization): Organic and/or inorganic analysts-Northeast Laboratory Services, Inc.-See preparation SOPs referenced on Worksheet #23
Sample Determinative Analysis (Personnel/Organization): Organic and/or inorganic analysts-Northeast Laboratory Services, Inc.-See determinative SOPs referenced on Worksheet #23
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): See Worksheet #19 and NEL SOP # 3.7.11. Samples will be shipped to lab within 1 day from collection
Sample Extract/Digestate Storage (No. of days from extraction/digestion): See Worksheet #19 and NEL SOP # 3.7.11. Extracts, digestates will be archived 6 months by lab after report is submitted to TTNUS.
Biological Sample Storage (No. of days from sample collection): Not applicable
SAMPLE DISPOSAL
Personnel/Organization: Northeast Laboratory Services, Inc.
Number of Days from Analysis: See NEL SOP# 3.7.11-Standard Operating Procedure for the proper disposal of samples. Leftover samples will be disposed by lab 60 days after report submittal.

SAP Worksheet #27 – Sample Custody Requirements Table

(UFP-QAPP Manual Section 3.3.3)

SAMPLE NOMENCLATURE

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 the investigation. All forms must be filled in as completely as possible.

Sample Identification

Refer to Worksheet #18 for sample labeling information.
Also, refer to Worksheet #20 for field QA/QC labeling information.

Sample Collection Documentation

Documentation of field observations will be recorded in a field logbook and/or field log sheets including sample collection logs, boring logs, VOC screening logs, and monitoring well construction logs. Field logbooks utilized on this project will consist of bound, water-resistant logbooks. All pages of the logbook will be numbered sequentially, and observations will be recorded with indelible ink.

Field sample log sheets will be used to document sample collection details, and other observations and activities will be recorded in the field logbook. Instrument calibration logs will be used to record the daily instrument calibration. Example field forms are included in Appendix E.

For sampling and field activities, the following types of information will be recorded in the field log as appropriate:

- Site name and location
- Date and time of logbook entries
- Personnel and their affiliations
- Weather conditions
- Activities involved with the sampling
- Subcontractor activity summary
- Site observations including site entry and exit times
- Site sketches made on site
- Visitor names, affiliations, and arrival and departure times
- Health and safety issues including personal protective equipment (PPE)

Sample Handling and Tracking System

These subsections outline the procedures that will be used by field and laboratory personnel to document project activities and sample collection procedures during this RI. All forms must be filled in as completely as possible.

Sample Handling

Sample handling is described in Worksheet #26.

Sample Delivery

Samples to be delivered to the laboratory will be made by a public courier (i.e., Federal Express). After samples have been collected, they will be sent to the laboratory within 24 hours. Under no circumstances will sample holding times be exceeded.

Sample Custody

To ensure the integrity of a sample from collection through analysis, it is necessary to have an accurate written record that traces the possession and handling of the sample. This documentation is referred to as the chain-of-custody (COC) form. The chain of custody begins at the time of sample collection.

A sample is under custody if:

- The sample is in the physical possession of an authorized person
- The sample is in view of an authorized person after being in his/her possession
- The sample is placed in a secure area by an authorized person after being in his/her possession
- The sample is in a secure area, restricted to authorized personnel only

Custody documentation is designed to provide documentation of preparation, handling, storage, and shipping of all samples collected. A multi-part form is used, with each page of the form signed and dated by the recipient of a sample or portion of sample. The person releasing the sample and the person receiving the sample each will retain a copy of the form each time a sample transfer occurs.

Integrity of the samples collected during the RI will be the responsibility of identified persons from the time the samples are collected until the samples, or their derived data, are incorporated into the final report. Sample custody is described in Worksheet #27.

The FOL is responsible for the care and custody of the samples collected until they are delivered to the laboratory or are entrusted to a carrier. When transferring samples, the individuals relinquishing and receiving them will sign, date, and note the time on the chain-of-custody form. This record documents the sample custody transfer from the sampler to the laboratory, often through another person or agency (common carrier). Upon arrival at the laboratory, internal sample custody procedures will be followed as defined in the laboratory SOPs included in Appendix F.

Laboratory Custody Procedures

The following SOPs are saved on CD (see Appendix F) and describe, in detail, the procedures referenced below:
NEL SOP # 3.4.0-Proper Sample Receipt and Log-in of Analytical Samples

NEL SOP# 3.7.11-Standard Operating Procedure of the Disposal of Samples
NEL SOP# 3.7.3-Determination of Hazardous Constituents for Sample Disposal

All Navy samples considered as Level IV data packages require the following procedure for sample receipt QA:

1 Samples are checked against the COC (form example attached in SOP # 3.4.0). The collection date and time are observed to ensure that samples arrive within the recommended holding time. Sample coolers are inspected for custody seals (example attached with SOP) and/or damage. Sample containers are also inspected for proper preservation. Based on the client's instructions, samples that are too old or are not preserved can be processed (preserved in-house and analyzed) or may be cancelled. A note concerning the condition of the sample(s) upon arrival at the laboratory and the client's instructions regarding that condition must appear on the final report.

2 The COC is signed and dated by the technician receiving the sample.

3 An SDG number is assigned to the COC.

4 Samples are numbered using the following procedure:

i. Samples are assigned a unique sequential laboratory identification (ID) number. Prefixes to these numbers vary by year and department, PG represents 2007 potability samples, AJ represent 2007 chemistry and samples with both microbiological and chemistry analyses, MG represents 2007 samples for strictly microbiological testing of air and food samples. The sequential numbers are fixed to the sample containers using neon labels. Yellow signifies chemistry samples and pink signifies microbiology samples. Permanent markers are used if the number of bottles per sample exceeds three for chemistry samples and one for microbiology samples.

ii. Samples containing more than one sample container per site are assigned an additional unique sample ID. Within the set, the original sample number is followed by an additional sequential number starting with 1, which is preceded by a decimal. For example, AJ13652.1, AJ13652.2 and so on until all bottles are labeled.

5 Samples with the laboratory ID number are then placed on a transport cart for the sample custodian to continue with the sample process procedure. The pH of all samples, except volatile organic samples, will be taken under a hood in the radon room by removing an aliquot of sample and then using a small disposable glass rod to sample from the aliquot and then touch the rod to a pH indicator strip and noted on the ICOC (Internal Chain of Custody form example attached in SOP # 3.4.0). When it is necessary to add acid, a notation is made on the ICOC and the client is contacted. The ICOC is completed and filed in the chemistry department. Samples are then placed in the appropriate sample storage cooler. The corresponding storage location and date and time the sample was placed there, is noted on the ICOC. Samples that need immediate attention are given to the appropriate analyst with a notation on the ICOC.

6 After samples are logged into the system, a sample log-in summary is printed. The log-in summary is then compared to the COC. The sample receipt printout, sample receipt form, copy of the COC and a cover letter are then faxed and/or e-mailed if required by the client to the client as soon as possible. This packet is then forwarded to the reporting clerk to file for later use. A copy of the Sample Receipt Report is always placed in the Chain-of-Custody Book in the chemistry laboratory with a copy of the COC.

7 Samples are to be disposed no earlier than 30 days after the report is issued. Clients are made aware of this in writing with the quotation for any particular project. If the client requests so, they will be contacted prior to disposal.

a. The LIMS is used to determine samples that are ready for disposal. This is done by a cross-reference search by report date. The data are then exported to a sample disposal log where the disposal date, disposal method, and initials of the disposer are recorded. The sample disposal log is a spreadsheet stored on the local network. ICOC forms are maintained until the sample is disposed.

8 Sample extracts/digests are to be disposed no earlier than 6 months after the report is issued. The same procedures for disposal of samples are followed for the disposal of sample extracts/digests.

b. After the sample and/or extract/digest has been designated for disposal refer to SOP # 3.7.3, Hazardous Waste Determination, to determine them as hazardous or non-hazardous.

c. If the sample and/or extract/digest is determined to be hazardous, it is to be sent back to the customer. If this is not an option, the sample and/or extract/digest is to labeled as hazardous waste and be placed in the hazardous waste shed for disposal within 90 days by a hazardous waste broker.

d. If the sample and/or extract/digest is liquid and non-hazardous or has the hazardous characteristic of corrosivity only, it is to be disposed of through the Elemental Neutralization Unit (ENU).

e. Solid non-hazardous samples are to be disposed via landfill.

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

Matrix	Aqueous
Analytical Group	Volatile Organic Compounds (VOCs) and Toxicity Characteristic Leaching Procedure (TCLP) VOCs
Analytical Method / SOP Reference	SW-846 1311/8260B/ NEL SOP 3.3.13

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 20 samples or one every 12 hour sequence	Positive detects less than the Quantitation Limit (QL)	Reanalyze affected samples if there are positive detects of the same analyte	Analyst	Bias and Contamination	Positive detects less than the QL
Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)	One set per 20 samples or one set every 12 hour sequence	90% of recoveries 60%-140%; 90% of Relative Percent Difference (RPDs) $\leq$ 20%	Reanalyze affected samples	Analyst	Accuracy/precision	90% of recoveries 60%-140%; 90% of RPDs $\leq$ 20%
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	One set per 20 samples	90% of recoveries 60%-140%; 90% of RPDs $\leq$ 20%	Reanalyze affected samples; if MS/MSD out of criteria and batch LCS/LCSD acceptable, then no laboratory action is required	Analyst	Accuracy/precision	90% of recoveries 60%-140%; 90% of RPDs $\leq$ 20%
Surrogate Recoveries:	One set of three	Recoveries	Reanalyze if out	Analyst	Accuracy/precision	Recoveries

Matrix	Aqueous					
1,2-dichloroethane-d4 Toluene-d8 4-bromofluorobenzene	surrogates per sample, blank, LCS/LCSD, MS/MSD	1,2-Dichloroethane-d4 70%-152% Toluene-d8 82%-140% bromofluorobenzene 82%-114%				1,2-Dichloroethane-d4 70%-152% Toluene-d8 82%-140% bromofluorobenzene 82%-114%
Internal Standard (IS) area Recoveries Pentafluorobenzene 1,4-difluorobenzene Chlorobenzene-d5 1,4-dichlorobenzene-d4	One set of four analytes per sample, blank, LCS/LCSD, MS/MSD	Recovery -50% to +100% compared to Continuing calibration verification (CCV) IS area Retention Time Window (RTW) $\pm$ 0.5 min compared to CCV IS Retention Time (RT)	Reanalyze if out	Analyst	Accuracy/precision	Recovery -50% to +100% compared to CCV IS area RTW $\pm$ 0.5 min compared to CCV IS RT
Temperature Blank	1 per cooler	4 $\pm$ 2 °C	Notify TtNUS Project Chemist or PM who will address the impact to the project and determine the appropriate action	TtNUS field operations leader	Precision / Representativity / Bias	4 $\pm$ 2 °C

Matrix	Aqueous
Analytical Group	1,4-dioxane
Analytical Method / SOP Reference	Modified SW-846 8260B/NEL SOP 3.3.50

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 12 hour sequence	Positive detects less than the QL	Reanalyze batch if positive detects are greater than the reporting level	Analyst	Bias and Contamination	Positive detects less than the QL
Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)	One set per 12 hour sequence	1,4-dioxane recovery 60%-140%; RPD $\leq$ 30%	Reanalyze affected samples	Analyst	Accuracy/precision	1,4-dioxane recovery 60%-140%; RPD $\leq$ 30%
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	One per 12 hour sequence as specified by the client	1,4-dioxane recovery 60%-140%; RPD $\leq$ 30%	If MS/MSD is outside the criteria and the batch LCS/LCSD is acceptable then no laboratory action is required.	Analyst	Accuracy/precision	1,4-dioxane recovery 60%-140%; RPD $\leq$ 30%
Internal Standard (IS) area	Bromofluorobenzen	Recovery -50% to +100% compared to CCV IS area Retention Time Window (RTW) $\pm$ 0.5 min compared to CCV IS Retention Time (RT)	Reanalyze	Analyst	Accuracy	Recovery -50% to +100% compared to CCV IS area; RTW $\pm$ 0.5 min compared to CCV IS RT
Surrogate recoveries	1,4-dioxane-d8	Recovery 20%-150%	Reanalyze	Analyst	Accuracy	Recovery 20%-150%

Matrix	Aqueous
Analytical Group	1,1-dichloroethene
Analytical Method / SOP Reference	Modified SW-846 8260B/NEL SOP 3.3.52

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 12 hour sequence	Positive detects less than the QL	Reanalyze batch if positive detects are greater than the reporting level	Analyst	Bias and Contamination	Positive detects less than the QL
Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)	One set per 12 hour sequence	1,1-dichloroethene recovery 60%-140%; RPD $\leq$ 30%	Reanalyze affected samples	Analyst	Accuracy/precision	1,1-dichloroethene recovery 60%-140%; RPD $\leq$ 30%
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	One per 12 hour sequence as specified by the client	1,1-dichloroethene recovery 60%-140%; RPD $\leq$ 30%	If Ms/MSD is outside the criteria and the batch LCs/LCSD is acceptable then no laboratory action is required.	Analyst	Accuracy/precision	1,1-dichloroethene recovery 60%-140%; RPD $\leq$ 30%
Internal Standard (IS) area	Bromofluorobenzen	Recovery -50% to +100% compared to CCV IS area RTW $\pm$ 0.5 min compared to CCV IS RT	Reanalyze	Analyst	Accuracy	Recovery -50% to +100% compared to CCV IS area; RTW $\pm$ 0.5 min compared to CCV IS RT
Surrogate recoveries	1,2-dichloroethane-d4	Recovery 60%-140%	Reanalyze	Analyst	Accuracy	Recovery 60%-140%

Matrix	TCLP
Analytical Group	Organics
Analytical Method / SOP Reference	SW-846 1311 8270C, 8081A, 8151A, NEL SOPs 3.3.15, 3.3.14, 3.3.29, 3.3.30, 3.3.5, 3.2.8

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 20 samples	Positive detects less than the reporting level	Re-extract affected samples if positive detects of the same analyte	Analyst	Bias and Contamination	Positive detects less than the reporting level
Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)	One set per 20 samples or one set every extraction batch	90% of recoveries 60%-140%; 90% of Relative Percent Difference (RPD) $\leq$ 20%	Re-extract affected samples	Analyst	Accuracy/precision	90% of recoveries 60%-140%; 90% of RPDs $\leq$ 20%
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	One set per 20 samples	90% of recoveries 60%-140%; 90% of RPDs $\leq$ 20%	Re-extract affected Samples; if MS/MSD out of criteria and batch LCS/LCSD ok, no laboratory action	Analyst	Accuracy/precision	90% of recoveries 60%-140%; 90% of RPDs $\leq$ 20%
Surrogate recoveries	8081A-two per sample and QC 8151A-one per sample and QC	8081A: Tetrachloro-m-xylene: percent recovery 18%-156% Decachlorobiphenyl: percent recovery 25%-158% 8151A- 2,4-dichlorophenylacetic acid percent recovery 60%-140%	Reanalyze first if any out If still out, re-extract to confirm matrix interference	Analyst	Accuracy	8081A: Tetrachloro-m-xylene: percent recovery 18%-156% Decachlorobiphenyl: percent recovery 25%-158% 8151A- 2,4-dichlorophenylacetic acid percent recovery 60%-140%

Matrix	TCLP					
Surrogate recoveries	8270C-6 per sample and QC	Percent recoveries 2-fluorophenol: 21%-100% Phenol-d5: 10%-94% Nitrobenzene-d5: 35%-114% 2-fluorobiphenyl: 43%-116% 2,4,6-tribromophenol: 10%-123% Terphenyl-d14: 33%-141%	Reanalyze first if any out If still out, re-extract to confirm matrix interference	Analyst	Accuracy	Percent recoveries 2-fluorophenol: 21%-100% Phenol-d5: 10%-94% Nitrobenzene-d5: 35%-114% 2-fluorobiphenyl: 43%-116% 2,4,6-tribromophenol: 10%-123% Terphenyl-d14: 33%-141%
Internal Standard (IS) Area – 8270C 1,4-dichlorobenzene-d4 Naphthalene-d8 Acenaphthene-d10 Phenanthrene-d10 Chrysene-d12 Perylene-d12	8270C-6 per sample and QC	Recovery -50% to +100% compared to CCV IS area RTW ± 0.5 min compared to CCV IS RT	Reanalyze first if any out If still out, re-extract to confirm matrix interference	Analyst	Accuracy	Recovery -50% to +100% compared to Continuing calibration verification (CCV) IS area Retention Time Window (RTW) ± 0.5 min compared to CCV IS Retention Time (RT)

Matrix	TCLP
Analytical Group	Inorganics
Analytical Method / SOP Reference	SW-846 1311/6010B/7470A NEL SOPs 3.2.8, 3.2.1, 3.2.3, 3.2.5

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 20 samples	Positive detects less than the reporting level	Redigest affected samples if positive detects of the same analyte	Analyst	Bias and Contamination	Positive detects less than the reporting level
Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)	One set per 20 samples or one set every extraction batch	All recoveries 80%-120%; RPD $\leq$20%	Re-extract affected samples	Analyst	Accuracy/precision	All recoveries 80%-120%; % RPDs $\leq$20%
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	One set per 20 samples	All recoveries 80%-120%; RPD $\leq$20%	Re-extract affected Samples; if MS/MSD out of criteria and batch LCS/LCSD ok, no laboratory action	Analyst	Accuracy/precision	All recoveries 80%-120%; RPDs $\leq$20%

Matrix	Soil
Analytical Group	TOC
Analytical Method / SOP Reference	Lloyd Kahn NEL SOP 3.2.83

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 batch of 20 or fewer samples	TOC less than the QL of 0.1%	1) Investigate source of contamination. 2) Re-prepare and reanalyze all associated samples if sample concentration ≥ QL	Analyst/ Supervisor	Bias and Contamination	TOC less than the QL of 0.1%
Laboratory Duplicate	As requested by the client	RPD ≤ 20%	1) Investigate source of contamination. 2) Re-prepare and reanalyze	Analyst/ Supervisor	Precision	RPD ≤ 20%
Laboratory Matrix Spike	One per matrix or batch of 20 or fewer samples, whichever is more frequent, as requested by the client	Recovery 75% – 125%	Note on sample report	Analyst/ Supervisor	Accuracy	Recovery 75% – 125%
Laboratory Matrix Spike Duplicate	One per matrix or batch of 20 or fewer samples, whichever is more frequent ,a s requested by the client	Recovery 75% - 125% ≤ 20 % RPD between MS and MSD	Note on sample report	Analyst/ Supervisor	Accuracy and precision	Recovery 75% - 125% ≤ 20 % RPD between MS and MSD
LCS/LCSD	One set per batch of 20 or fewer samples	Recovery 80% - 120%	Reanalyze all affected samples.	Analyst/ Supervisor	Accuracy	Recovery 80% - 120%
Replicate	Every QC and field sample	Coefficient of variation ≤ 20%	Reanalyze fresh sample aliquot to confirm matrix interference	Analyst/ Supervisor	Accuracy	Coefficient of variation ≤ 20%

Note: TtNUS has reviewed the analytical laboratory QC detection limits and finds them to be acceptable for the project.

SAP Worksheet #29 -- Project Documents and Records Table

(UFP-SAP Manual Section 3.5.1)

Document	Where Maintained
Sample Collection Documents and Records Field logbook (and sampling notes) Field sample forms (e.g. boring logs, sample log sheets, drilling logs, etc.) Chain-of-custody records Sample shipment airbills Equipment calibration logs Photographs Field Task Modification Forms Sampling and Analysis Plan Field Sampling SOPs	TtNUS project file, results will be discussed in subject document.
Laboratory Documents and Records Sample receipt/login form Sample storage records Sample preparation logs Standard traceability logs Equipment calibration logs Sample analysis run logs Equipment maintenance, testing , and inspection logs Corrective action forms Reported field sample results Reported results for standards, quality control checks, and quality control samples Data completeness checklists Sample storage and disposal records Telephone logs Extraction/clean-up records Raw data	TtNUS project file, Long-term data package storage at third-party professional document storage firm (BRM), results will be discussed in subject document.
Data Assessment Documents and Records Field Sampling Audit Checklist (if an audit is conducted) Analytical Audit Checklist (if an audit is conducted) Data Validation Memoranda	TtNUS project file, results will be discussed in subject document.

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

Matrix	Analytical Group	Sample Locations/ID Number	Analytical Method	Data Package Turnaround Time	Laboratory / Organization (name and address, contact person and telephone number)	Backup Laboratory / Organization (name and address, contact person and telephone number)
Aqueous	TCL VOCs	See Worksheet 18	SW-846 8260B (with SIM for 1,1-DCE)	21 calendar days	Pam Doughty Northeast Laboratory Services, Inc. P.O. Box 788 Waterville, ME 04903-0788 (207) 873-7022	NA
Aqueous	1,4-Dioxane	See Worksheet 18	SW-846 8260B SIM	21 calendar days (standard turn) 24 hours (quick turn)	Pam Doughty Northeast Laboratory Services, Inc. P.O. Box 788 Waterville, ME 04903-0788 (207) 873-7022	NA
Solid	TOC	See Worksheet 18	Lloyd Kahn or Walkley Black	21 calendar days	Pam Doughty Northeast Laboratory Services, Inc. P.O. Box 788 Waterville, ME 04903-0788 (207) 873-7022	NA
IDW/ Solid	TCLP Organic and Inorganic	See Worksheets 17 and 20	SW-846 1311/8260B, 8270C, 8081A, 8151A, 8330, 6010B, 7470	21 calendar days	Pam Doughty Northeast Laboratory Services, Inc. P.O. Box 788 Waterville, ME 04903-0788 (207) 873-7022	NA

NA Not Applicable.
SIM Selective ion monitoring.
TCLP Toxicity Characteristic Leaching Procedure.
TOC Total organic carbon.
VOC Volatile organic compound.

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 Supervision	Daily	Internal	TtNUS	FOL (TBD), TtNUS	FOL (TBD), TtNUS	FOL and field crew	FOL, PM, QAM (Tom Johnston)
Project Supervision	Monthly	Internal	TtNUS	PM (Linda Klink), TtNUS	FOL (TBD), TtNUS	FOL (TBD), TtNUS	PM (Linda Klink), FOL (TBD), and Program Manager (John Trepanowski), TtNUS
Field Sampling System Audit	One per contract year	Internal	TtNUS	TBD	PM (Linda Klink), TtNUS FOL (TBD), TtNUS	Auditor, FOL, and PM	TtNUS QAM (Tom Johnston)
Laboratory System Audit	Every 18 months	External	NFESC	TBD, NFESC	Laboratory QA Manager or Laboratory Manager	Laboratory Manager and QA Manager	NFESC

System audits will be performed as appropriate to ensure that the work is being implemented in accordance with the approved project SOPs and in an overall satisfactory manner.

- The TtNUS FOL will supervise field operations. The FOL will perform a daily check to ensure that the field instruments are calibrated, equipment is properly decontaminated, samples are collected and handled properly, and fieldwork is accurately and neatly documented. Corrective actions will be implemented immediately if any non-compliance is detected. The FOL will also check records for reasonable results to ensure that the field crew has time to double check apparent anomalies, if they occur.
- System audits for the laboratory will be performed regularly and in accordance with NFESC guidance and Department of Defense Quality Systems Manual for Environmental Laboratories (DoD QSM) (2006), as provided in the Laboratory Quality Assurance Plan (LQAP).
- The data validator(s) will review the data to ensure that the analytical results were obtained through the approved methodology and that the appropriate levels of QC were followed. The data review effort will be supervised by the TtNUS Project Chemist.

The Project Manager will oversee the FOL and data reviewer and check that management of the acquired data proceeds in an organized and expeditious manner.

An independent performance audit of field activities may be conducted at the discretion of and under the direction of the CLEAN QAM. If a formal field audit is conducted, the QA Officer will check that sample collection, handling, and shipping protocols, as well as equipment decontamination and field documentation procedures, are being performed in accordance with the approved project planning documents and SOPs. These audits and laboratory systems audits will identify the following:

- The assessed entity (e.g., field crew, office personnel, etc. and the associated project, field event, office, etc.)
- Whether the audit is internal or external
- Location and date(s) of assessment

- Assessment of team members
- Type of assessment
- Scope of assessment
- Documents to be reviewed
- Notification dates
- Proposed assessment schedule
- Assessment number
- Contract number

Performance audits of laboratories are coordinated through NFESC and are conducted every 18 months by NFESC's independent quality assurance contractor.

Assessment findings that require corrective action initiate a sequence of events that include documentation of deficiencies, notification of findings, request for corrective action, implementation of corrective action, and follow-up assessment of the corrective action effectiveness. The procedures for handling any QAPP deviations and project deficiencies that are identified through the planned project assessments are summarized in Worksheet #32.

Potential problems may involve nonconformance with the SOPs and/or analytical procedures established for the project or other unforeseen difficulties. Any person identifying a condition adverse to project quality will notify the PM. The TtNUS PM, with the assistance of the QAM, will be responsible for developing and initiating appropriate corrective action. If the identified deficiencies involve field work, this will be done through the FOL; if the deficiencies involve the laboratory, this will be done through the laboratory project manager. The corrective actions will continue until verification that the corrective action has been effective. Corrective actions may include resampling and/or reanalyzing samples or amending or adjusting project procedures. If warranted by the severity of the problem (e.g., if a change in the approved plan is required), the Navy will be notified in writing and the Navy's approval will be obtained before any change is implemented. Minor changes will be documented for the main file by the TtNUS PM. Additional work that depends on a nonconforming activity will not be performed until the problem has been eliminated. The overall corrective action responsibility for system audits will reside with the PM. The overall corrective action responsibility for field audits will reside with the QAM.

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 Supervision	Site logbook	PM (Linda Klink), TtNUS FOL (TBD), TtNUS	Immediately	Entry in site log book	PM (Linda Klink), TtNUS FOL (TBD), TtNUS	24 hours
Project Supervision	Written report	Program Manager (John Trepanowski), TtNUS Deputy Program Manager (Garth Glenn), TtNUS	Monthly	Written memo	PM (Linda Klink), TtNUS	Within a week of notification
Field Sampling System Audit	Audit checklist and written audit finding summary	PM (Linda Klink), TtNUS FOL (TBD), TtNUS Program Manager (John Trepanowski), TtNUS Deputy Program Manager (Garth Glenn), TtNUS	Dependant on the finding, if major, a stop work may be issue immediately; however, if minor, within 1 week of audit	Written memo	QAM (Tom Johnston),TtNUS Auditor (TBD), TtNUS Program Manager (John Trepanowski), TtNUS	Within 48 hours of notification
Laboratory System Audit	Written audit report	Laboratory Manager, QAM	Not specified by NFESC	Letter	NFESC	Specified by NEFSC

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	After validation is complete	DVM or designee (TtNUS)	PM (TtNUS), project file
Major analysis problem identification (Internal Memorandum)	When persistent analysis problems are detected	Immediately upon detection of problem	Project Chemist	PM (TtNUS), DVM (TtNUS), QAM (TtNUS), and project file
Project monthly progress report	Monthly for duration of the project	Monthly	PM (TtNUS)	Navy, project file
Field status reports	Daily, oral, during the course of sampling	Everyday that field sampling is occurring	FOL (TtNUS)	PM (TtNUS)
Laboratory QA Report	When significant plan deviations result from unanticipated circumstances	Immediately	Subcontracted laboratory	TtNUS, project file

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)
Chain-of-custody forms	The TtNUS FOL or designee will review and sign the chain-of-custody form to verify that all samples listed are included in the shipment to the laboratory and the sample information is accurate. The forms will be signed by the sampler and a copy will be retained for the project file, the Project Manager, and the data validators. See SOP SA-6.3	Internal	TtNUS sampler and FOL
SAP Sample tables	Verify that all proposed samples listed in the SAP tables have been collected.	Internal	TtNUS, FOL or designee
Sample log sheets	Verify that information recorded in the log sheets is accurate and complete.	Internal	TtNUS, FOL or designee
Sample coordinates	Verify that sample locations are correct and in accordance with the SAP proposed locations.	Internal	TtNUS, FOL or designee
Field QC samples	Check that field QC samples listed in Worksheet #20 were collected as required.	Internal	TtNUS, FOL or designee
Chain-of-custody forms	The laboratory sample custodian will review the sample shipment for completeness and integrity and sign accepting the shipment. The data validators will check that the chain-of-custody form was signed/dated by the TtNUS FOL or designee relinquishing the samples and also by the laboratory sample custodian receiving the samples for analyses.	Internal/ External	1 - Laboratory sample custodian 2 - TtNUS, data validators
Analytical data package	All analytical data packages will be verified internally for completeness by the laboratory performing the work. The laboratory QAM will sign the case narrative for each data package.	Internal	Laboratory QAM
Analytical data package	The data package will be verified for completeness by TtNUS data validators. Missing information will be requested from the laboratory and validation will be suspended until missing data are received.	External	TtNUS, data validators
Electronic data deliverables	The electronic data will be verified against the chain-of-custody and hard copy data package for accuracy and completeness.	External	TtNUS, data validators

Verification includes field data verification and laboratory data verification. Verification inputs as per SAP Worksheet 34 will be checked.

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 all sampling SOPs were followed and any field deviations were documented.	TtNUS PM, FOL, or designee
IIa	Analytical SOPs	Ensure that the laboratory followed the analytical SOPs cited in the SAP and any method deviations were approved by TtNUS and documented in the case narrative.	TtNUS Project Chemist or data validators
IIa	Chain-of-custody forms	Ensure that the custody and integrity of the samples were maintained from collection to analysis, that the custody records are complete, and that any deviations are recorded.	TtNUS Project Chemist or data validators
IIa	Holding times	Ensure that the samples were shipped and stored at the required temperature and that sample pH for chemically preserved samples meet the requirements listed in Worksheet #19. Verify that the analyses were performed within the holding times listed in Worksheet #19.	TtNUS Project Chemist or data validators
IIa	Data results	Check the summary form results against the raw data. Check calculations for accuracy.	TtNUS Project Chemist or data validators
IIa	Standards	Ensure that the standards used in the field and laboratory are traceable and meet the contract, method, and procedural requirements.	TtNUS Project Chemist or data validators
IIa/IIb	Laboratory data results for accuracy	Ensure that the laboratory QC samples listed in Worksheet #28 were analyzed and that the measurement performance criteria listed in Worksheet #12 were met for all field samples and QC analyses. Verify that specified field QC samples were collected and analyzed and that the analytical quality control criteria set up for this project were met.	TtNUS Project Chemist or Data Validators
IIa/IIb	Laboratory duplicate analyses for precision	Ensure laboratory precision by checking the RPD or percent difference values from laboratory duplicate analyses, matrix spike/matrix spike duplicates (MS/MSDs), and laboratory control sample/laboratory control sample duplicates (LCS/LCSDs). Ensure compliance with the methods and project MPC accuracy goals listed in Worksheet #12.	TtNUS Project Chemist or data validators
IIa/IIb	Sample results for representativeness	Validate that the laboratory recorded the temperature at sample receipt and the pH of the chemically preserved samples to ensure sample integrity from sample collection to analysis.	TtNUS Project Chemist or data validators
IIb	Project Quantitation Limits (PQLs) for sensitivity	Validate that the project QLs and MDLs listed in Worksheet #15 were achieved.	TtNUS Project Chemist or data validators
IIa/IIb	Project action limits (PALs)	Discuss the impact of matrix interferences or sample dilutions performed because of the high concentration of one or more contaminant, on the other target compounds reported as not detected. Document this usability issue and inform the project manager.	TtNUS Project Chemist or data validators

Step IIa / IIb[1]	Validation Input	Description	Responsible for Validation (name, organization)
IIb	Analytical data deviations	Determine the impact of any deviation from sampling or analytical methods and SOP requirements and matrix interference effects on the analytical results.	TtNUS Project Chemist or data validators
IIb	Sample coordinates	Verify that sample locations are correct and in accordance with the SAP proposed locations.	TtNUS FOL or designee
IIa/IIb	Data validation report	Summarize deviations from methods, procedures, or contracts. Qualify data results based on method or QC deviation and explain all the data qualifications. Print a copy of the qualified project database depicting data qualifiers and data qualifier codes that summarize the reason for data qualifications. Determine if the data met the MPC and determine the impact of any deviations on the technical usability of the data.	TtNUS Project Chemist or data validators

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).

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	Groundwater	VOCs with SIM for 1,1-DCE and 1,4-Dioxane	Tier III data validation. Criteria for SW-846 8260B listed in Worksheets #12, #15, and #28 and the DOD QSM. If not included in Worksheet #12, #15 or #28, the logic outlined in Region 1 EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, December 1996 (EPA, 1996) will be used to apply qualifiers to data.	TtNUS DVM and staff chemists
IIa / IIb	Soil	TOC	EPA Region 1 Tier 1, Verification of Results	TtNUS DVM and staff chemists
IIa and IIb	IDW/Soil	TCLP Organics	Tier I data validation. Data verification for completeness.	TtNUS DVM and staff chemists
IIa and IIb	IDW/Soil	TCLP Inorganics	Tier I data validation. Data verification for completeness.	TtNUS DVM and staff chemists

SAP Worksheet #37 -- Usability Assessment

(UFP-SAP Manual Section 5.2.3)

Data Usability Assessment

The usability of the data directly affects whether project objectives can be achieved. The following characteristics will be evaluated at a minimum. The results of these evaluations will be included in the project report. The characteristics will be evaluated for multiple concentration levels if the evaluator determines that this is necessary. To the extent required by the type of data being reviewed, the assessors will consult with other technically competent individuals to render sound technical assessments of these data characteristics:

Completeness

- For each matrix that was scheduled to be sampled, the FOL acting on behalf of the project team will prepare a table listing planned samples/analyses to collected samples/analyses. If deviations from the scheduled sample collection or analyses are identified the TtNUS PM and risk assessor will determine whether the deviations compromise the ability to meet project objectives. If they do, the TtNUS PM will consult with the Navy RPM and other project team members, as necessary (determined by the Navy RPM), to develop appropriate corrective actions.

Precision

- The Project Chemist acting on behalf of the project team will determine whether precision goals for field duplicates and laboratory duplicates were met. This will be accomplished by comparing duplicate results to precision goals identified in Worksheets #12 and #28, respectively. This will also include a comparison of field and laboratory precision with the expectation that field duplicate results will be no less precise than laboratory duplicate results. If the goals are not met, or data have been flagged as estimated (J qualifier), limitations on the use of the data will be described in the project report.

Accuracy

- The Project Chemist acting on behalf of the project team will determine whether the accuracy/bias goals were met for project data. This will be accomplished by comparing percent recoveries of LCS, LCSD, MS, MSD, and surrogate compounds to accuracy goals identified in Worksheet #28. This assessment will include an evaluation of field and laboratory contamination; instrument calibration variability; and analyte recoveries for surrogates, matrix spike, and laboratory control samples. If the goals are not met, limitations on the use of the data will be described in the project report. Bias of the qualified results and a description of the impact of identified non-compliances on a specific data package or on the overall project data will be described in the project report.

Representativeness

- A project scientist identified by the TtNUS PM and acting on behalf of the project team will determine whether the data are adequately representativeness of intended populations, both spatially and temporally. This will be

accomplished by reviewing where and when the samples were collected as well as the spatial and temporal variations and comparing these to expectations. The usability report will describe the representativeness of the data for each matrix and analytical fraction. This will not require quantitative comparisons unless professional judgment of the project scientist indicates that a quantitative analysis is required.

Comparability

- The Project Chemist acting on behalf of the project team will determine whether the data generated under this project are sufficiently comparable to historical site data generated by different methods and for samples collected using different procedures and under different site conditions. This will be accomplished by comparing overall precision and bias among data sets for each matrix and analytical fraction. This will not require quantitative comparisons unless professional judgment of the Project Chemist indicates that such quantitative analysis is required.

Sensitivity

- The Project Chemist acting on behalf of the project team will determine whether project sensitivity goals listed in Worksheet #15 are achieved. The overall sensitivity and quantitation limits from multiple data sets for each matrix and analysis will be compared. If sensitivity goals are not achieved, the limitations on the data will be described. The Project Chemist will enlist the help of the project risk assessor to evaluated deviations from planned sensitivity goals.

Project Assumptions and Data Outliers

- The TtNUS Project Manager and designated team members will evaluate whether project assumptions were valid. This will typically be a qualitative evaluation but may be supported by quantitative evaluations. The type of evaluation depends on the assumption being tested. Quantitative assumptions include assumptions related to data distributions (e.g., Normal versus log-normal) and estimates of data variability. Potential outliers will be removed if a review of the associated documentation indicates that the results have an assignable cause the renders them inconsistent with the rest of the data.

Describe the evaluative procedures used to assess overall measurement error associated with the project:

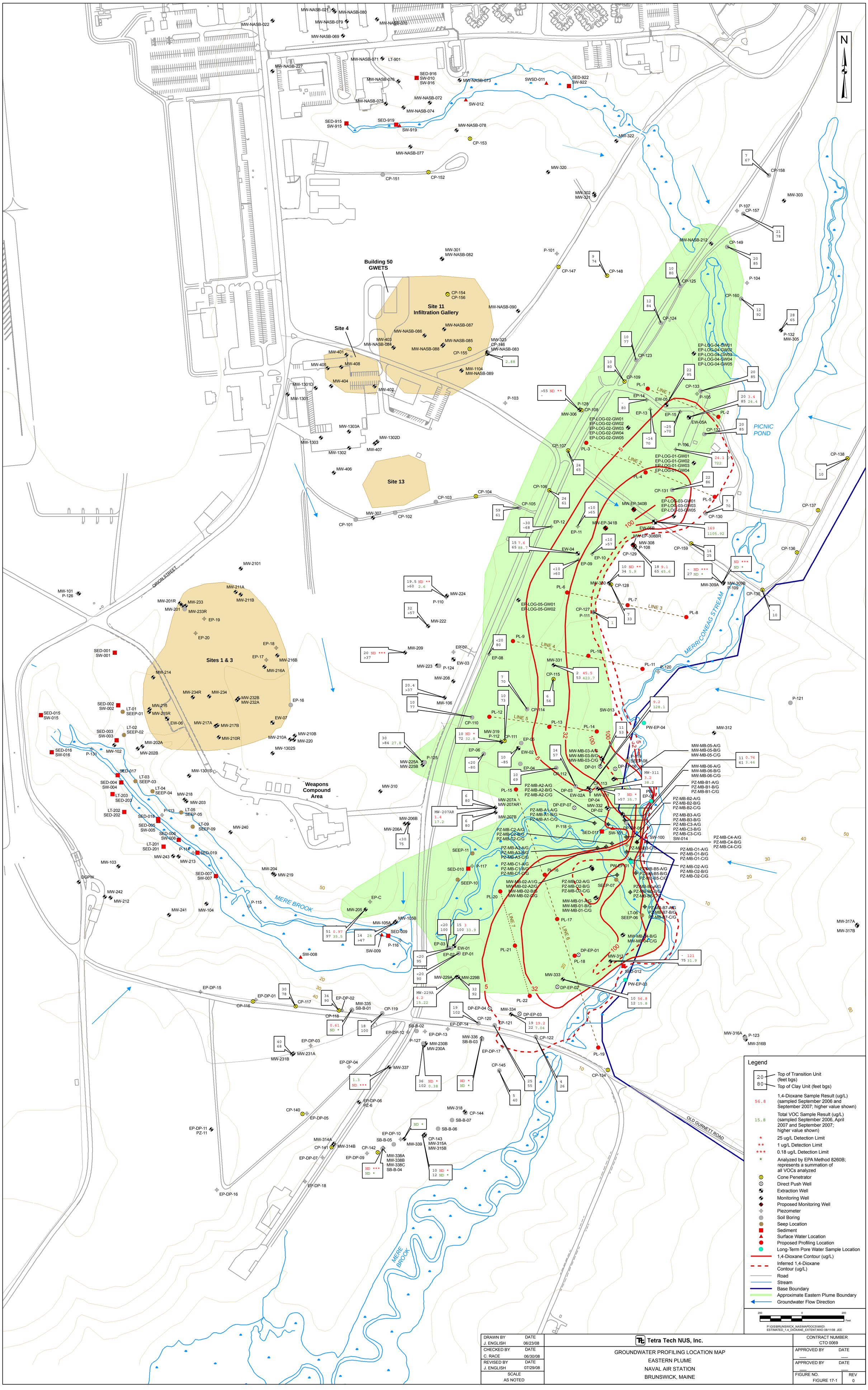
After completion of the data validation, the data and data quality will be reviewed to determine whether sufficient data of acceptable quality are available for decision making. In addition to the evaluations described above, a series of inspections and statistical analyses will be performed to estimate these characteristics. The statistical evaluations will include simple summary statistics for target analytes, such as maximum concentration, minimum concentration, number of samples exhibiting non-detected results, number of samples exhibiting positive results, and the proportion of samples with detected and non-detected results. The project team members identified by the project manager will assess whether the data collectively support the attainment of project objectives. They will consider whether any missing or rejected data have compromised the ability to make decisions or to make the decisions with the desired level of confidence. The data will be evaluated to determine whether missing or rejected data can be compensated by other data. Although rejected data will generally not be used, there may be reason to use them in a weight of evidence argument, especially when they supplement data that have not been rejected. If rejected data are used, their use will be supported by technically defensible rationales.

For statistical comparisons and mathematical manipulations, non-detected values will be represented by a concentration equal to one-half the sample-specific reporting limit. Duplicate results (original and duplicate) will not be averaged for the purpose of representing the range of concentrations. However, the average of the original and duplicate samples will be used to represent the concentration at a particular sampled location.

Statistical tests for outliers will be conducted using standard statistical techniques appropriate for this task. Potential outliers will be removed if a review of field and laboratory documents indicates that they are a result of quality control issues and they are not representative of site conditions. If no physical cause for a statistical outlier can be identified, the data point will not be removed from the data set. However, if the data point is found to truly represent a physical quantity that is different from the rest of the data set, it will be removed.

The suitability of any given statistical test will be assessed based on the completeness of the data sets and the conditions observed at the site. For example, when a single data value is available for soil or water samples at a given sampling location, statistical tests cannot be conducted for that individual sampling location. However, pooling of data across sampling locations may be possible and, if logical to do so, may be implemented at the discretion of the TtNUS project manager. Statistical testing will generally be conducted at the five percent significance level. Statistical testing at other significance levels may also be warranted to provide perspective on the results of testing at 5 percent significance. If other significance levels are used, they will be supported with rationales for their use.

Identify the personnel responsible for performing the usability assessment: The TtNUS Project Manager, Project Chemist, FOL, Project Geologist, Risk Assessors, and others as designated by the Project Manager will be responsible for conducting the listed data usability assessments. The data usability assessment will be reviewed by the Navy RPM, TtNUS Project Manager, the EPA Remedial Project Manager and their contractors, and the MEDEP Project Manager and their contractors. If deficiencies affecting the attainment of project objectives are identified, the review will take place either in a face to face meeting or a teleconference depending on the extent of identified deficiencies. Also, the use of rejected data will be discussed with the regulators. If no significant deficiencies are identified, the data usability assessment will simply be documented in the project report and reviewed during the normal document review cycle.
Describe the documentation that will be generated during usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies: The data will be presented in tabular format, including data qualifications such as estimation (J, UJ) or rejection (R). Written documentation will support the non-compliance estimated or rejected data results. The project report will identify and describe the data usability limitations and suggest re-sampling or other corrective actions to fill the data gaps, if necessary.

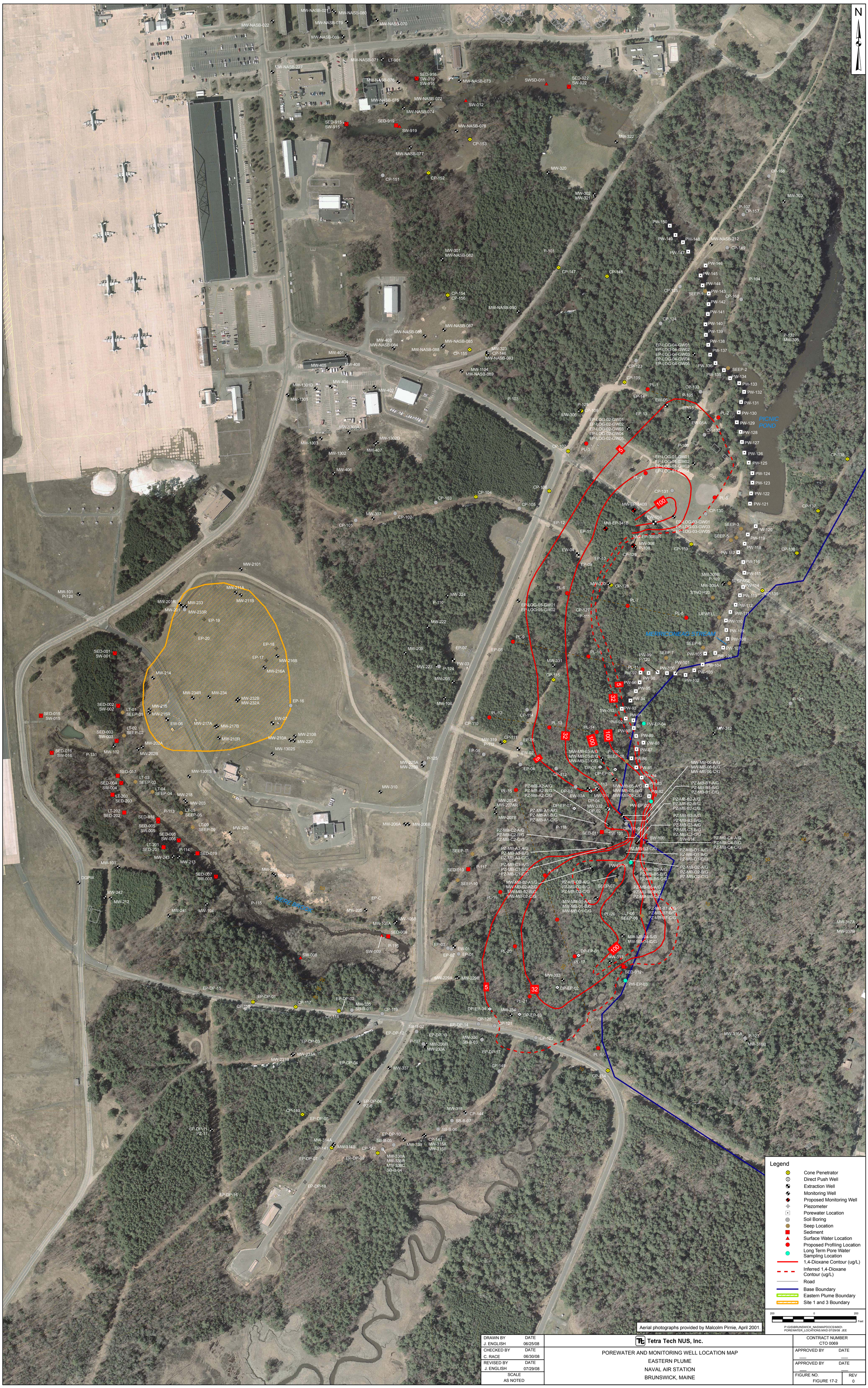


DRAWN BY	DATE
J. ENGLISH	06/23/08
CHECKED BY	DATE
C. RACE	06/30/08
REVISED BY	DATE
J. ENGLISH	07/29/08
SCALE	
AS NOTED	

Tetra Tech NUS, Inc.

GROUNDWATER PROFILING LOCATION MAP
EASTERN PLUME
NAVAL AIR STATION
BRUNSWICK, MAINE

CONTRACT NUMBER	
CTO 0069	
APPROVED BY	DATE
APPROVED BY	DATE
FIGURE NO.	REV
FIGURE 17-1	0



DRAWN BY J. ENGLISH	DATE 06/25/08	Tetra Tech NUS, Inc.	CONTRACT NUMBER CTO 0069
CHECKED BY C. RACE	DATE 06/30/08	POREWATER AND MONITORING WELL LOCATION MAP EASTERN PLUME NAVAL AIR STATION BRUNSWICK, MAINE	APPROVED BY DATE
REVISED BY J. ENGLISH	DATE 07/29/08		APPROVED BY DATE
SCALE AS NOTED			FIGURE NO. FIGURE 17-2 / REV 0

APPENDIX A

RESPONSE TO COMMENTS FROM THE REGULATORS ON DRAFT SAP

Response to EPA Comments dated 09/23/08
Draft Sampling and Analysis Plan (Field Sampling Plan) dated September 2008
for Supplemental Remedial Investigation of 1,4-Dioxane in the Eastern Plume and Bedrock
Naval Air Station Brunswick, Maine

General Comments

1. **Comment:** EPA conducted an accelerated focused review of the work plan in order to accommodate the Navy's fast-track field schedule to implement field activities detailed in the document. Significant and accelerated efforts have recently been made by MEDEP, Navy, and EPA in the way of completion of a comprehensive pore water study of Merriconeag Stream & Picnic pond to support the Navy's planned Eastern Plume ground water transect profiling effort as well as holding highly productive meetings and conference calls to discuss the essential details of the work plan. EPA believes it is critical that the field work detailed in this plan be implemented on schedule. The Navy intends to commence ground water transect profiling field activities on 20 October 2008.

   Response: The Navy appreciates the team effort involved with this project.

2. **Comment:** The document contains many elements (e.g., field and laboratory SOPs) that EPA has already reviewed and provided comments on for other Tetra Tech-developed work plans. EPA has focused only on those elements that substantively affect this work plan's objectives. EPA expects that previous comments on these elements will be addressed in this work plan.

   Response: Agree.

Specific Comments

3. **Comment:** Figure 17-1: A conference call among stakeholders was convened on 15 September 2008 to discuss the draft groundwater transect profiling design outlined in the work plan and the August 2008 pore water sampling results. As a result of the call, agreement was reached on the design of a revised ground water transect profiling plan. Figure 17-1 should be revised and provided to the stakeholders. If deviations from the revised conceptual design changes as a result of "ground truthing" mapped sampling locations, then a second figure should be created to document the variation between the conceptualized design and the field-located sampling design.

   Response: The transect locations shown in the draft UFP-SAP (Revision 0) were refined during the referenced conference call of 09/15/08 (Revision 1) and further refined during ground "truthing" later that same week (Revision 2). Figures reflecting the respective refinements of the 9/15/08 conference call and further refinements after ground "truthing" were provided by the Navy in an e-mail dated 10/6/08 and were discussed during a conference call later that same day.

   As discussed during the 09/15/08 conference call, the SAP text and figures will not be revised because the intent is not to make the UFP-SAP a living document, which would otherwise result in an extensive rewrite of the SAP. The documentation of the pore water sampling and subsequent refinements to the groundwater profiling transects will be documented in the future RI Report.

The latest version of the groundwater profiling transect map (Figure 17-1, Revision 2) is attached to this comment response letter, supersedes Revision 0 within the SAP, and will be used by the field team in conducting the field investigation. Also attached is an accompanying table entitled RELOCATION OF PROFILING TRANSECT POINTS BASED ON PORE WATER RESULTS documenting the final locations of the profiling points with cross references to locations provided in the SAP.

Note that the attached Figure 17-1, Revision 2, now incorporates recently updated Base CADD mapping of roads, buildings, and Base boundaries. Also, as per MEDEP Specific Comment #27, the attached Figure 17-1, Revision 2, corrects the location for Mere Brook piezometers "PZ-MB-B4-A/B/C," which had been mislabeled as "PZ-MB-O2-A/B/C".

4. **Comment:** Figure 17-2: This figure should be revised to include the actual GPS'ed pore water and seep sampling locations collected by MEDEP and EPA. EPA has previously provided the coordinates to the Navy in an e-mail. EPA also requests that those pore water & seep locations that had hits of 1,4-Dioxane and/or VOCs be added to the figure. Pore water chemical data was also provided to the Navy in the e-mail.

   Response: Please see the response to EPA Specific Comment #3. As discussed during the 09/15/08 conference call, the SAP text and figures will not be revised because the intent is not to make the UFP-SAP a living document. The actual pore water locations and associated analytical results will be documented in the future RI Report.

5. **Comment:** Bedrock Wells Investigation Figure: The work plan describes but does not depict on a figure the locations of the bedrock well couplets proposed in the vicinity of bedrock monitoring well MW-308. EPA requests that a potentio-metric surface map with the proposed well pairs depicted on it be prepared and included in the work plan. The well pair locations described in the work plan are consistent with agreements reached during the February 2008 1,4-dioxane & bedrock meeting. A conceptual cross-section figure would also be useful in depicting the bedrock surface in the vicinity of MW-308 and how these wells will be screened in the overburden and bedrock. These additional figures do not need to be added to the work plan now in order to execute the ground water transect profiling work but should be transmitted to the regulators for review and comment prior to implementing the bedrock ground water investigation. EPA understands that MEDEP has viewed the staked locations in the field but EPA was unaware that these locations had been field-located.

   Response: The Navy agrees to add bedrock cross-sections and the bedrock surface contour map to Appendix C-2 Historical Geology/Hydrogeology of the SAP. The information was extracted from the ECC presentation at the 02/28/08 technical meeting. In addition, a new "zoom-in" figure is attached showing the planned bedrock cluster locations. The "zoom-in" figure was developed from Figure 17-2 and, as a result, has been numbered Figure 17-2A.

   A potentiometric surface map has not been prepared due to the lack of monitoring wells in the vicinity of the proposed bedrock well locations. At each of the three bedrock well locations, both an overburden and bedrock well(s) will be completed. The overburden well will be screened above bedrock in sandy materials; the open bedrock borehole in each well cluster will be completed 50 feet into competent bedrock. The overburden and weathered bedrock in the open-hole bedrock boring will be sealed off with 6-inch steel casing grouted 2 feet into competent bedrock. One or two nested monitoring wells will be completed in each open-hole bedrock boring based on borehole geophysical logging.

6. **Comment:** Page 90 of 147, SAP Worksheet #17, Task 1 – Pore Water Sampling: Although not critical to the execution of the work plan field activities, Task 1 – Pore Water Sampling, has been completed. The results could be summarized with the revised Figure 17-2 requested in Comment #4 above.

   Response: Please see the response to EPA Specific Comment #3. As discussed during the 09/15/08 conference call, the SAP text and figures will not be revised because the intent is not to make the UFP-SAP a living document. The results will be documented in the future RI Report.

7. **Comment:** Page 91 of 147, SAP Worksheet #17, Task 4 – Overburden Monitoring Well Construction and Well Survey: It states that all new monitoring wells be sampled using bladder pumps following low-flow sampling protocols but SOP #SA-1.1 indicates that submersible pumps are to be used for 2-inch diameter and larger wells. EPA has commented in other ground water sampling work plans that submersible bladder pumps are readily available for 1" direct push wells. QED and other manufacturers offer these small diameter pumps. EPA recommends the use of small diameter bladder pumps over peristaltic pumps to sample for VOCs.

   Response: The use of bladder pumps is planned, as stated in SAP Worksheet #17, and the pumps are readily available from a local equipment rental company.

Response to MEDEP Comments dated 09/26/08
Draft Sampling and Analysis Plan (Field Sampling Plan) dated September 2008
for Supplemental Remedial Investigation of 1,4-Dioxane in the Eastern Plume and Bedrock
Naval Air Station Brunswick, Maine

General Comments

1. **Comment**: MEDEP did not comment on the profile locations (PL) proposed in this workplan for the geophysical transects because these locations and transects were refined during a conference on September 15, 2008 between MEDEP, EPA, the Navy and Carolyn Lepage, consultant for the Brunswick Area Citizens for a Safe Environment. MEDEP anticipates that the agreed upon changes will be reflected in the final SAP.

   Response: The following response is a repeat of the Navy's response to EPA Specific Comment #3:

   The transect locations shown in the draft UFP-SAP (Revision 0) were refined during the referenced conference call of 09/15/08 (Revision 1) and further refined during ground "truthing" later that same week (Revision 2). Figures reflecting the respective refinements of the 9/15/08 conference call and further refinements after ground "truthing" were provided by the Navy in an e-mail dated 10/6/08 and were discussed during a conference call later that same day.

   As discussed during the 09/15/08 conference call, the SAP text and figures will not be revised because the intent is not to make the UFP-SAP a living document, which would otherwise result in an extensive rewrite of the SAP. The documentation of the pore water sampling and subsequent refinements to the groundwater profiling transects will be documented in the future RI Report.

   The latest version of the groundwater profiling transect map (Figure 17-1, Revision 2) is attached to this comment response letter, supersedes Revision 0 within the SAP, and will be used by the field team in conducting the field investigation. The latest version of the groundwater profiling transect map (Figure 17-1, Revision 2) is attached to this comment response letter, supersedes Revision 0 within the SAP, and will be used by the field team in conducting the field investigation. Also attached is an accompanying table entitled RELOCATION OF PROFILING TRANSECT POINTS BASED ON PORE WATER RESULTS documenting the final locations of the profiling points with cross references to locations provided in the SAP.

   Note that the attached Figure 17-1, Revision 2, now incorporates recently updated Base CADD mapping of roads, buildings, and Base boundaries. Also, as per MEDEP Specific Comment #27, the attached Figure 17-1, Revision 2, corrects the location for Mere Brook piezometers "PZ-MB-B4-A/B/C," which had been mislabeled as "PZ-MB-O2-A/B/C".

2. **Comment**: The figures provided with the workplan provided a great deal of information but it is hard to discern the proposed sampling locations. MEDEP suggests including an insert to Figure 17-1 that "zooms in" on the confluence area, where there is a concentration of points.

   Also, a plain 11x17 figure showing only the proposed new borings/wells and the Mere Brook wells targeted for sampling would help clarify the work proposed for the RI. Removing the tag data and showing only a limited number of LTM points would make it easier to identify where additional data will be obtained.

Response: The suggested enhancements will be made for figures presented in the RI Report. The UFP-SAP figures will remain as is due to the expedited nature of this project. Note that Worksheet #18 (and the attached table documenting refinements from the 9/15/08 conference call and subsequent ground truthing) serves as a "checklist" of the wells to be installed and sampled. Also note that the locations have already been staked in the field.

3. **Comment**: Please provide MEDEP three hard copies of the final workplan.

   Response: Response: Agree. Two copies will be provided to Claudia Sait and one copy will be provided to Chris Evans.

4. **Comment:** MEDEP requests that the Remedial Investigation (RI) and future Tech Memo data be submitted electronically along with the draft hard copies. Results must be provided electronically in the Department's Environmental Data Deliverable (EDD) format for the Environmental and Groundwater Database (EGAD). This should include field parameter data, water level data, and laboratory analytical data. Quality control sample results such as surrogate recoveries must be in percent. Specific EDD formats and additional information can be found on the Department's EGAD web page at http://www.maine.gov/dep/rwm/egad/.

   Response: The Navy agrees to submit electronic versions of the documents.

   Results will be provided in the MEDEP EDD format as requested and will include field parameter data, water level data, and laboratory analytical data. This information will be provided as the deliverable "Raw Data provided to EPA/MEDEP" identified on SAP Worksheet #16 – Project Schedule. A footnote will be added to Worksheet #16 describing the contents of the raw data deliverable as well as a reference to the MEDEP EGAD web page.

   Surrogate recoveries are in percent, as shown on Worksheet #28.

5. **Comment**: In addition to supplying both their mobile lab and fixed lab for analyses EPA spearheaded the porewater sampling in August 2008. MEDEP only assisted in the porewater collection effort. This should be changed throughout the document.

   Response: Comment noted.

6. **Comment**: The field operations leader (FOL) is not identified in the SAP. Once Tetra Tech NUS has determined who will be the FOL for this SAP they must send an email identifying the person and providing the necessary contact information to the stakeholders.

   Response: Agree.

Specific Comments:

7. **Comment**: Page 5, SAP Worksheets: Please update the page numbers for the SAP worksheets.

Response: The table of contents has been updated to reflect revision pages.

8. **Comment**: Page 32, Worksheet (WS) 10, History, para 2: "The GWETS has been successful in achieving hydraulic control..."

The GWETS has not achieved complete hydraulic control as evidenced by the confirmed discharge zones near the confluence of Mere Brook and Merriconeag Stream. Please delete this portion of the sentence.

Response: The sentence will be revised to...."The GWETS ~~has been successful in achieving~~ *objective is to achieve* hydraulic control (via extraction) and ~~in reducing~~ *to reduce* hot-spot concentrations ....."

9. **Comment**: Page 33, WS 10 – Problem Definition, para. 1: *"Elevated concentrations of 1,4-dioxane (180 ug/L) compared to criteria..."*

During the April 2005 monitoring event, the data for the GWETS combined effluent were 43 J ug/L and 20 J ug/L (duplicate). These data and the potential for untreated discharge of 1,4 dioxane exceeding the Maine criteria were also important triggers for this RI. Please revise the above statement to reflect this data.

Response: The following sentence will be added to Worksheet #10, History, 3rd paragraph just before the sentence beginning "As a result...":

"Also, during the April 2005 monitoring event, the data for the GWETS combined effluent were 43 J ug/L and 20 J ug/L (duplicate), resulting in a concern about the potential for untreated discharge of 1,4-dioxane."

The same sentence will be added to Worksheet #10, DQO Step 1 (Day 1), State the Problem, Background, following the first sentence of the first paragraph.

10. **Comment**: Page 41, WS 10, Problem Definition, para. 2: *"This is the only bedrock well with detected VOCs."*

This statement is incorrect and contradicts earlier text in the section (MW-323 has also had persistent low level VOC detections). Please revise the text to note that MW-308 is the only location where VOCs have exceeded regulatory criteria.

Response: Agree.

11. **Comment**: Page 45, WS 10, Bedrock Contamination..., para 2, first sentence: Does the Navy mean "The team discussed whether *it* is ..."? Please correct.

Response: "if" will be revised to "*whether.*"

12. **Comment**: Page 46, WS 10, Bedrock Contamination..., bullet 3: Please remove the name of the homeowner from this bullet and replace it with Residential Well (RW) 1 to ensure the privacy of the homeowner.

Response: Agree. There are two instances in the referenced paragraph. In addition, the change will be made to page 31, Action Item 14.

13. **Comment**: Page 56, WS 11, Step 7: Develop the Plan... bullet 4: "Permanent monitoring wells for groundwater will be LTMP for the Eastern Plume."

a.) Does the Navy mean "Permanent monitoring wells for groundwater will be *included in the* LTMP for the Eastern Plume." ? Please clarify.

b.) Also when the new monitoring wells are considered for the LTMP, some of the Mere Brook wells also must be considered for inclusion.

Response:

a.) and b.) "*The Supplemental RI* permanent monitoring wells for groundwater will be *added to* LTMP for the Eastern Plume; *also, currently existing Mere Brook wells will also be considered for inclusion.*"

14. **Comment**: Page 57, WS 11, Decision Statement #1: "Abandonment of monitoring well MW-308 may be delayed until the team evaluates data from the new well couplets..."

MEDEP meeting notes clearly state that the group did not want MW-308 abandoned until after the group reviewed the data and made a final decision on the adequacy of the new wells. Please revise accordingly.

Response: The Navy prefers to first abandon MW-308 before proceeding with installation of the replacement well primarily because if the structural integrity of MW-308 is compromised, contaminated groundwater may be pulled in via fractures by the replacement well. Also, if the abandonment of MW-308 is deferred, a separate mobilization effort would be necessary in the Spring of 2009. An alternative viewpoint is that the continued record of monitoring results at this well would be lost by abandoning the well. This issue was discussed during the 10/06/08 conference call without resolutions. The team agreed to give it further thought and make a decision prior to the start of field work.

The existing text will be revised as follows:

"Determine if bedrock groundwater is contaminated in the vicinity of MW-308. If the bedrock is contaminated, then continue bedrock investigation. If the bedrock is not contaminated, then terminate bedrock investigation. *The team will decide if and when to* ~~and~~ abandon MW-308*; this decision will be made by the team prior to initiation of the bedrock investigation field work.* Abandonment of monitoring well MW-308 may be delayed until the team evaluates data from the new well couplets (i.e., paired wells at a given location monitoring different depth intervals). The new overburden and bedrock wells will be located approximately 10 feet northwest (upgradient) of MW-308 to avoid potential impacts from MW-308 that may have compromised well integrity. *Conversely, MW-308 may first be abandoned before proceeding with installation of the*

replacement well primarily because if the structural integrity of MW-308 is compromised, contaminated groundwater may be pulled in via fractures by the replacement well."

15. **Comment**: Page 58, WS 11, Site 11 Tasks, bullet 2: Does the Navy mean "Discrete *data* from one bedrock borehole location with one or two monitoring wells *screens* installed in the same borehole..." Please check for clarity and correct.

    Response: The correction will be made as suggested.

16. **Comment**: Page 58, WS 11, MW-308 Area, bullet 1: "One couplet is required to address the vicinity of MW-308 (and existing monitoring well MW-308 will be abandoned because it does not monitor the bedrock)."

    It is unclear if construction of MW-308 compromised the well and overburden groundwater may be getting into the bedrock well but at this time it is unknown if the VOC concentration accurately reflect bedrock groundwater concentrations in the area of MW-308 or not. Therefore MEDEP suggests the following language: "One couplet is required to address the vicinity of MW-308 (and existing monitoring well MW-308 will be abandoned ~~because~~ *if* it does not monitor the bedrock)."

    Response: Please see the Navy's response to MEDEP Specific Comment #14. The sentence will be revised as follows: "One couplet is required to address the vicinity of MW-308 (and *the team will make a decision as to if and when* existing monitoring well MW-308 will be abandoned)."

17. **Comment**: Page 58, WS 11, Project Quality Objectives..., Decision Statement #4 and Page 59, Bullet 6: The text indicates that no transducer data collection is anticipated for this RI to evaluate whether the residential wells are hydraulically linked to the wells on the west side of Mere Brook. When the report for the groundwater model is issued, the Aquifer Recovery Test data for MW-308 and MW-309B may be able to provide an initial indication of whether there are any pumping effects influencing groundwater elevations in bedrock near MW-308. The Navy must include and evaluate this data as part of this RI.

    Response: The Navy agrees to the evaluation in the RI but may reference the modeling report rather than include this data. The following sentence will be added to the last bullet of MW-308 Area: *"When the report for the groundwater model is issued, the Aquifer Recovery Test data for MW-308 and MW-309B may be able to provide an initial indication of whether there are any pumping effects influencing groundwater elevations in bedrock near MW-308; these results will be referenced and evaluated as part of this RI."*

18. **Comment**: Page 59, WS 11, MW-308 Area, bullet 5:

    a.) First sentence please change is to *if* and in the second sentence please change steam to *stream.*

    b.) "However, for any new wells, hydraulic head data will be collected at surveyed Merriconeag Stream steam [sic] gauges, and other accessible wells east of Merriconeag Stream to evaluate groundwater flow direction."

Also see comment 17 above. MEDEP will accept this in place of the transducers if the Navy guarantees that it will be willing to pull the pumps from the residential drinking water wells as necessary and replace them to the owner's satisfaction. Otherwise this remains a critical data gap identified during the DQO process.

Response:

a.) and b.) The typographical errors will be fixed.

The following paragraph will be added after the 3rd paragraph of Task 5: "*Water levels will be manually collected from residential wells. Small-diameter PVC drop pipes will be temporarily inserted into the well and the water level probe lowered into the well through the drop tube, to minimize the potential for the cable to become entangled with the in-place pump piping and wiring. Should the construction of the well be such that a water level measurement cannot be obtained unless the pump is removed, the Tetra FOL will inform the Tetra Tech C.G., who will then consult MEDEP. A water level from a residential well will not be obtained unless the well is deemed critical to the effort (i.e. there are no other nearby wells that can be used instead). If pump removal is required, the pump will be reinstalled (after water level collection) as per the pre-existing conditions and the well will be checked to ensure that it is fully operational.*"

19. **Comment**: Page 59, WS 11, General Information: "Inventory and information, if available, from the following sources:"

Please review the syntax and correct.

Response: "*will be obtained*" will be inserted into the sentence.

20. **Comment**: Pages 60 & 61, DQO Step 4: MEDEP requests that the Navy consider adding a bedrock designation to the new bedrock wells identifier (e.g., MW-EP-340 BR and MW-EP-308-BR-R).

Response: DQO Step 4 provides the bedrock well locations as MW-EP-308R, MW-EP-340, and MW-EP-341. Because clusters will be installed at these locations, there is a need to add identifiers for each well at a given cluster. Worksheet #18 provides the detailed numbering system where the suffix "S" for shallow represents the overburden bedrock well, the suffix "B1" represents the shallow bedrock well, and the suffix "B2" represents the deep bedrock well.

21. **Comment**: Pages 62, Decision Rule #2, 1st sentence: Please delete the extra "and".

Response: Agree.

22. **Comment**: Page 62, Decision Rule #3, 1st sentence: Please identify if it is the bedrock wells that the contamination must be present or not present.

Response: The sentence will be clarified to "If the contaminants *in the bedrock wells or bedrock well clusters* are not present..."

23. **Comment**: Page 63, WS 11, Project Quality Objectives..., DQO Step 6, para. 2: The text implies there will be a confirmation with the project team for the bedrock well locations after the groundwater profiling task is complete, however recent discussion indicates that the bedrock drilling may proceed prior to completion of the direct push profiling. Also MEDEP's hydrogeologist met with Tetra Tech representatives and staked initial locations during a recent site visit so if the bedrock drilling is to proceed concurrently with the profiling please revise the text.

Response: The sentence will be revised as follows: "It was decided during the conference call that the bedrock drilling locations would be recommended to the project team after the results from the pore water ~~and groundwater profiling~~ task~~s~~ ~~are~~ *is* reviewed *and groundwater profiling transects are refined.* ~~and after h~~*Historical* information from the Sites 4, 11, and 13 source areas ~~are reviewed~~ *will be considered in evaluating results of the bedrock investigation.*"

Note that the draft SAP was written to install the bedrock wells after the pore water and groundwater profiling effort. To expedite the project, the bedrock well installation is now to occur concurrently with the groundwater profiling effort.

24. **Comment**: Page 64, 11, Project Quality Objectives..., Bullet 3: Please revise the text to clarify all locations will have borehole geophysics and one of those will be cored for the top 30 feet.

Response: Agree.

25. **Comment**: Page 69, WS 14, Off-Site Laboratory Data Package Deliverables: Please ensure that the NIRIS deliverable is compatible and contains the necessary data for MEDEP's EDD which is required by 10 MRSA section 9418(1) and 9418(2)(A). (Also see comment 4 above.)

Response: Please see the response to MEDEP General Comment #4. While the NIRIS deliverable does contain the necessary data, Tetra Tech will export the data into the format required for the MEDEP's EDD and this note of clarification will be added to Worksheet #14.

26. **Comment**: Page 82, WS 15, Reference Limits and Evaluation Table:

a.) For clarity please define Method Detection Limit in the footnotes for this worksheet.

b.) For vinyl chloride, since the project quantitation limit and the method detection limit are higher than the regulatory limit please report any detections to the method detection limit.

Response:

a). Agree to define MDL in a footnote to the worksheet.

b). Agree to report vinyl chloride detections to the MDL.

27. **Comment**: Page 90, WS 17, Sample Design, Task 2 and Figures 17-1 & 17-2: The location for Mere Brook piezometers "PZ-MB-B4-A/B/C" has been mislabeled as "PZ-MB-O2-A/B/C". Please revise both figures.

Response: The change will not be made to the SAP figure. However, the change has been captured for Figure 17-1 Revision 2 attached to this comment response letter and will be carried forth to the Supplemental RI Report.

28. **Comment**: Page 91, WS 17, Sample Design, Task 3, Well Construction Bullet #2: Please revise to note that the well designation is MW-308R and that the top 30 feet of rock will be cored as stated in WS 11, page 61 first paragraph.

Response: Agree.

29. **Comment**: Pages 94-106, WS 18, Sampling Locations...: Please revise the table entries for the "PL" sample points along the profile lines. The sample design does not call for well installation at those points, so there will not be any "synoptic water level" data, although a snapshot water level can be obtained through the drill rods. The SOP references must be revised as needed for sampling with the direct push probe rather than from a temporary well. MEDEP suggests changing the description in the "Rationale for Sampling Location" from "new temporary overburden well" to "discrete groundwater sample" to clarify the proposed sample technique.

Response: For the PL locations, an asterisk will be added along with a descriptor at the end of the table indicating: "* *Although synoptic water levels cannot be collected for these discrete groundwater sampling locations, snapshot water-levels can be obtained through the drill rods and these water levels will be documented in the Supplemental RI Report.*"

30. **Comment**: Page 107, WS 18, Sampling Locations...[Bedrock section] Rationale for Sampling Location: "Consult with TtNUS MECG regarding final well location and well screen interval, which may be changed based on borehole geophysical and groundwater profiling results. Significant changes in well locations/screens depth are subject to prior concurrence of MEDEP and EPA."

This statement conflicts with footnote #4 which states: "The TtNUS Maine Certified Geologist and MEDEP Geologist (Chris Evans) will be consulted by the TtNUS field Operations Leader to establish the screen length and interval for each new monitoring well..." Please revise the Rationale for Sampling Locations ... to make it compatible with the footnote.

Response: The worksheet cells for bedrock well rationale will be replaced with: "*See footnote 4. Final well location and well screen interval may be changed based on borehole geophysical results.*"

31. **Comment**: Pages 108-109, WS 18, Sampling Locations...: Please update and provide this information with the groundwater profiling results.

Response: As discussed during the 09/15/08 conference call, the SAP figures will not be revised because the intent is not to make the UFP-SAP a living document. The refinements will be documented in the future RI Report.

The latest version of the groundwater profiling transect map (Figure 17-1, Revision 2) is attached to this comment response letter, supersedes Revision 0 within the SAP, and will be used by the field team in conducting the field investigation. Also attached is an accompanying table

documenting the final locations of the profiling points with a cross reference to locations provided in the SAP.

32. **Comment**: Page 111, WS 20, Field Quality Control Sample Summary Table: No proficiency (PT) samples are proposed for this project. One 1,4 Dioxane PT sample must be included since 1,4 dioxane is the main focus of this study and is difficult analytically.

Response: Given that an appropriate level of quality control (including laboratory control samples, matrix spikes and replicate samples) is included in the analytical scheme, the Navy respectfully disagrees that PT samples are necessary at this time. If, however, analytical problems arise that indicate a laboratory deficiency, PT sample use will be considered in concert with the state of Maine and EPA. Footnote 5 on Worksheet #20 will be clarified in this regard.

33. **Comment**: Page 128 & 129, WS 28, Laboratory QC Samples Table: Surrogate recoveries for 1, 4 dioxane and 1,2-dichloroethane must not be lower than the Method/SOP QC Acceptance limits. Please revise.

Response: For surrogate recoveries, the last column of the table "Measurement Performance Criteria" should match the third column "Method/SOP QC Acceptance Limits" and will be corrected.

34. **Comment**: Page 146, WS 37, Usability Assessment: "Potential outliers will be removed if a review of the field and laboratory documents indicates that the results are true outliers."

Please revise as follows: "Potential outliers will be removed if a review of the field and laboratory documents indicates that they are a result of quality control issues and they are not representative of site conditions."

Response: Agree.

Response to Follow-up Navy Chemist Comment verbally provided on 09/15/08
Draft Sampling and Analysis Plan (Field Sampling Plan) dated September 2008
for Supplemental Remedial Investigation of 1,4-Dioxane in the Eastern Plume and Bedrock
Naval Air Station Brunswick, Maine

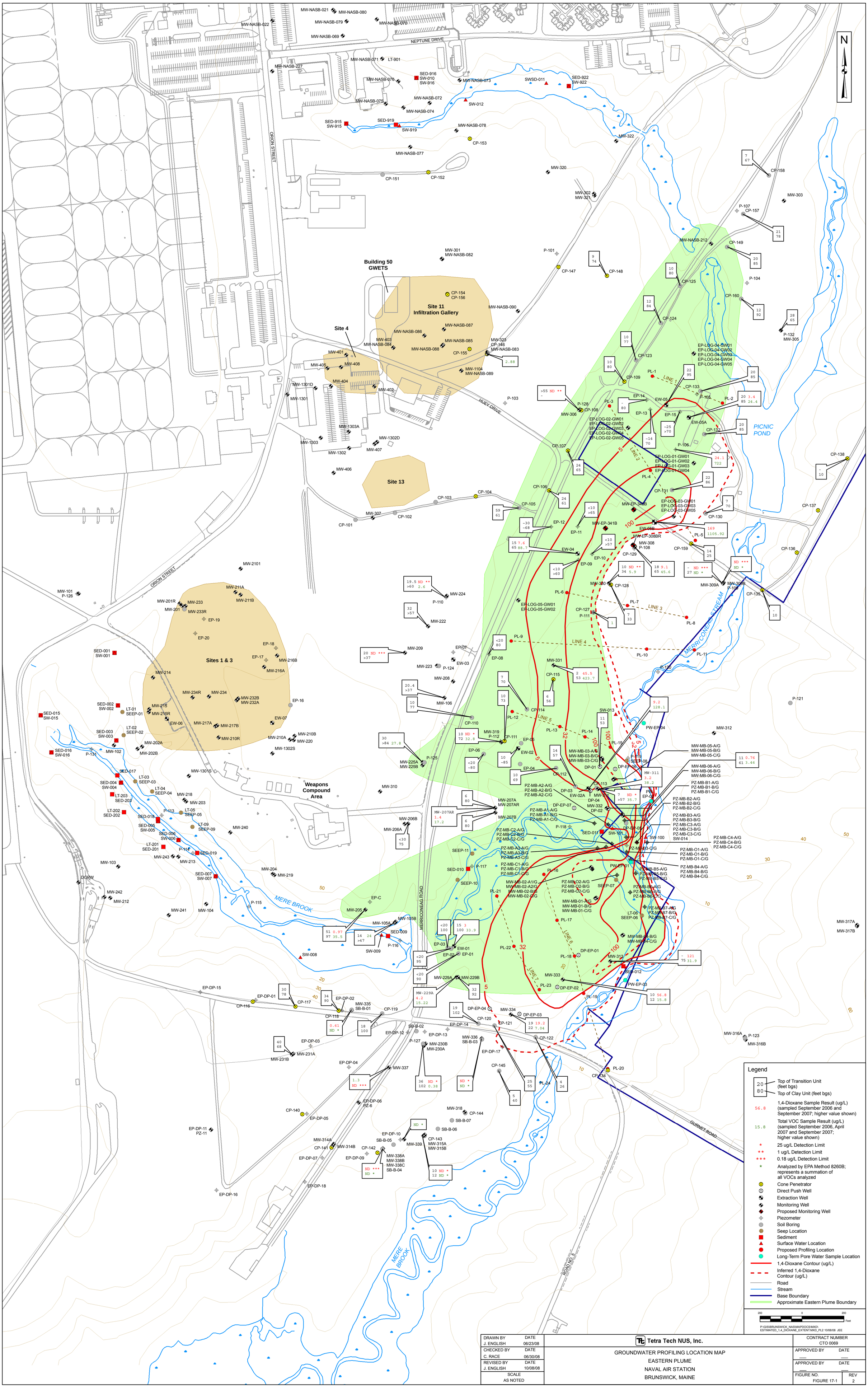
The Navy Chemist reviewed the SAP prior to submittal of the draft to the EPA and MEDEP. A follow-up comment was verbally received by Tetra Tech NUS on 09/15/08 and will be incorporated into the final SAP, as follows:

- SAP Worksheet #9.2 – Project Scoping Session Participants Sheet: As per the request of the Navy Chemist, point of contact information will be added directly to the table rather than referring the reader to point of contact information on SAP Worksheet #3 – Distribution List.

Appendix A – Attachment 1

Relocation of Profiling Transect Points Based on Pore Water Results

- Figure 17-1, Revision 2
- Table


N
NEPTUNE DRIVE
ORION STREET
HUEY DRIVE
MERRICONEAG ROAD
GURNET ROAD
ROAD NO. 5
MERE BROOK
MERRICONEAG STREAM
PICNIC POND
Building 50 GWETS
Site 11 Infiltration Gallery
Site 4
Site 13
Sites 1 & 3
Weapons Compound Area
LINE 1
LINE 2
LINE 3
LINE 4
LINE 5
LINE 6
LINE 7
Legend
Top of Transition Unit (feet bgs)
Top of Clay Unit (feet bgs)
1,4-Dioxane Sample Result (ug/L) (sampled September 2006 and September 2007; higher value shown)
Total VOC Sample Result (ug/L) (sampled September 2006, April 2007 and September 2007; higher value shown)
25 ug/L Detection Limit
1 ug/L Detection Limit
0.18 ug/L Detection Limit
Analyzed by EPA Method 8260B; represents a summation of all VOCs analyzed
Cone Penetrator
Direct Push Well
Extraction Well
Monitoring Well
Proposed Monitoring Well
Piezometer
Soil Boring
Seep Location
Sediment
Surface Water Location
Proposed Profiling Location
Long-Term Pore Water Sample Location
1,4-Dioxane Contour (ug/L)
Inferred 1,4-Dioxane Contour (ug/L)
Road
Stream
Base Boundary
Approximate Eastern Plume Boundary
Feet
P:\GIS\BRUNSWICK_NAS\MAPDOCS\MXD\ESTIMATED_1,4_DIOXANE_EXTENT.MXD_PL2 10/08/08 JEE
DRAWN BY J. ENGLISH DATE 06/23/08
CHECKED BY C. RACE DATE 06/30/08
REVISED BY J. ENGLISH DATE 10/08/08
SCALE AS NOTED
Tetra Tech NUS, Inc.
GROUNDWATER PROFILING LOCATION MAP
EASTERN PLUME
NAVAL AIR STATION
BRUNSWICK, MAINE
CONTRACT NUMBER CTO 0069
APPROVED BY DATE
APPROVED BY DATE
FIGURE NO. FIGURE 17-1
REV 2

RELOCATION OF PROFILING TRANSECT POINTS BASED ON PORE WATER RESULTS
EASTERN PLUME
NAS BRUNSWICK, MAINE

Profile Location ID (new)	Profile Location ID (as per Draft SAP, refer to Figure 17-1, Revision 0)	Rationale for Relocation (Refer to Figure 17-1, Revision 1)	Further Modification based on Field Conditions (Refer to Figure 17-1, Revision 2)
PL-1	PL-1 Transect Line #1	Move Transect Line #1 north 75' to better target between PW-130 and PW-131 where VOCs were detected during pore water sampling. (PW-132 = ND; PW 131> PW-130>PW129). Also will address data gap in clay layer.	No change.
PL-2	PL-2 Transect Line #1	See rationale PL-1.	No change.
PL-3	PL-3 Transect Line #2	Swivel transect line to move location out of cemetery.	No change.
PL-4	PL-4 Transect Line #2	Swivel transect line to move location out of cemetery.	No change.
PL-5	PL-5 Transect Line #2	Move to complete alignment with relocated points PL-3 and PL-4. Also, new location will be better positioned to target PW-SP-14 where 1,4-dioxane was detected during pore water sampling.	No change.
PL-6	PL-6 Transect Line #3	No change	No change.
PL-7	PL-7 Transect Line #3	No change	No change.
PL-8	PL-8 Transect Line #3	No change	No change.
PL-9	PL-9 Transect Line #4	No change	No change.
PL-10	PL-10 Transect Line #4	Move northeast to target cluster PW-SP02 and PW-106 where 1,4-dioxane was detected during pore water sampling (plus VOCs at PW-SP-05 and PW-SP-03). (New location approximately 75' west of PW-SP-05).	No change.
PL-11	PL-11 Transect Line #4	Move northeast to target cluster PW-SP02 and PW-106 where 1,4-dioxane was detected during pore water sampling. Move to complete alignment with PL-9 and relocated PL-10.	No change.
PL-12	PL-12 Transect Line #5	Slide east along transect to a point between CP110 and CP-114 to be closer to the plume.	No change.
PL-13	PL-13 Transect Line #5	Slide east 60' along transect considering PL-12 move to the east.	No change.
PL-14 (New-3)	---	New point further east of PL-14 along the transect (if possible based on field conditions) to target PW-SP-09 where unique cold water discharge, preferential pathway, and VOCs were detected during pore water sampling. Location to be field located after results from Transect Lines #5 is available.	No change.

Profile Location ID (new)	Profile Location ID (as per Draft SAP, refer to Figure 17-1, Revision 0)	Rationale for Relocation (Refer to Figure 17-1, Revision 1)	Further Modification based on Field Conditions (Refer to Figure 17-1, Revision 2)
PL-15	PL-14 Transect Line #5	No change (Need to retain to target anticipated narrow 100 ug/L band of 1,4-dioxane contamination).	No change.
NA	PL-15 Transect Line #6	Eliminate point because geology is known in the area and newly added points based on pore water sampling are more important than this location.	No change.
PL-16	PL-16 Transect Line #6	No change	No change.
PL-17	PL-17 Transect Line #6	No change	No change.
PL-18	PL-18 Transect Line #6	Move point off of the transect to nearby DP-EP-01S/D. No EC profiling necessary (GW sampling only) since DP-EP-01 lithology is available	No change.
PL-19 (New-4)	---	New point along transect to target leading edge of the most contaminated portion of the plume (> 100 ug/L contour)	No change.
PL-20	PL-19 Transect Line #6	No change	No change.
PL-21	PL-20 Transect Line #7	No change	No change.
PL-22	PL-21 Transect Line #7	Move between MW-333 and MW-334 (move roughly 75' northeast at 32 ug/L contour line). Important to obtain more vertical data (MW-334 at 47 feet while PL22 indicates a change to clay at 77')	No change.
PL-23	---	New point to target floodplain area downgradient of leading edge of plume.	No change.
PL-24 (New-6)		New point to target plume discharge zone at leading edge of plume and define the northern edge of clay that rises sharply in the area.	Not located due to lack of access.
PL-25 (New-1)	---	New point to target area between PW-SP-14 and PW-SP-02/PW-106 cluster where 1,4-dioxane was detected during pore water sampling. Location to be field located after results from Transect Lines #2 and #3 are available.	Not located because location to be determined based on a review of the results from Transect Lines #2 and #3 (when available).
PL-26 (New-2)	---	New point to target other side of stream downstream of VOC/1,4-dioxane cluster encountered during pore water sampling. Utility clearances needed for off Base. Accessibility may be an issue since located in floodplain.	Not located due to lack of access.
PL-27 (New-5)	---	New point to target other side of stream. Utility clearances needed for off Base. Accessibility may be an issue since located in floodplain.	Not located due to lack of access.

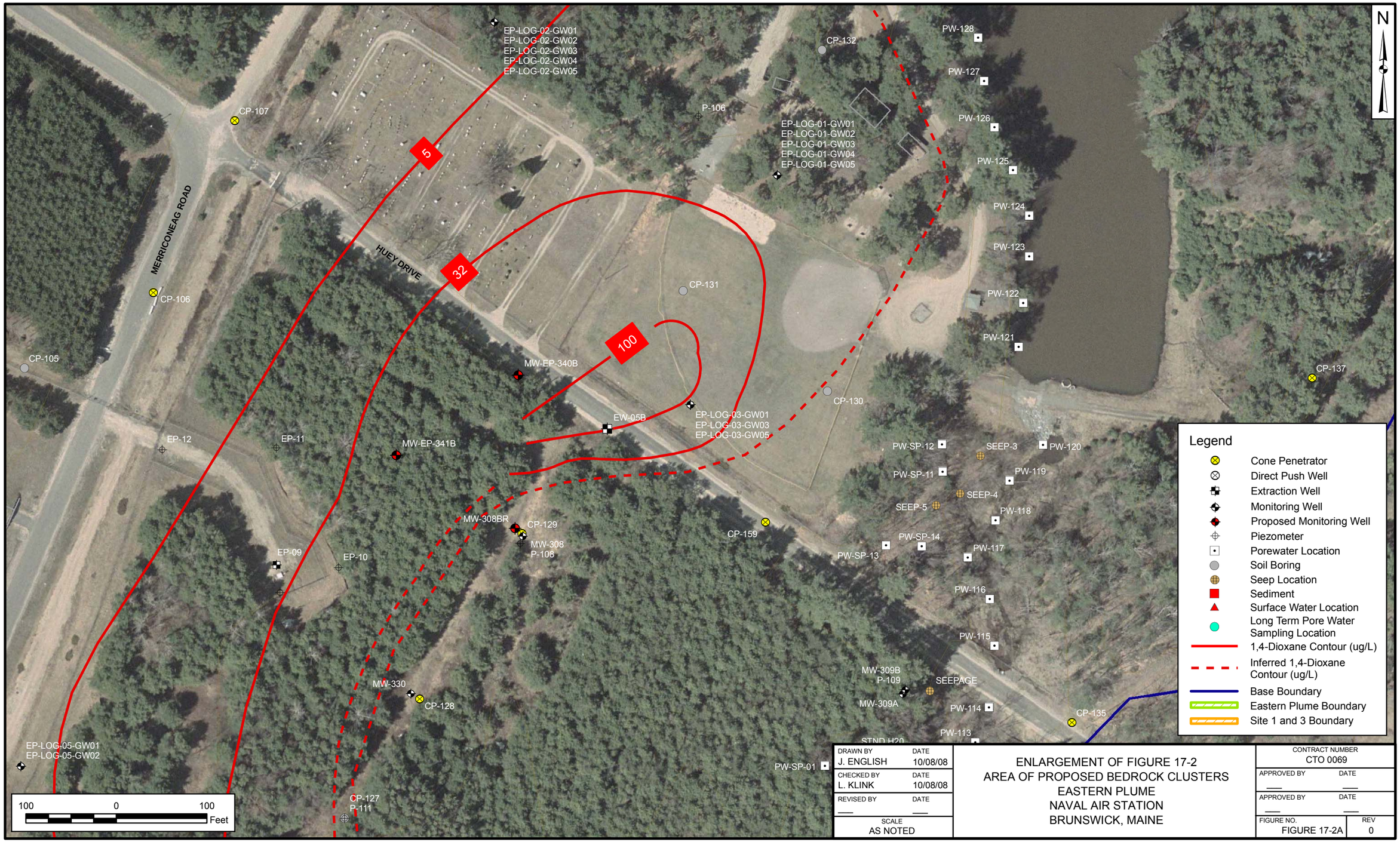
Notes:
Figure 17-1 Revision 0 is in the Draft SAP for the 1,4-Dioxane Investigation.
Figure 17-1 Revision 1 reflects changes made to Figure 17-1, Revision 0 as a result of a conference call with the Regulators.
Figure 17-1 Revision 2 reflects changes made to Figure 17-1, Revision 1, as a result of ground truthing.

Appendix A – Attachment 2

Enlargement of Figure 17-2 Area of Proposed Bedrock Clusters

- Figure 17-2A, Revision 0

P:\GIS\BRUNSWICK_NAS\MAPDOCS\MXD\BEDROCK_WELL_CLUSTERS.MXD 10/08/08 JEE



DRAWN BY J. ENGLISH	DATE 10/08/08	ENLARGEMENT OF FIGURE 17-2 AREA OF PROPOSED BEDROCK CLUSTERS EASTERN PLUME NAVAL AIR STATION BRUNSWICK, MAINE	CONTRACT NUMBER CTO 0069
CHECKED BY L. KLINK	DATE 10/08/08		APPROVED BY ___ DATE ___
REVISED BY ___	DATE ___		APPROVED BY ___ DATE ___
SCALE AS NOTED			FIGURE NO. FIGURE 17-2A / REV 0

APPENDIX B

CONCEPTUAL SITE MODEL

Final
Summary Report
for the Mere Brook and Merriconeag Stream Floodplain and Upland Area Investigation, Eastern Plume

Naval Air Station Brunswick, Maine

Prepared for

Department of the Navy
Naval Facilities Engineering Command
BRAC Program Management Office – Northeast
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APPENDIX F

REVISIONS TO THE CONCEPTUAL SITE MODEL FOR THE EASTERN PLUME

CONCEPTUAL MODEL OF THE EASTERN PLUME, NAVAL AIR STATION, BRUNSWICK, MAINE
Revision 4
July 2008

This Conceptual Model was generated with input from the U.S. Navy, site regulators including the U.S. Environmental Protection Agency (EPA), Maine Department of Environmental Protection (MEDEP), and the Brunswick Area Citizens for a Safe Environment (BACSE). The Conceptual Site Model of the Eastern Plume was originally developed in 2000 and it updated typically during the December Technical Meetings in conjunction with the project stakeholders. The goal of the Conceptual Model is to provide a brief summary overview of the current understanding of the geology and hydrogeology of the Eastern Plume so areas of consensus are clearly defined. The Conceptual Model is updated the when new information becomes available on the Eastern Plume. This Document is revised in corroboration and consensus with the Project Stakeholder and meant to be used as a tool by site decision-makers. This Conceptual Model will be updated in the future as new data are collected and agreed on by the Project Stakeholders. This is the fourth revision of the Conceptual Model for the Eastern Plume. This Model was last updated during the December 2006 Technical Meeting and incorporates comments provided by the Project Stakeholders in November 2006 (MEDEP). Since that time, new information has become available and is briefly summarized in this document. This document is considered to be a "work-in-progress" for use by the NAS Brunswick Project Team.

INTRODUCTION

This brief description of the Conceptual Model of the Eastern Plume provides a generalized overview of the geology and contaminant distribution as it is currently understood by EPA, MEDEP, and the Navy. This Conceptual Model is not intended to be an exhaustive summary of site geology, but will provide an overall framework within which current and future site data can be interpreted. The foundation of this Conceptual Model is based on the data collected and summarized in the Remedial Investigation (E.C. Jordan 1990, 1991). Much subsurface data has been collected by direct-push soil and groundwater sampling in specific areas during the past 5 years, and has been integrated into this model. Data collected during the Long-Term Monitoring Program have also been used to develop this understanding of site conditions, and to refine the Conceptual Model. The site monitoring data at the Eastern Plume were collected during Monitoring Events (ECC 2007a,b,c,d,e and 2008b). Currently, the Navy is in the process of issuing Monitoring Event 31 (September 2007) for the Eastern Plume. Beginning with the April 2008 sampling round, the monitoring event reports for the Eastern Plume will be issued as one separate monitoring event report and not combined with Sites 1 and 3 Landfill.

Future site data will be incorporated into this Conceptual Model, considered as a work in progress, and will berevised as needed. This revision is based on the site understanding as of February 2007, and includes the general findings from various recent supplemental investigations that were conducted at the Eastern Plume by the EPA, MEDEP or the Navy. Additional data collected at the Eastern Plume which have been used to update this Conceptual Model include the following:

Site 11 Bedrock Investigations (2003-2005)

- A geophysical investigation that was completed by EPA for Site 11 and areas downgradient (Hager GeoScience, Inc. 2004). In September 2003, the EPA's consultant contracted with Hager

GeoScience to conduct a geophysical investigation at NAS Brunswick. The objectives of this investigation were to map the bedrock surface, ground-truth the photo-lineaments previously identified throughout the site by the EPA's consultant and identify other possible fracture zones. A secondary objective included mapping key stratigraphy horizons in the study area. The purpose of this study was to provide further understanding of the possible migration pathways in bedrock fractures and deeper stratigraphic zones which may be controlled by bedrock structure.

- In 2004 a bedrock well (MW-NASB-11BR) was installed at Site 11 based on fracture trace analysis and geophysical investigations (ECC 2005).

Mere Brook and Merriconeag Stream Investigations (2005-2007)

- The MEDEP, EPA and Navy conducted a pore water investigation in the area of MW-313 of Eastern Plume. This investigation was conducted to further assess the notion that the Eastern Plume contaminants within the lower sand were discharging (upwelling) into Mere Brook and its flood plain in the vicinity of monitoring well MW-313. Two sampling events were included in this investigation: August 2005 and September 2005. The later of which focused on the areas of the confluence for Mere Brook and Merriconeag Stream and their floodplain areas. The EPA provided a mobile lab to field screen the samples and a selected portion of the samples were analyzed offsite for VOCs and for 1,4 –dioxane. The MEDEP issued a report entitled Pore water Sampling Data From Mere Brook and Merriconeag Stream Area (MEDEP 2005). This investigation concluded that VOCs and 1,4-dioxane are migrating into Mere Brook, Merriconeag Stream and the associated floodplain areas at concentrations in exceedance of MEDEP Maximum Exposure Guidelines (MEGs) and/or Federal Maximum Contaminant Levels (MCLs).

- Between February 2007 and September 2007, the Navy conducted a groundwater investigation in the floodplain and upland areas of Mere Brook and Merriconeag Stream in the vicinity of the confluence in order to address data gaps associated with previous investigative actions conducted in Mere Brook, Merriconeag Stream, and the associated floodplain areas. Specific objectives of the 2007 investigation included the following: (1) determine the extent of Eastern Plume migration to surface water within the vicinity of the Mere Brook, Merriconeag Stream confluence and associated floodplains, and (2) refine understanding of localized subsurface geology and flow regime within the vicinity of Mere Brook and Merriconeag Stream confluence and associated floodplains (ECC 2008a). The findings of the investigation also indicated that VOCs and 1,4-dioxane from the Eastern Plume are discharging into Mere Brook and Merriconeag Stream and the associated flood plain areas in the vicinity of the confluence (ECC 2008a).

New Extraction Well Siting and Installation (2005-to present)

- Rotosonic and Direct-Push Groundwater Sampling of Five Borings at the Eastern Plume (EA 2005). In June 2005, a total of five rotosonic soil borings (EP-LOG-01, EP-LOG-02, EP-LOG-02, EP-LOG-03, EP-LOG-04, and EP-LOG-05) were installed and direct-push groundwater samples were collected to provide data to support the placement of additional extraction wells at the Eastern Plume. The borings were advance from the ground surface into the Presumpscot Clay formation. The boring locations were located in conjunction with Project Stakeholders.

- Extraction Well Installation EW-5B was installed by Navy to reduce concentrations of contaminants in the portions of the Eastern Plume with the highest levels and to limit migration of the Eastern Plume towards surface water (Final Extraction Well Installation Work Plan; ECC 2007f). This well is the first of two additional extraction wells identified as necessary for the site

remedy of the Eastern Plume during the second Five-Year Review (EA/ECC 2005). The rationale for the well placement was that between 1999 and 2003, monitoring point P-106 historically noted the highest concentrations of total VOCs (over 6,000 ug/L) in the Eastern Plume. In October 2003, total VOC concentrations noted at P-106 were 1,268 ug/L. Groundwater data collected during the Spring 2006 monitoring event from P-106 identified total VOCs at a concentration of 850 ug/L. Groundwater samples collected in June 2005 from boring EP-LOG-03 just above the clay surface identified a total VOC concentration of 4,405 ug/L, consisting of 1,1,1-TCA (2,810 ug/L), 1,1-DCE (515 ug/L) and TCE (1,080 ug/L) indicating that the plume is migrating southward and that this area of the Eastern Plume would benefit from a focused mass removal effort by installing an additional extraction well at the location of boring EP-LOG-03.

Therefore the extraction well was located to reduce the high concentrations of VOCs in the vicinity of P-106 and to limit the migration of the Eastern Plume towards surface water. The new extraction well EW-5B was installed in July 2007. The piping and vaulting for this well is planned to be completed in Spring 2008.

Southern Boundary of Eastern Plume Investigations (2000-2003)

- Summary Report of the Direct-Push Investigation of the Southern Boundary of the Eastern Plume and Site 11 (EA 2001). This investigation was conducted to address data gaps identified during the Technical Meetings held on 13 and 14 December 2000 which specifically focused on the Southern Boundary of the Eastern Plume and the bedrock knob area in the vicinity of bedrock monitoring well MW-323 at Site 11. The data gaps identified during the December 2000 Technical Meeting are as follows: (1) potential bedrock exposure at monitoring well MW-323 and the potential presence of dense non-aqueous phase liquid (DNAPL) at Site 11; (2) the terminus of the lower sand in the southern boundary of the Eastern Plume; (3) groundwater flow determination for the southern boundary area; and (4) confirm cone penetrometers point CP-118 in the southern boundary area. The Summary Report (EA 2001) included a proposed updated version of the Conceptual Model as Appendix H.

- Summary Report for the Direct –Push Investigation at the Southern Boundary of the Eastern Plume (EA 2004). This investigation was conducted as a follow-up to the initial investigation activities in the Southern Boundary of the Eastern Plume which began in 2001 (EA 2002). The summary report provides the findings of the investigation activities between 2001 and 2003 focused on the southern boundary of the Eastern Plume. The objectives of this investigation were to obtain additional subsurface geological data to augment the existing data on the southern boundary of the Plume and to assess whether preferential migration pathways (i.e., sand layers within the transition unit) are located along the Southern Boundary of the Eastern Plume. The third objective of the investigation was to determine the lateral and vertical distribution of VOC impacts in the study area. The Summary Reported (EA 2004) included an updated version of the Conceptual Model (dated December 2004) as Appendix H.

Monitored Natural Attenuation within the Eastern Plume (2003- 2005)

- The Navy conducted a monitored natural attenuation assessment on selected groundwater sampling locations over four monitoring events between October 2003 and April 2005 in order to determine any evidence of CVOC degradation by reductive dechlorination in the Eastern Plume (EA/ECC 2006). The assessment indicated inadequate evidence for biodegradation of chlorinated

hydrocarbons and concluded that the Eastern Plume lacks the benefit of strongly reducing conditions resulting in a limited extent of reductive dechlorination activity.

Residential Well Sampling (1990 -2007)

- Following the Supplemental Remedial Investigation (E.C. Jordan 1991), off-site private wells have been sampled periodically by either the Navy or MEDEP. In October 1990, the Navy sampled 23 off-site private wells for VOCs. The samples were analyzed via methods applicable to drinking water standards. Appendix L of the 1991 Draft Final Supplemental RI Report provides the list of the 23 homeowners and privately owned wells that were sampled in this initial effort. The 1990 residential sampling as part of the Supplement RI concluded that one VOC (TCE) was reported in one private well at 0.5 ug/L; however, it was concluded not to be associated with the Eastern Plume and may be a false positive. The well was re-sampled in February 1991 and no VOCs were detected during the second round of sampling. It was concluded (E.C. Jordan 1991) that the eastern boundary of the Plume occurs approximately 1, 500 feet east of the private wells and is restricted to the overburden soils (E.C. Jordan 1991).

- Due to elevated concentrations of 1,1-DCE reported at bedrock well MW-308 (7.5 ug/L to 29 ug/L) which exceeded both the MCL and MEG, and TCE (13.7 ug/L and 49 ug/L, which is over the MCL) over 3 sampling events (Spring 2006, Spring 2007 and Fall 2007) of , additional groundwater samples were collected for VOCs from the Smith Residential Well located 38 Purington Road, which is locted approximately 675 feet from bedrock well MW-308. The October 2007 results indicated that VOCs (including 1,4-dioxane) were not present in the Smith Residential Well. Another bedrock well, MW-309B, is located approximately 300 ft east of MW-308 and is sampled annually in the Long-Term Monitoring Program for the Eastern Plume. VOCs have not been detected in this well. The occurrence of reported VOCs in this one bedrock well (MW-308)location is currently being evaluated by the Project Team Technical Evaluation Group.

- A list of the residential wells sampled during the 1990 Remedial Investigation is provided n Appendix L of the Draft Final Remedial Investigation Report (E.C. Jordan 1991).

  Since that time, the following residential wells have been sampled by either the MEDEP or Navy.

  - Smith Well – 38 Purinton Road – Sampled 11 November 2004, 26 May 2005 and 27 November October 2007. No VOCs reported.

  - Berg Well – 246 Coombs Road – 9 January 2006. No VOCs reported.

  - Ferguson Well – 282 Coombs Road – 9 and 17 January 2006. TCE was reported at very low levels from 0.4 to 0.74 ppb, below the MEG and MCL. No other VOC were detected.

  Additionally, the MEDEP provided a map of property locations and a list of available well information on 20 nearby residential wells located on Purinton and Coombs Road (which was compiled in 2006 by the MEDEP) to Project Stakeholders in February 2008.

1,4-Dioxane Impacts Associated with the Eastern Plume in Groundwater and Surface Water (2004-to present)

- Results of the groundwater investigations Mere Brook and Merriconeag Stream, indicate 1-4 dioxane and Eastern Plume contaminants are discharging to surface water near the confluence of these two surface water bodies along the eastern edge of the Eastern Plume in the upland areas and within the flood plains of Mere Brook and Merriconeag Stream. (ECC 2008a).

- 1,4-Dioxane has been reported in groundwater samples collected at selected sampling locations throughout the Eastern Plume. 1,4- Dioxane was not identified as a Contaminant of Concern in the Record of Decision (REF?), but is sampled at select groundwater and pore water locations within the Eastern Plumeand is analyzed separate from the Long-Term Monitoring Plan. a The Navy is planning to conduct a separate remedial investigation on 1,4-dioxane to determine an appropriate remedial actionto address this VOC compound at the Eastern Plume (TtNUS issued Sampling and Analysis Plan for a Supplemental Remedial Investigation of 1,4-Dioxane in the Eastern Plume in October 2007 (TtNUS 2006).

A complete reference list is provided for these and other key reports used for the development of this Conceptual Model for the Eastern Plume.

The objective of defining the Conceptual Model at this time is to summarize and document the commonly agreed upon interpretation of site geology, contaminant transport, and contaminant distribution. The Conceptual Model of the Eastern Plume will continue to be refined based on additional investigations and changes in contaminant concentrations over time and noted during the Long-Term Monitoring Program. It is anticipated that this Conceptual Model will serve as the framework to be used to interpret future data collected as part of the Long-Term Monitoring Program, and to enhance optimization of remedial operations.

OVERBURDEN GEOLOGY

The stratigraphy of the portion of Naval Air Station Brunswick that includes the Eastern Plume is comprised of interbedded sand, silt, and clay units that overlie the undulating bedrock surface and some occasional discontinuous till. The majority of the overburden units at the Eastern Plume, with the exception of the upper sand and till, are interpreted to be part of the Presumpscot Formation, which are comprised of water-laid clay, silt, and sand with some minor gravel units (E.C. Jordan 1990). The formation exhibits a general coarsening upwards sequence (E.C. Jordan 1990). Three major overburden layers are present: upper sand, transition which contains a sandy interval often referred to as the lower sand, and clay.

The Presumpscot Formation was deposited on the ocean floor during a submergence of the area during the last period of glaciation. Sediments were deposited over a till or directly over bedrock (E.C. Jordan 1990). The Presumpscot clay and transition are comprised of marine deposits (i.e., deposited under low energy conditions) which resulted in the generally fine-grained nature of the overburden material. Clay thickness is variable, and the thickness of the clay unit may have a relationship to the bedrock surface topography upon which it was deposited (E.C. Jordan 1990).

The upper sand unit has a non-marine origin, and was deposited by the Androscoggin River as it shifted laterally in a post-glacial course following a drop in sea level. In some areas, the upper sand was

reworked by wind action. Following deposition of these units, streams have carved steep sided gullies into these units, particularly the upper sand (E.C. Jordan 1990).

- The upper sand unit consists of fine sands that readily transmit groundwater. The unit is approximately 10- to 20-ft thick across the Eastern Plume area. In the vicinity of Sites 1 and 3, this upper sand unit thickens to greater than 40 ft (EA 2000). The upper layers of fine sand are mostly silt-free and have the highest conductivity of the overburden units (E.C. Jordan 1990).

- The transition unit lies between the upper sand and the clay, and is composed of interbedded sands, silts, and clays (E.C. Jordan 1990). The transition unit ranges in thickness from 0 to 80 ft (EA 2000). The transition forms a wedge that increases in thickness from west to east across the Eastern Plume, and pinches out just east of the Eastern Plume. The upper portions of the transition unit exhibit unconfined groundwater conditions, while deeper intervals in the transition exhibit confined conditions due to the presence of low permeability zones within this unit. The majority of the transition unit is silt and clay, although sand layers are present with thickness ranging from inches to several feet. The transition unit contains relatively thin and possibly continuous sand layers that may act as preferential pathways for groundwater movement within this unit. A mappable sandy interval has been identified near the base of the transition unit in some portions of the Eastern Plume. This sandy interval is referred to as the lower sand. Both the upper and lower sands have been targeted by remedial extraction at the Eastern Plume since remedial operations (i.e., groundwater pump and treat) were started in 1995. Approximately 499 kg of contaminant mass has been removed by the extraction system and treated by the treatment plant as of April 2005 the bulk of the mass resided in the deep sand unit.

- Vertical movement of groundwater is limited by interbedded sands and silts of the transition, and vertical hydraulic conductivity is at least one order of magnitude lower than horizontal hydraulic conductivity (E.C. Jordan 1990). The transition unit is acting as an aquitard between the upper sand and lower sand. The lateral continuity of transition zone sand layers appears highly variable. That is, some sand layers may provide conduits to groundwater flow, although others may be isolated and, therefore, may not conduct significant amounts of groundwater very far (E.C. Jordan 1990).

- Sand bodies are present in the lower transition unit south of New Gurnett Road, and these units extend northward to interfinger with the mappable lower sand unit present in the vicinity of Mere Brook. Boring logs and analytical data from the Southern boundary area suggest that preferential pathways to the south of New Gurnett Road are present, but less extensive than to the north.

- The transition unit has a horizontal hydraulic conductivity 1-2 orders of magnitude below that of the upper sand (E.C. Jordan 1990). Hydraulic conductivity for the transition units range from 10^{-4} to 10^{-5} cm/sec (from 0.3 to 0.03 ft/day) for the fine grained interbeds, and can range up to 10^{-3} cm/sec (3 ft/day) for sandy intervals (E.C. Jordan 1990). Although not tested, the vertical hydraulic conductivity is believed to be one order of magnitude lower than the horizontal hydraulic conductivity.

- The Presumpscot Clay underlies the transition unit and is draped over the bedrock or till surface in a nearly continuous layer. The clay has low permeability which ranges from 10^{-6} to 10^{-8} cm/sec (from 3×10^{-3} ft/day to 3×10^{-5} ft/day) (E.C. Jordan 1990). The clay thickness has been measured up to 80 ft, although the thickness of the clay may be greater in some areas (EA 2000). In one area, near MW-323 and MW-NASB-11-BR, the clay appears to be very thin or may not be present. The remaining portions of the Eastern Plume appear to be underlain by several feet or

tens of feet of clay. This clay forms the base of the overburden flow system and prevents significant groundwater flow between the overburden and bedrock or till.

- A geophysical investigation was completed by EPA in late 2003 for Site 11 and areas to the east (Hager GeoScience 2004). The results of the geophysical investigation focused on identifying unit thickness and the top elevations of the transition, clay unit, and bedrock. The topography of the top of the clay unit is similar to the top of bedrock surface, exhibiting an easterly slope from high elevations near Site 11, decreasing eastward. However, the thickness of the clay also shows considerable variation over the entire plume. Minimal clay thickness was interpreted east of Site 11 (0 to 10-ft thick) with much greater thickness (up to 110+ ft) reported further east in the vicinity of a bedrock depression parallel with the Weapons Area Road. The clay thins further east toward Picnic Pond with a thickness varying from near zero to 25 ft. The top elevation and thickness of the transition unit generally mimics the clay, showing an eastward slope with greatest thickness noted where bedrock depressions are present.

BEDROCK GEOLOGY

The bedrock underlying the site has been mapped as the Cape Elizabeth Formation, comprised of a micaceous schist, with pegmatite veins up to several feet thick. Regional bedrock features have been identified which trend to the north-northeast. Existing bedrock data for the site are based on seven boring locations. Based on the available core logs, the rock shows decreasing rock fracturing with depth as measured by rock quality designation (RQD). Major fractures were not typically encountered within drilled depths, however, bedrock test wells do yield water. Very minor effects due to weathering were noted (E.C. Jordan 1991) in the locations where bedrock was drilled during initial characterization of the site. Based on six slug tests, the bedrock has permeability which ranges from 10^{-4} to 10^{-5} cm/sec (from 0.3 ft/day to 0.03 ft/day) (E.C. Jordan 1991).

The bedrock well (MW-NASB-11-BR) drilled in 2004 based on the geophysical investigation targeted a bedrock knob identified near Site11. The upper 30 feet of bedrock at that location was extensively fractured, and RQDs ranged from 21 to 35%, with iron staining common on fracture faces. Overall bedrock is overlain by relatively impermeable Presumpscot clay, creating confined conditions (E.C. Jordan 1991). However, boring log information and geophysical data indicate that the clay material is thin or not present in the vicinity of bedrock monitoring well MW-323 and MW-308.

As a result of data gaps identified during Technical Meetings held in 13 and 14 December 2000 which specifically focused on the Southern Boundary of the Eastern Plume and the bedrock knob area in the vicinity of monitoring well MW-323 at Site 11 a direct-push investigation was conducted in the southern boundary of the Eastern Plume and at Site 11. This investigation included the use of *in-situ* downhole logging techniques, such as electrical conductivity and a membrane interface probe, groundwater sampling for VOC analysis and the installation of four piezometers in the southern boundary of the Eastern Plume (for details refer to EA 2001).

As a follow-up on the direct-push investigations at Site 11, in September 2003 Hager GeoScience, Inc. was contracted by the EPA to perform a geophysical investigation at the NAS Brunswick. The primary objectives of the investigation were to map the bedrock surface, ground-truth photo-lineaments which had been identified throughout the Base area, and to identify other possible fracture zones. The secondary objectives were to map the key stratigraphic zones beneath the site (refer to Hager GeoScience, Inc. 2004 for details).

In June 2004, the Navy installed one bedrock well at Site 11 (MW-NASB-11BR) on the basis of the results of the geophysical study and fracture-lineament trace analysis conducted by EPA (Hager GeoScience, Inc. 2004). At the Site 11 bedrock well location, the bedrock is overlain by approximately 30 ft of overburden. The bedrock was cored at this location and was recorded as grey micaeous schist with recorded fracture and foliation zones. The bedrock was cored to 94.3 ft below the ground surface. The overburden was cased off and cement grouted in place in order to prevent the potential migration of contaminants into the bedrock during the drilling activities. This bedrock well was sampled for VOCs using passive diffusion bag sampling. One VOC compound 1,1-DCA was reported at concentrations well below the specified MEG concentration. (refer to ECC 2005 for details on the Site 11 bedrock well installation). Generally, groundwater flow is through fractures and joints in the bedrock, and the horizontal and vertical distribution of groundwater in these water-bearing zones is believed to be highly variable beneath the site. Upper portions of the bedrock have higher permeability than deeper zones (E.C. Jordan 1990).

Pressurized packer tests were completed on bedrock wells at numerous intervals, which noted a similar range of hydraulic conductivity (E.C. Jordan 1991). Hydraulic conductivity values recorded at the two bedrock wells within the Eastern Plume (MW-308 and MW-309A) were in the 10^{-5}-10^{-4} cm/sec range (0.03 ft/day – 0.3 ft/day) (E.C. Jordan 1991). The inferred fractured intervals identified with packer tests generally noted a hydraulic conductivity of approximately 10^{-4} cm/sec (0.3 ft/day) at 40 psi. No hydraulic conductivity testing or borehole geophysics were conducted at MW-NASB-11-BR due to instability of the borehole walls and fractured bedrock character at this location.

The results of the geophysical investigation for Site 11 and areas to the east show the top of bedrock surface with significant topographic relief and sloping strongly to the east. Bedrock elevations at Site 11 range from 0 to 40 ft mean sea level with the top of the bedrock surface dropping to approximately -50 ft mean sea level near Picnic Pond. The top-of-bedrock contours show depressions with a low of approximately -110 ft mean sea level. The bedrock surface is irregular throughout the northern Merriconeag Stream valley, but indicates a general valley beneath the Eastern Plume, with a north-northeast/south-southwest-oriented axis. Areas with low seismic velocity were noted during the geophysical investigation which suggest bedrock fractures or faults may be present. Based on observations of bedrock along the west face of Buttermilk Mountain Ridge, southwest-northeast fractures may be expected, which is consistent with known regional fault trend directions. These outcrops and the distribution of low seismic velocity intervals also suggest a second set of bedrock fractures may be present trending roughly east-west.

HYDROGEOLOGY

The groundwater flow system in the upper sand unit is unconfined. Groundwater flows laterally through the upper sand, while downward groundwater flow is much slower due to the presence of the underlying transition unit. Groundwater migrates in the upper sand toward surface waterbodies (Mere Brook and Merriconeag Stream) and some portion discharges to these streams and associated wetlands.

The majority of groundwater flow in the transition unit occurs within the lower sand interval. Throughout much of the Eastern Plume, sandy intervals within the transition are confined by the overlying fine-grained clay and silt within the transition unit. The substantial potentiometric differences between the upper sand unit and lower sand intervals in the transition suggest that groundwater movement from above the transition unit to underlying units is moderately restricted. The sand intervals within the transition unit thin and become finer-grained to the south, and this change is believed to further increase confinement of groundwater within the deeper transition unit. Thinning and less conductive sand intervals near the present terminus of the plume are thought to slow or restrict to some degree

groundwater movement in these layers. The lower sand becomes thin to the east and pinches-out east of MW-311. East of Merriconeag Stream and Mere Brook, thin overburden that is insufficient to transmit significant quantities of groundwater cover bedrock on the west-facing ridge flank.

Groundwater discharge from the confined lower sand may occur largely by upward diffusive flow through fine-grained units of the overlying transition unit, and through several small springs and seeps observed near MW-207A/B (E.C. Jordan 1990) and to the north near Picnic Pond (U. S. EPA, March 2003). Relatively high potentiometric head and artesian conditions in the lower sand have been noted, reflecting groundwater confinement within the lower sand caused by: (a) underlying and overlying clay and silt, (b) reductions in transmissivity of the overall transition unit south of the east-west reach of Mere Brook, and (c) a pronounced shallowing of the Presumpscot Clay along the west side of the north-south reach of Mere Brook. Groundwater potentiometric surface maps indicate groundwater flow potential in the transition unit is to the south-southeast.

In the Southern Boundary area of the Eastern Plume (south of Mere Brook and extending south of New Gurnett Road), shallow groundwater flow has easterly and northeasterly components. Deep groundwater flow in this area is predominantly to the southeast and east, toward Mere Brook.

Groundwater extraction has been occurring since 1995 to reduce volatile organic compound (VOC) concentrations and maintain hydraulic control of the Eastern Plume. Deep overburden groundwater flow patterns have been locally altered by pumping at extraction wells, but only small changes in groundwater flow patterns have been noted away from the immediate vicinity of most extraction wells. The largest changes in lower groundwater flow patterns are seen at EW-2A, which was installed to intersect only the lower sand interval. Potentiometric head at this location has been approximately 12 ft lower than noted in the shallow flow system (EA 2000). A reversal of shallow and deep heads near the confluence of Mere Brook and Merriconeag Stream has persisted since 1998. Extraction well EW-5 has been replaced by EW-5A, which is screened only across the deep sand interval. Other operating extraction wells (EW-1 and EW-4) are screened across the upper sand and lower transition intervals. Effects on the deep flow system due to extraction well pumping at these two wells have been limited by shallow groundwater preferentially moving into extraction wells. Overall, the groundwater extraction system has not established hydraulic control of the Eastern Plume, although natural geologic conditions appear to have helped contain groundwater contamination.

Groundwater flow potential in bedrock beneath the Eastern Plume appears to be toward the southeast (E.C. Jordan 1991). In places, bedrock groundwater is confined by the overlying unconsolidated units.

Hydrogeological data obtained during the 2007 Mere Brook and Merriconeag Stream Investigation, which was focused on the flood plain and upland areas near the confluence of Mere Brook and Merriconeag Stream, suggest the following groundwater flow conditions. These findings are summarized in the Draft Summary Report for the Mere Brook and Merriconeag Stream Flood Plain and Upland Area Investigation, Eastern Plume, Naval Air Station Brunswick," Maine (ECC 2008a).

- Shallow zone groundwater flow within both the Mere Brook flood plain and upland areas in the upper sand unit, and intermediate groundwater within the flood plain areas in the transition zone, is predominately north to south in the northern portion of the site (above the Mere Brook and Merriconeag Stream confluence), and west to east in the southern portion of the site (at and below the southern portion of the confluence). Shallow groundwater flow is generally horizontal and unconfined within the upper sand unit. Intermediate zone groundwater flow within the upland area in transition zone is transmitted primarily through intermittent silt and sand layers which are confined by layers of overlying and underlying clay and silt. The gauging data for the monitoring wells located within the

intermediate transition zone in the upland areas indicate that groundwater flow in generally north to south and easterly flowing to Mere Brook,. The intermediate groundwater in the transition zone is discharging to Mere Brook.

- Deep groundwater flow within the lower sand unit and the transition zone/top of clay in the upland areas is found to be confined or semi-confined at some locations. The groundwater gauging data indicate artesian well conditions are present in the lower sand unit in monitoring wells near the Mere Brook and Merricongeag confluence confirming the confined/semi-confined conditions.

CONTAMINANT TRANSPORT AND DISTRIBUTION

Based on data collected during the Remedial Investigation and Long-Term Monitoring Program, the following interpretations are made regarding contaminant transport and distribution within the Eastern Plume:

- Sites 4, 11, and 13 have been identified as source areas of the Eastern Plume, and are located to the north-northwest of the current plume. These sites have been investigated, and source removal occurred in the early 90s, and subsequently, natural attenuation has occurred. These events appear to have effectively depleted nearly all the residual fuel and solvents in the source areas. Chlorinated solvents released at these sites entered the upper sand, migrated around or through upper transition unit, and eventually reached the lower sand deep within the transition unit.

- Within the Eastern Plume historic footprint, the upper sand unit is nearly remediated of contaminants. Some limited impacts have been observed in the shallow interval, near monitoring wells MW-1104, MW-209, MW-224 and MW-332. Near the source area at Site 11 (MW-1104), residual concentrations of VOCs have declined below State MEGs or Federal MCLs (ECC 2005) . South of Sites 4,11 and 13, groundwater at MW-209 had been non-detect for VOCs in 1998, however a deep interval diffusion sample in Fall 2002 detected VOCs in excess of 130 ug/L, with TCE over 40 ug/L. Low detections of VOCs at MW-224 have been below the MEGs and MCLs. Shallow groundwater contamination near MW-332 is believed to have resulted from unrestricted artesian flow of contaminated groundwater from MW-311 from 1990 to 1995.

- The majority of the groundwater plume is located within the lower sand near the base of the transition unit. Based on existing data, the Eastern Plume extends south to the New Gurnett Road and east to the confluence of Mere Brook and Merriconeag Stream as reported as part of the MEDEP pore water sampling effort in 2005 (MEDEP 2005). Data from April 2005 indicate the leading edge of the plume extends east to MW-313, and south to MW-337. The clay unit underlying the transition forms a clay trough whose eastern slope rises toward the southeast and western slope rises toward the southwest where the lower sand unit pinches out. The constriction of the lower sand south of New Gurnett Road, deep groundwater flow to the east, and the presence of this clay unit along the southern plume boundary influence migration of the Eastern Plume toward Mere Brook.

- No permeable pathways for contaminants have been identified along the southwestern boundary of the base where overburden consists of low-permeability units such as silt and clay. These units do not conduct significant amounts of groundwater. Along the southern base boundary, overburden has been measured to be approximately 50-ft thick.

- The seven monitoring wells located in bedrock have not shown concentrations of VOCs above State MEGs or Federal MCLs. Several detections of 1 μg/L were recorded in the 1990s, and

MW-323 has recent detections of 1,1 dichloroethane (1,1 DCA) ranging from 1 to 3 ug/L, well below the State MEG. A single round of diffusion sampling was completed at MW-NASB-11-BR in 2005, 1,1 DCA was detected at multiple depths at 1 to 2 ug/L. Low flow groundwater samples collected from MW-308 in October 2007 reported concentrations of 1,4-dioxane (9.1 µg/L), TCE (26.7 µg/L), 1,1 DCA (6.8 µg/L), and 1,1 DCE (12.1 µg/L). With the exception of 1,1 DCE (detected at 29 µg/L), these concentrations are lower than those detected in 2006 (TCE at 49 µg/L and 1,1 DCA at 15 µg/L. 1,4-Dioxane was not sampled for in 2006). Passive Diffusion Bag (PDB) samples collected from MW-308 in April 2008 reported significantly lower concentrations of VOCs. 1,1 DCE and TCE were detected at concentrations of 0.44 µg/L and 0.84 µg/L, respectively. Low flow sample results for 1,4-dioxane was reported as non-detect. The Navy is planning a bedrock groundwater investigation which will provide additional data with regards to the geologic, hydrogeologic, and groundwater contaminant properties of the bedrock within the Eastern Plume.

- The rotosonic borings completed in 2005 targeted clay "bowls" identified in the northern portion of the Eastern Plume by the 2003 geophysics investigation. Data collected indicate that the depressions in the clay topography may contain residual VOCs at concentrations exceeding those in most monitoring wells within the plume. One of these locations will be the focus of a new extraction well installation planned for 2007 (EA 2005).

- Shallow diffusion samplers placed in Mere Brook and associated wetland showed concentrations of Eastern Plume VOCs in upwelling groundwater north of New Gurnett Road, but not south of this road. Upwelling of the Eastern Plume at low concentrations has also been confirmed in Merriconeag Stream just north of Picnic Pond and in Picnic Pond (Gannett Fleming, 2003).

- Data from 2003 to 2005 at MW-313 and MW-333 indicate Eastern Plume migration is continuing toward this area. A detection of TCE at SW-12 in 2003 (ME22), as well as a few detections at SW-09 and SW-13 since 2000 support the concept of plume discharge to the surface water floodplains.

- Pore water sampling conducted in 2005 along Mere Brook's eastern bank from the vicinity of MW-313 north to the confluence with Merriconeag Stream (triggered in part by the surface water detections) indicate the plume is upwelling into the Mere Brook floodplain, with highest measured concentrations currently found near the confluence. Further investigation planned for 2007 will refine the understanding of the plume migration in this area.

- 1,4-Dioxane investigations are still on-going and the Navy is preparing a Remedial Investigation Work Plan to investigate the extent of the existing groundwater impacts associated with the Eastern Plume.

- Residential sampling efforts by MEDEP and Navy are on-going. To date, the residential sampling data has indicated that VOCs associated with the Eastern Plume have not been detected in the residential wells with exception of (1) low concentrations of carbon disulfide (1 ug/L) reported in the November 2004 sample collected from the Smith well, which is not expected to be associated with the Eastern Plume, and (2) the low concentrations of TCE (0.4 and 0.74 ug/L) detected in the Ferguson well in two samples collected in January 2006 by the MEDEP. It was recommended by the MEDEP that the well be re-sampled in February 2006.

In summary, the Eastern Plume has been slowly migrating to the south and southeast, with minor discharge to Picnic Pond. Monitoring wells MW-230A, MW-313, and MW-333 have all had sporadic VOC concentrations in excess of the State Maximum Exposure Guideline and/or Federal Maximum Contaminant Level standards since 2003. These detections, in addition to low VOC detections at MW-334, MW-337 and MW-338B, indicate minor migration on base to the south and east of the junction of New Gurnett and Merriconeag Roads.

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APPENDIX C

SUPPORTING INFORMATION

Appendix C-1

Correspondence

DEPARTMENT OF THE NAVY
BASE REALIGNMENT AND CLOSURE
PROGRAM MANAGEMENT OFFICE, NORTHEAST
4911 SOUTH BROAD STREET
PHILADELPHIA, PA 19112-1303

5090
BPMO NE/LM
Ser 07-098
July 27, 2007

Ms. Christine A. P. Williams, RPM
Federal Facilities Superfund Section
US EPA New England Region 1
1 Congress Street, Suite 1100 (HBT)
Boston, MA 02114-2023

Dear Ms Williams:

SUBJECT: 1,4-DIOXANE AT THE NAVAL AIR STATION BRUNSWICK, MAINE

This is in response to your letter dated June 6, 2007 concerning 1,4-dioxane within the Eastern Plume at Naval Air Station Brunswick (NASB), Maine.

Sampling results for 1,4-dioxane conducted by the Navy at monitoring wells within the Eastern Plume since April 2004 have ranged from non-detect (ND) to 180 parts per billion (ppb), with the highest contaminant levels seen in the south and southeastern portions (leading edge) of the plume. In addition, shallow groundwater sampling conducted along Mere Brook and Merriconeag Stream in 2005 indicated upwelling of 1,4-dioxane as well as volatile organic compounds (VOCs), which prompted the Navy to conduct the presently ongoing investigation in these areas.

The April 2007 groundwater monitoring event shows 1,4-dioxane levels ranging from ND to 92.0 ppb, with only 2 monitoring wells (MW-313 and MW-333) exceeding the state maximum exposure guideline (MEG) of 32 ppb (92.0 ppb and 47.9 ppb, respectively). Both of these wells are located within the southeastern portion of the Eastern Plume.

The Navy began collecting monthly influent and effluent sampling data for 1,4-dioxane from the treatment plant in July 2006. Those results show an average influent concentration of 7.1 ppb and an average effluent concentration of 7.2 ppb, with the range of values being 4.5 ppb to 9.9 ppb, and 4.6 ppb to 10.2 ppb, respectively.

The Navy sampled the extraction wells for 1,4-dioxane during the April 2007 monitoring event . Results for those wells were as follows:

EW-1	1.4 ppb
EW-2A	9.7 ppb
EW-4	6.5 ppb
EW-5A	3.8 ppb

Although none of the sampling results from the extraction wells exceeds the state MEGs, the groundwater extraction and treatment system (GWETS), as presently configured, is not suited for remediating 1,4-dioxane in the Eastern Plume .

The interim remedial action that the Navy implemented for the Eastern Plume in 1995 was designed to extract and treat the contaminants of concern (COCs) listed in the 1992 interim ROD. This remedial action became final when the final ROD was issued in 1998. Neither the interim nor final RODs included 1,4-dioxane as a COC; therefore, the extraction wells were not located to remove this contaminant. This is further evidenced by the fact that the original groundwater treatment for the known COCs used ultraviolet oxidation (UVOx) and was subsequently replaced by carbon filtration, documented in the explanation of significant differences (ESD) in 2000. Activating the former UVOx system in the current GWETS at this time would not achieve the treatment goals for the COCs, including 1,4-dioxane, nor for hydraulic containment and recovery as set forth in the ROD. As a result, it is necessary to fully characterize the extent of the contamination via a remedial investigation (RI) before deciding whether a remedial action plan (supported by an FS) or an amendment to the remedial design is appropriate.

The Navy proposes to complete the CERCLA remediation process as follows:

Supplemental Contract Award(s)	14 Sep 07
Draft Site Characterization (RI) Plan	19 Oct 07
Draft Characterization (RI) Report	08 Feb 08
Supplemental RD/RA Contract Award	14 Mar 08
Public Meeting	19 Mar 08
Draft ESD or ROD Amendment	11 Apr 08
Draft Supplemental RD Work Plan	09 May 08
Begin Supplemental Remedial Action	27 Jun 08

The Navy looks forward to discussing the proposed plan and schedule with you and the other NASB stakeholders at the upcoming technical meetings and conference calls. Please contact Lonnie Monaco at (215) 897-4911 or me at (215) 897-4915 with any questions or concerns.

Sincerely,

Dawn C. Kincaid

Dawn C. Kincaid, P.E.
BRAC Environmental Coordinator
By Direction of BRAC PMO

Copy to:
Claudia Sait - MEDEP
Dale Mosher, Lisa Joy - NASB

Carolyn LePage - LePage Environmental
Al Easterday, Gina Calderone, Catherine Guido – ECC
Linda Klink – TtNUS
Bonnie Capito – NAVFAC Atlantic (Admin Record)
Jennifer Wright, Dan Waddill – NAVFAC Atlantic
Lonnie Monaco, Bob Schirmer - MidLANT

DEPARTMENT OF THE NAVY
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4911 SOUTH BROAD STREET
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5090
BPMO NE/DK
Ser 07-091
June 26, 2007

Ms. Christine A. P. Williams, RPM
Federal Facilities Superfund Section
US EPA New England Region I (EPA)
1 Congress Street, Suite 1100 (HBT)
Boston, MA 02114-2023

Dear Ms. Williams:

SUBJECT: 1, 4-DIOXANE AT THE NAVAL AIR STATION BRUNSWICK, MAINE

This is an interim response to your letter on this subject dated June 6, 2007. In that letter you indicated that EPA expected the Navy to provide a schedule within 30 days. However, in order to address this matter appropriately the Navy will need extra time. This will allow us to consider the serious policy implications raised by 1,4 dioxane as well as allow representatives from the EPA, MEDEP, and Navy to discuss this subject at a meeting on July 10, 2007. Accordingly, we anticipate being able to respond more completely by the first week of August and respectfully ask that you agree to the delay.

Thank you for your cooperation in this matter. Please contact Lonnie Monaco at (215) 897-4911 or me at (215) 897-4915 with any questions or concerns.

Sincerely,

Dawn C. Kincaid

Dawn C. Kincaid, P.E
BRAC Environmental Coordinator
By Direction of BRAC PMO

Copy to:
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Dale Mosher -NASB
Carolyn LePage - LePage Environmental
Al Easterday – ECC
Linda Klink – TtNUS
Jennifer Wright, Dan Waddill, Bonnie Capito – NAVFAC Atlantic
Lonnie Monaco, Bob Schirmer – NAVFAC Midlant
Lawrence Lansdale – BRAC PMO HQ

DEPARTMENT OF THE NAVY
BASE REALIGNMENT AND CLOSURE
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4911 SOUTH BROAD STREET
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5090
BPMO NE/DK
Ser 07-071
June 5, 2007

Ms. Christine A. P. Williams, RPM
Federal Facilities Superfund Section
US EPA New England Region 1 (USEPA)
1 Congress Street, Suite 1100 (HBT)
Boston, MA 02114-2023

Dear Ms Williams:

SUBJECT: EASTERN PLUME (OU 5)1, 4-DIOXANE; NAVAL AIR STATION BRUNSWICK, MAINE

This letter is in response to Comments 1 and 2 of your letter of April 10, 2007 and Comments 1 and 3 of your May 4, 2007 letter, all relating to 1, 4-dioxane within the Eastern Plume (OU 5) at Naval Air Station Brunswick (NASB), Maine.

The Navy, with concurrence from the USEPA and Maine Department of Environmental Protection (MEDEP), has been sampling selected monitoring wells within the Eastern Plume for 1, 4-dioxane in order to identify the presence and extent of the contaminant. As of September 2006, seven (7) wells had been sampled for 1, 4-dioxane, some beginning in April 2004. Highest 1, 4-dioxane concentrations have been found in the southeastern portion of the plume. Stakeholders recently agreed to modify to the Eastern Plume monitoring plan to increases the number of wells sampled for 1, 4-dioxane. This will yield data that better defines its presence within the Eastern Plume.

The status of 1, 4-dioxane as an emerging contaminant makes it difficult to select defensible criteria for initiating and completing remedial action. The Navy proposes the following approach to address 1, 4-dioxane within the Eastern Plume.

- Use groundwater monitoring data to develop a site specific risk assessment
- Prepare a report which addresses the levels and locations of 1, 4-dioxane in the Eastern Plume, evaluates and identifies 1, 4-dioxane ARARs, risk assessment findings and recommendations to support discussions on future actions

As you are aware, the original process at the groundwater extraction treatment system (GWETS) used ultraviolet oxidation (UVOx) equipment to treat the Eastern Plume. Subsequent optimization decisions were made to replace the UVOx system with the more effective and efficient granular activated carbon (GAC) system. The UVOx treatment equipment was taken off-line and mothballed. Maintenance, including replacement of bulbs and other system components requiring repair, was performed on the UVOx equipment. The equipment is now operational, but not on line. The Navy would complete an engineering analysis to incorporate the UVOx equipment into the treatment train, if the decision is made to do so.

Treatment decisions are affected by the fact that 1, 4-dioxane is currently recognized as an emerging contaminant (EC) by the Navy, attributed to uncertainties and changes proposed in its toxicological information. For example, the EPA Integrated Risk Information System (IRIS) does not currently provide a Reference Dose for Chronic Oral Exposure (RfD) or a Reference Concentration for Chronic Inhalation Exposure (RfC) for 1, 4-dioxane. Based on animal data, IRIS classifies 1, 4-dioxane as a probable human carcinogen and provides an oral slope factor, but states that human carcinogenicity data are inadequate. Currently, the EPA is updating the IRIS health assessment for 1, 4-dioxane. The assessment is expected to present reference values for the non-cancer effects of 1, 4-dioxane (RfD and RfC), where supported by available data, and a cancer assessment. The IRIS Tracking System indicates a draft assessment will be released for external peer review in July 2008. However, the Navy acknowledges the State of Maine's Maximum Exceedance Guidance (MEG) of 32 ppb for 1, 4-dioxane. This will be reviewed as part of the proposed analysis and report.

The Navy, along with the USEPA and MEDEP, are responsible for ensuring the protection of human health and the environment. As we jointly develop a path forward on the investigation and remediation of emerging contaminants, we must ensure that this process is based on scientifically sound data. Accordingly, the Navy proposes to continue to monitor for 1, 4-dioxane while the risk assessment for 1, 4-dioxane is conducted and EC guidance and policy is developed by our respective organizations.

The Navy looks forward to discussing this direction with you and the other NASB Stakeholders. Please contact Lonnie Monaco at (215) 897-4911 or me at (215) 897-4915 with any questions or concerns.

Sincerely,

Dawn C. Kincaid

Dawn C. Kincaid, P.E.
BRAC Environmental Coordinator
By Direction of BRAC PMO

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Carolyn LePage - LePage Environmental
Al Easterday – ECC
Linda Klink – TtNUS
Bonnie Capito –NAVFAC Atlantic (Admin Record)
Lonnie Monaco, Jennifer Wright, Dan Waddill – LANTDIV
Amy Walker - NAVFAC HQ
Kim Parker-Brown - NAVFAC HQ
Wanda Holmes - CNO N45
Bob Schirmer - Midlant

Appendix C-2

Historical Geology/Hydrogeology

Final
Long-Term Monitoring Plan
Eastern Plume
Naval Air Station Brunswick, Maine

Prepared for

Department of the Navy
Naval Facilities Engineering Command
BRAC Program Management Office – Northeast
4911 South Broad Street
Philadelphia, Pennsylvania 19112-1303

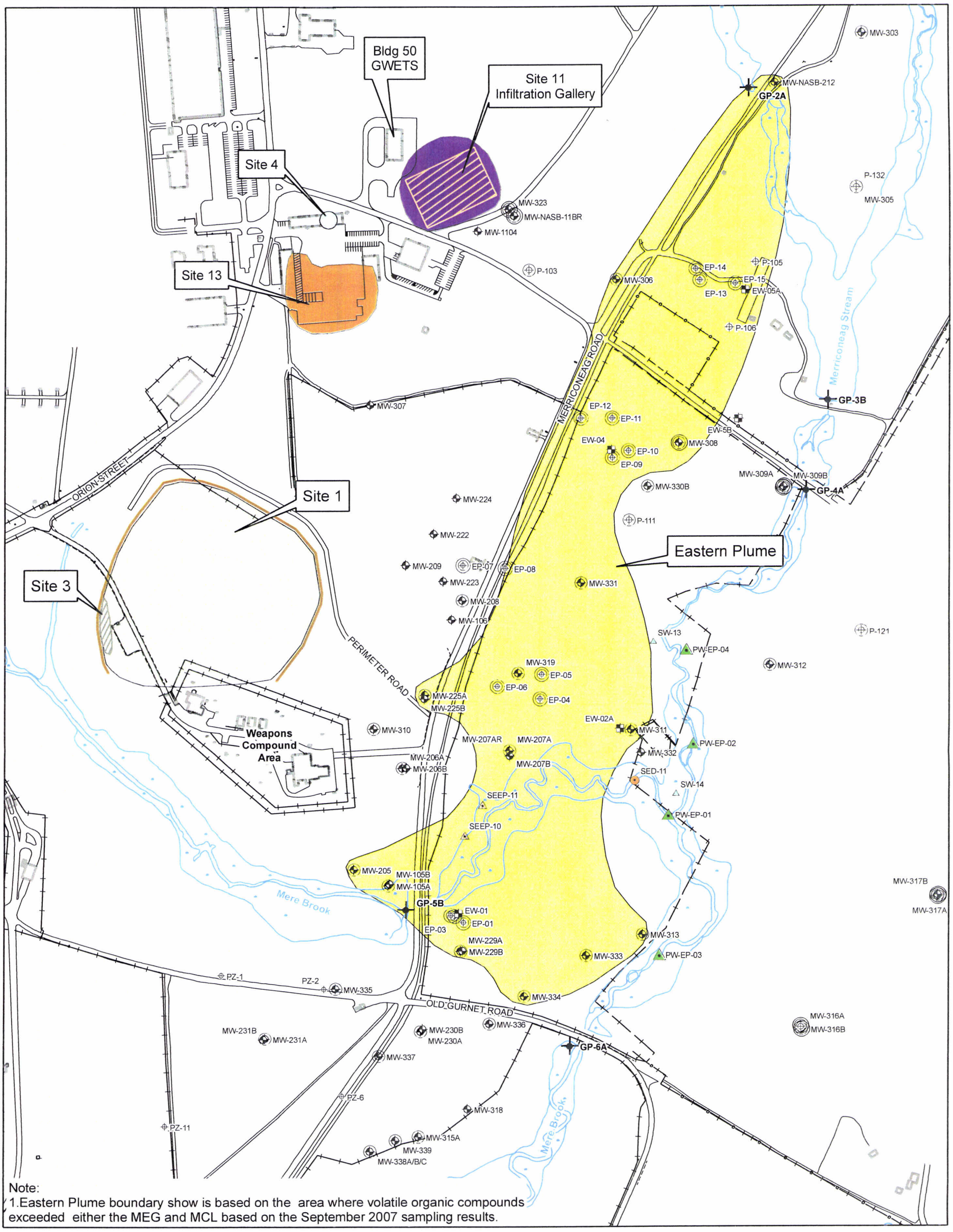
Contract No. N62472-02-D-0810
Contract Task Order No. 017

February 2008

Prepared by

ECC
33 Boston Post Road West, Suite 340
Marlborough, Massachusetts 01752

February 2008
Version: Final; Revision: 1
5700.017

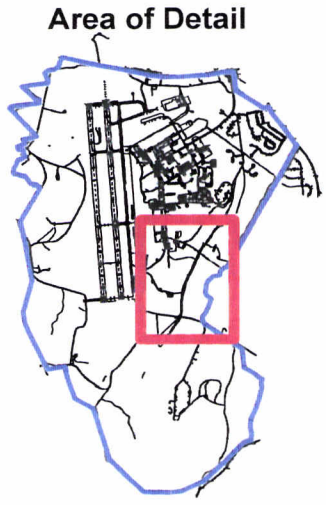


Note:
1.Eastern Plume boundary show is based on the area where volatile organic compounds exceeded either the MEG and MCL based on the September 2007 sampling results.

Contract No.	5700.007
Description	NAS Brunswick, ME
Coordinate system	NAD 1983, UTM, Zone 19N in meters
Sources	Naval Base Boundary provided by Navy.

Date	09-AUG-2007	Rev.	Date	App. By
DB	C. Guido	1	12-FEB-08	G. Calderone
CB	G. Calderone			
AB				

Area of Detail



Legend

PW-EP-01	Pore Water Sample Locations
MW- 225B	Shallow Monitoring Well
MW- 105A	Deep Monitoring Well
MW- 308	Bedrock Well
EW-01	Extraction Well
P-111	Shallow P-Series Piezometer
P-106/PZ-1	Deep P-Series Piezometer
EP-03	Deep EP-Series Piezometer
GP-1A	Surface Water Gauging Location
SEEP-10	Seep Station
SED-11	Sediment Station
SW-01	Surface Water
	Approx. Location of Slurry Wall
	Streams and Wetlands
	GWETS Infiltration Gallery
	Inferred Extent of Eastern Plume (Sept '07)
	Approx. Limits of Sites 1 and 3

Figure 2

Site Plan
Eastern Plume

Naval Air Station
Brunswick, Maine

ECC GIS Server
C:\NAVY_GIS\TO07_Brunswick\Site1and3\MapDocuments\EP_LTMP\EP_LTMP_Fig2.mxd

0 150 300 600 Feet

Final
Summary Report
for the Mere Brook and Merriconeag Stream Floodplain and Upland Area Investigation, Eastern Plume

Naval Air Station Brunswick, Maine

Prepared for

Department of the Navy
Naval Facilities Engineering Command
BRAC Program Management Office – Northeast
4911 South Broad Street
Philadelphia, Pennsylvania 19112-1303

Contract No. N62472-02-D-0810
Contract Task Order No. 017

July 2008

Prepared by

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33 Boston Post Road West, Suite 340
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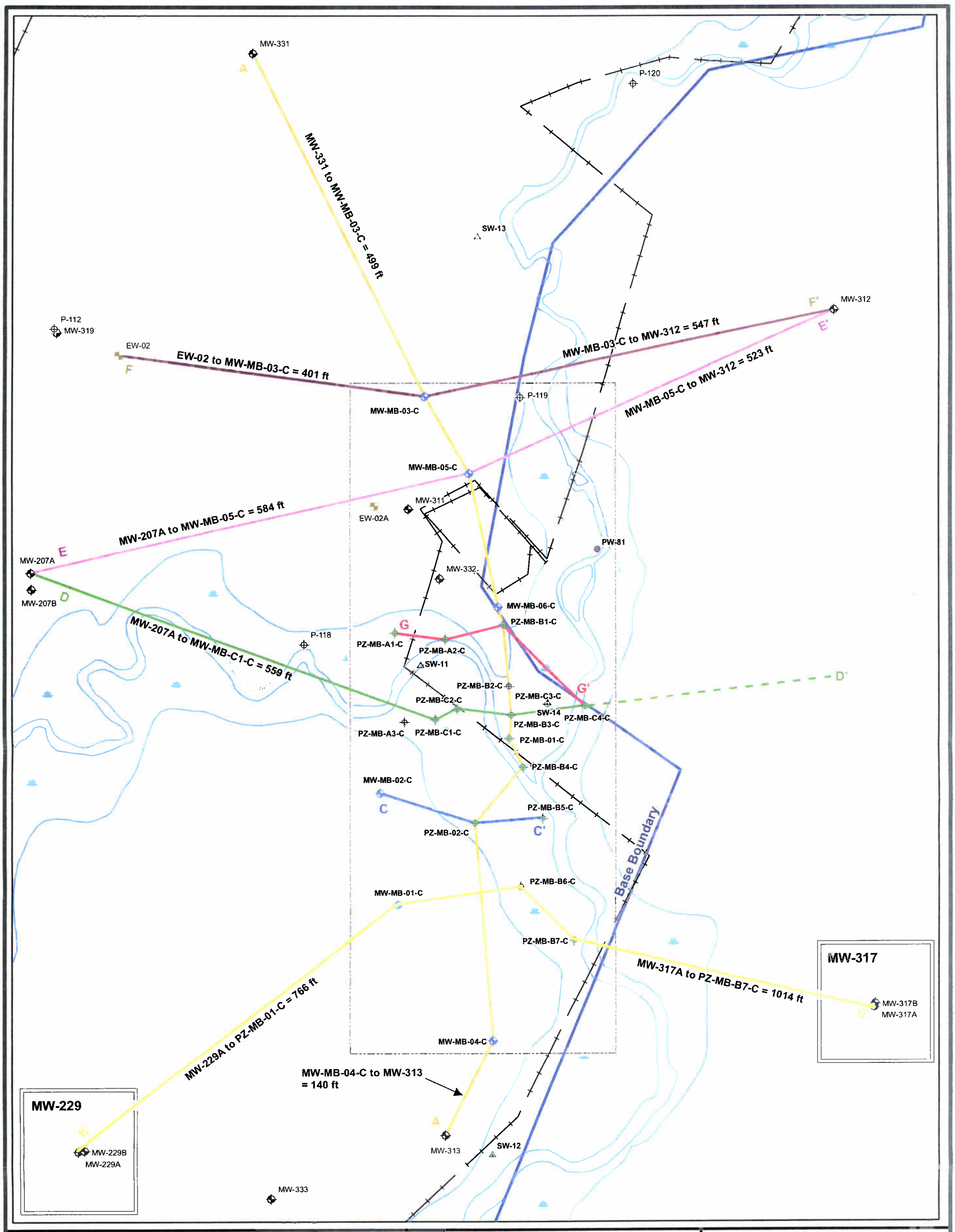
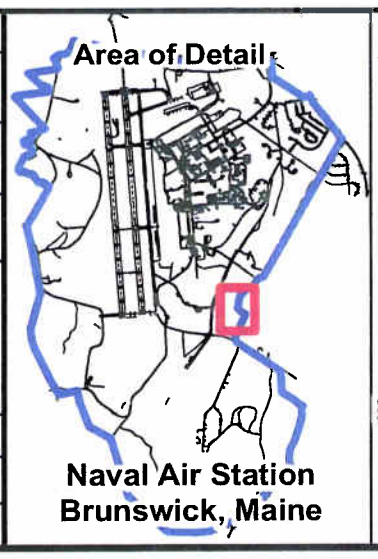
July 2008
Version: Final; Revision: 0
5700.017



Contract No.	N624R-02-D-0810
Description	Cross Sections in Mere Brook in NASB, ME
Coordinate system	NAD 1983, UTM, Zone 19N in meters
Note	
Sources	Naval Base Boundary provided by the Navy.

Date	13-FEB-2008	Rev.	Date	App. By
DB	C. Guido			
CB	J. Donovan			
AB				



LEGEND

Recently Installed Locations
- Monitoring Well
- Piezometer

Existing Locations
- Existing Extraction Well
- Existing Monitoring Well
- Existing Piezometer
- Surface Water Location
- Pore Water Location
- Base Boundary
- Approximate Area of Investigation

Geologic Cross-Sections
- A-A'
- B-B'
- C-C'
- D-D'
- E-E'
- F-F'
- G-G'

Figure 4

Geologic Cross-Section Locations

Mere Brook/Merriconeag Stream
Flood Plain & Upland Area Investigation

ECC GIS Server
C:\NAVY_GIS\TO07_Brunswick\MereBrook\MapDocuments\CompletionReport_Nov07\Fig4_MereBrook_CrossSections.mxd

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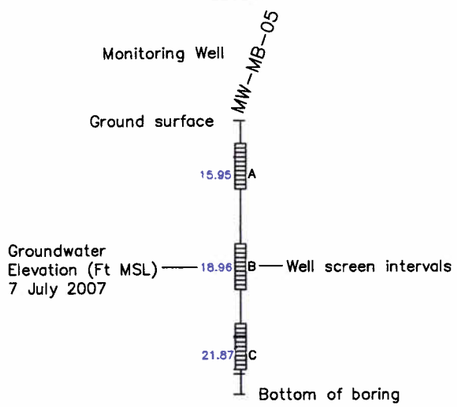
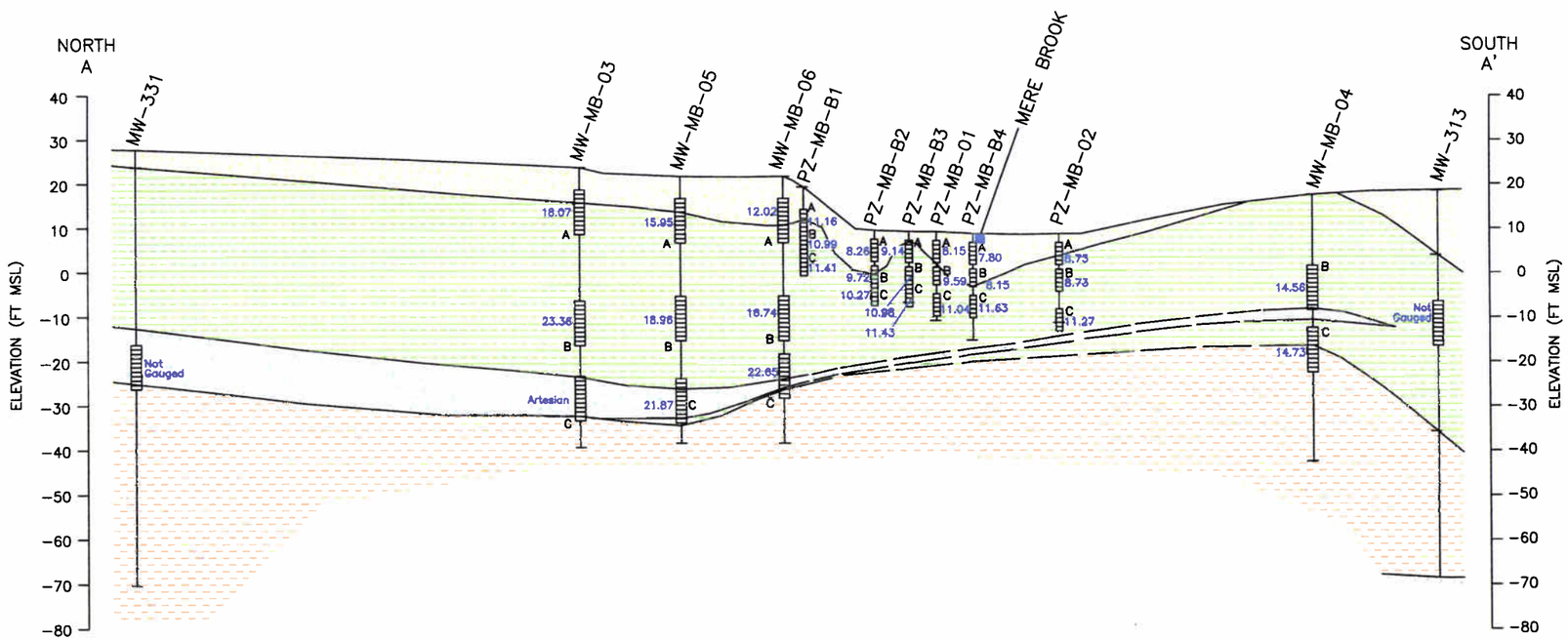
LEGEND



SOIL DESCRIPTION

UPPER SAND:	Tan to light brown fine sand, trace to little medium sand and silt, poorly graded, loose to moderately dense, slightly stratified with depth.
TRANSITION:	Predominantly gray very fine to medium sand (coarser in isolated areas) in layers ranging from less than 0.5-ft up to 8-ft thick, interbedded with silt, silty sands, silty clay, or clay in layers ranging from fine lamina fractions of an inch thick up to 5.5-ft thick. Significant sand interbeds (confirmed in soil boring logs) greater than 2-ft thick are shown in the cross-section
LOWER SAND:	Gray/brown very fine to coarse sand.
PRESUMPSCOT CLAY:	Gray silty clay to clayey silt (rock flour), medium to high plasticity, very soft.
BEDROCK:	Highly foliated, mica (biotite) schist; higher metamorphic grade quartz, garnet and biotite also common; slightly to moderately weathered, not fractured



Contract No.	N624R-02-D-0810
Description	Cross-sections in Mere Brook at NASB, ME
Coordinate system	NAD 1983, UTM, Zone 19N in feet
Notes	1. Soil and bedrock contacts are inferred below and between locations. Dashed line indicates contacts are less precisely located. 2. The geologic unit depths shown are based on soil boring records 3. Ground elevations along cross section line from USGS Quad
Sources	1. Soil boring logs

Date	16-JAN-2008	Rev.	Date	App. By
DB	J. Gatherer			
CB	J. Donovan			
AB				

0 100 200 400

GRAPHIC SCALE IN FEET
VERTICAL EXAGGERATION = 5X

INDEX MAP

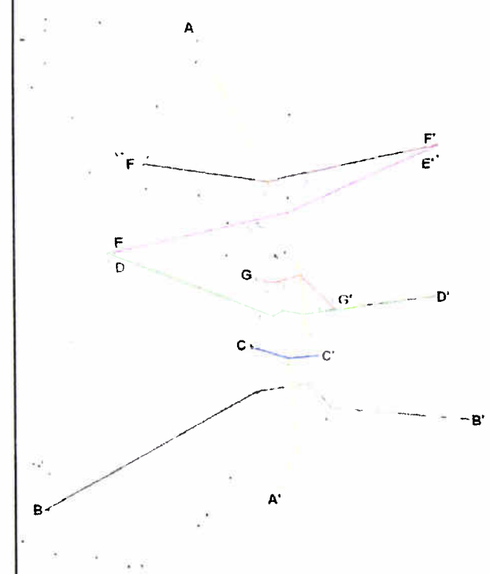


FIGURE 5

CROSS-SECTION A-A'
MERE BROOK, EASTERN PLUME

NAVAL AIR STATION
BRUNSWICK, MAINE

LEGEND

Monitoring Well — MW-MB-05

Ground surface

15.95 A

Groundwater Elevation (Ft MSL) 7 July 2007 — 18.96 B — Well screen intervals

21.87 C

Bottom of boring

SOIL DESCRIPTION

	UPPER SAND:	Tan to light brown fine sand, trace to little medium sand and silt, poorly graded, loose to moderately dense, slightly stratified with depth.
	TRANSITION:	Predominantly gray very fine to medium sand (coarser in isolated areas) in layers ranging from less than 0.5-ft up to 8-ft thick, interbedded with silt, silty sands, silty clay, or clay in layers ranging from fine lamina fractions of an inch thick up to 5.5-ft thick. Significant sand interbeds (confirmed in soil boring logs) greater than 2-ft thick are shown in the cross-section
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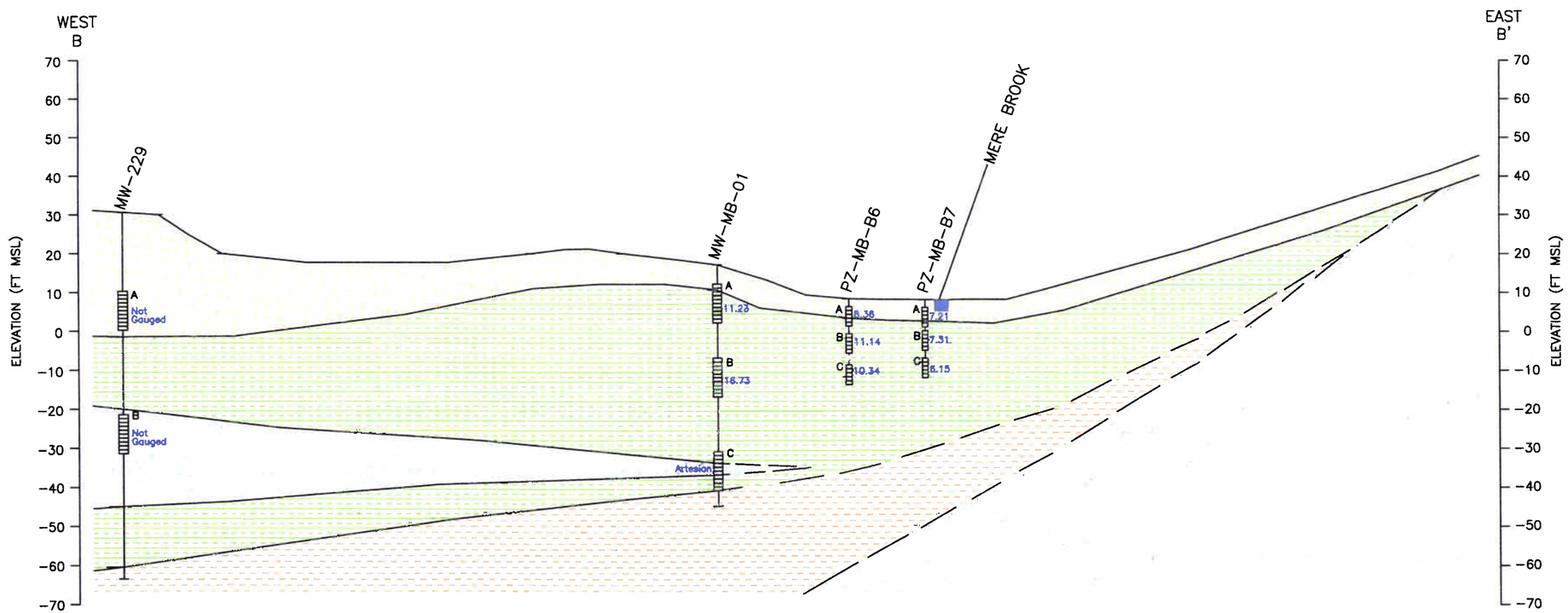


Contract No.	N624R-02-D-0810
Description	Cross-sections in Mere Brook at NASB, ME
Coordinate system	NAD 1983, UTM, Zone 19N in feet
Notes	1. Soil and bedrock contacts are inferred below and between locations. Dashed line indicates contacts are less precisely located. 2. The geologic unit depths shown are based on soil boring records 3. Ground elevations along cross section line from USGS Quad
Sources	1. Soil boring logs

Date	16-JAN-2008	Rev.	Date	App. By
DB	J. Gatherer			
CB	J. Donovan			
AB				

0 100 200 400

GRAPHIC SCALE IN FEET
VERTICAL EXAGGERATION = 5X

INDEX MAP

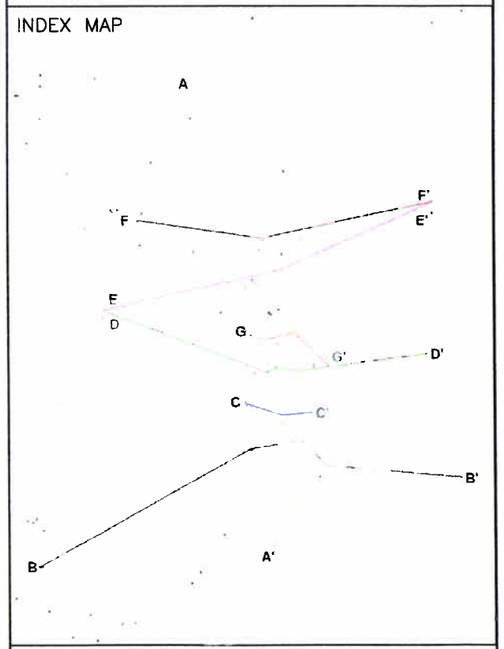
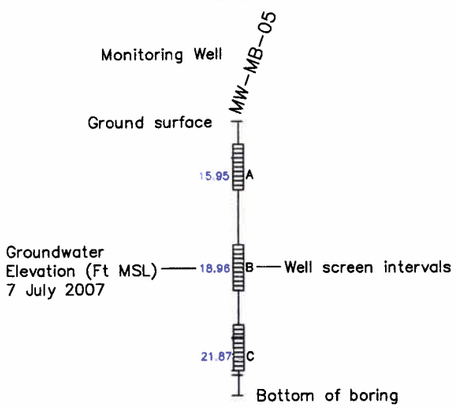


FIGURE 6

CROSS-SECTION B-B'
MERE BROOK, EASTERN PLUME

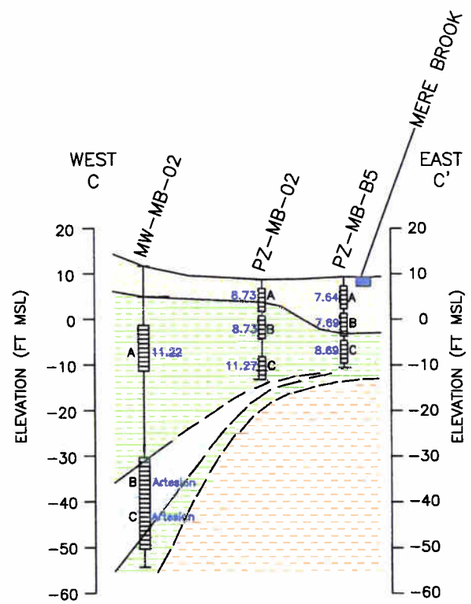
NAVAL AIR STATION
BRUNSWICK, MAINE

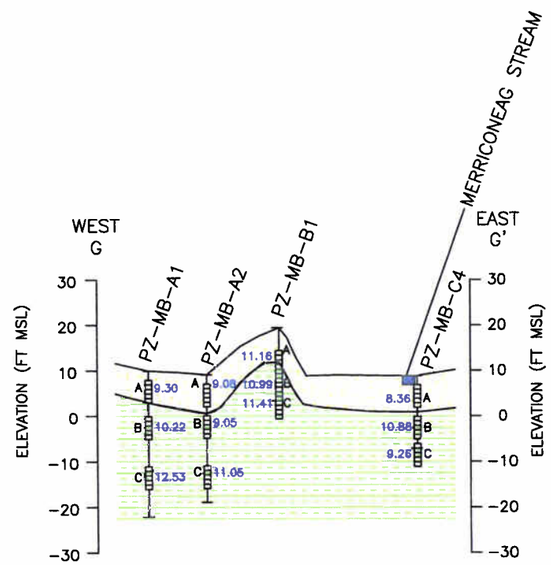
LEGEND



SOIL DESCRIPTION

	UPPER SAND:	Tan to light brown fine sand, trace to little medium sand and silt, poorly graded, loose to moderately dense, slightly stratified with depth.
	TRANSITION:	Predominantly gray very fine to medium sand (coarser in isolated areas) in layers ranging from less than 0.5-ft up to 8-ft thick, interbedded with silt, silty sands, silty clay, or clay in layers ranging from fine lamina fractions of an inch thick up to 5.5-ft thick. Significant sand interbeds (confirmed in soil boring logs) greater than 2-ft thick are shown in the cross-section
	LOWER SAND:	Gray/brown very fine to coarse sand.
	PRESUMPSCOT CLAY:	Gray silty clay to clayey silt (rock flour), medium to high plasticity, very soft.
	BEDROCK:	Highly foliated, mica (biotite) schist; higher metamorphic grade quartz, garnet and biotite also common; slightly to moderately weathered, not fractured





Contract No.	N624R-02-D-0810
Description	Cross-sections in Mere Brook at NASB, ME
Coordinate system	NAD 1983, UTM, Zone 19N in feet
Notes	1. Soil and bedrock contacts are inferred below and between locations. Dashed line indicates contacts are less precisely located. 2. The geologic unit depths shown are based on soil boring records 3. Ground elevations along cross section line from USGS Quad
Sources	1. Soil boring logs

Date	16-JAN-2008	Rev.	Date	App. By
DB	J. Gatherer			
CB	J. Donovan			
AB				

0 100 200 400

GRAPHIC SCALE IN FEET
VERTICAL EXAGGERATION = 5X

INDEX MAP

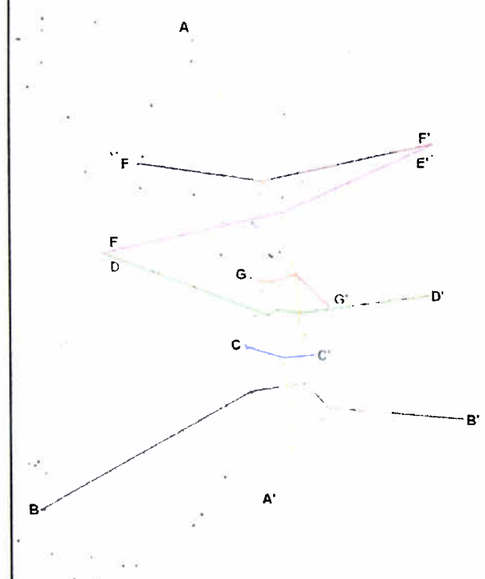
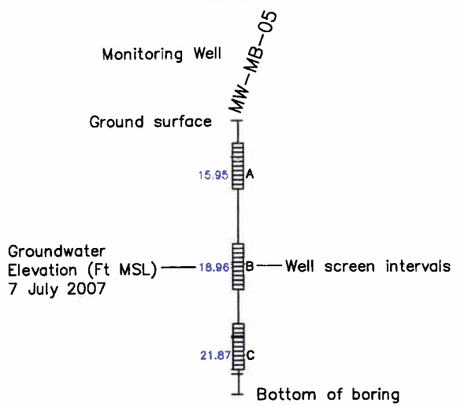


FIGURE 7

CROSS-SECTION C-C' AND G-G'
MERE BROOK, EASTERN PLUME

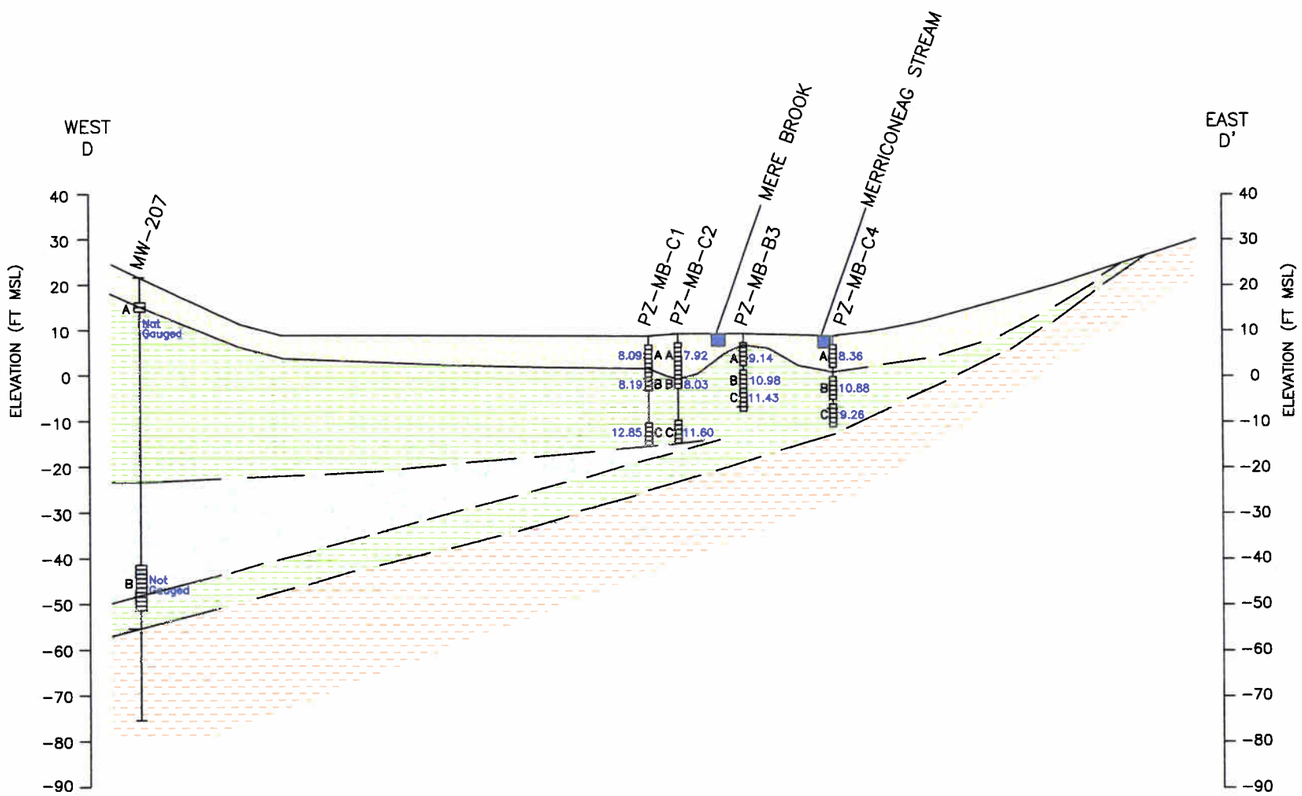
NAVAL AIR STATION
BRUNSWICK, MAINE

LEGEND



SOIL DESCRIPTION

UPPER SAND:	Tan to light brown fine sand, trace to little medium sand and silt, poorly graded, loose to moderately dense, slightly stratified with depth.
TRANSITION:	Predominantly gray very fine to medium sand (coarser in isolated areas) in layers ranging from less than 0.5-ft up to 8-ft thick, interbedded with silt, silty sands, silty clay, or clay in layers ranging from fine lamina fractions of an inch thick up to 5.5-ft thick. Significant sand interbeds (confirmed in soil boring logs) greater than 2-ft thick are shown in the cross-section
LOWER SAND:	Gray/brown very fine to coarse sand.
PRESUMPSCOT CLAY:	Gray silty clay to clayey silt (rock flour), medium to high plasticity, very soft.
BEDROCK:	Highly foliated, mica (biotite) schist; higher metamorphic grade quartz, garnet and biotite also common; slightly to moderately weathered, not fractured



Contract No.	N624R-02-D-0810
Description	Cross-sections in Mere Brook at NASB, ME
Coordinate system	NAD 1983, UTM, Zone 19N in feet
Notes	1. Soil and bedrock contacts are inferred below and between locations. Dashed line indicates contacts are less precisely located. 2. The geologic unit depths shown are based on soil boring records 3. Ground elevations along cross section line from USGS Quad
Sources	1. Soil boring logs

Date	16-JAN-2008	Rev.	Date	App. By
DB	J. Gatherer			
CB	J. Donovan			
AB				

0 100 200 400

GRAPHIC SCALE IN FEET
VERTICAL EXAGGERATION = 5X

INDEX MAP

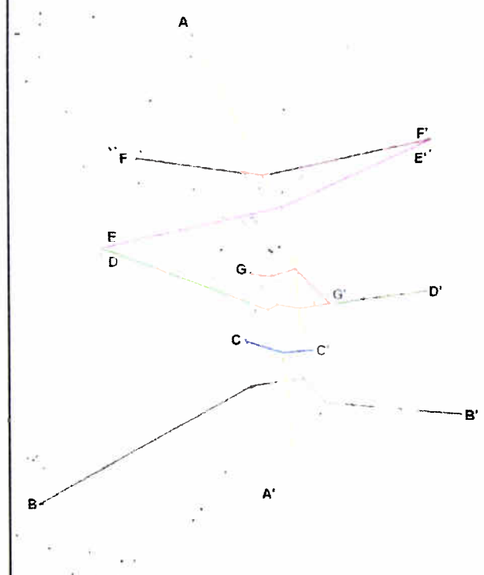
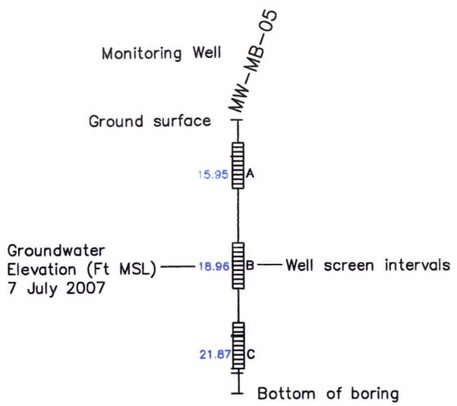


FIGURE 8

CROSS-SECTION D-D'
MERE BROOK, EASTERN PLUME

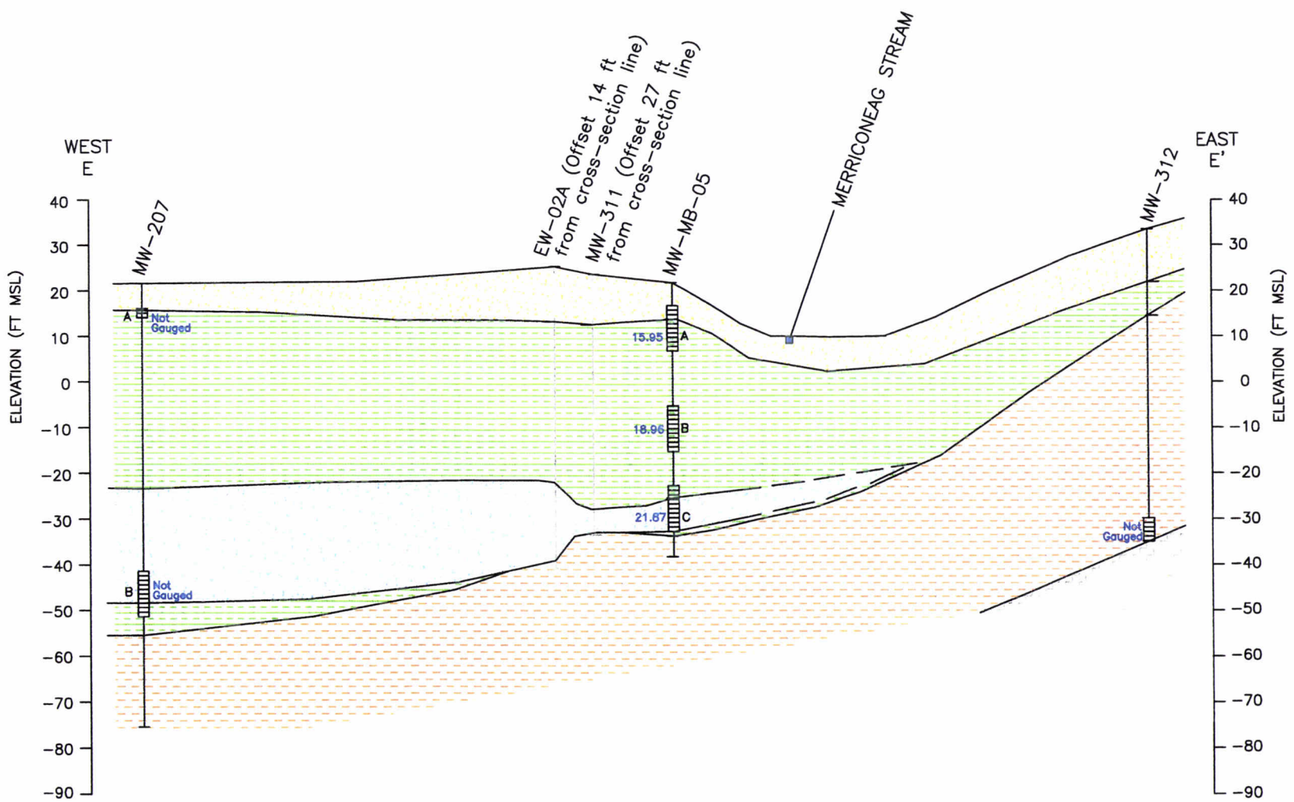
NAVAL AIR STATION
BRUNSWICK, MAINE

LEGEND



SOIL DESCRIPTION

UPPER SAND:	Tan to light brown fine sand, trace to little medium sand and silt, poorly graded, loose to moderately dense, slightly stratified with depth.
TRANSITION:	Predominantly gray very fine to medium sand (coarser in isolated areas) in layers ranging from less than 0.5–ft up to 8–ft thick, interbedded with silt, silty sands, silty clay, or clay in layers ranging from fine lamina fractions of an inch thick up to 5.5–ft thick. Significant sand interbeds (confirmed in soil boring logs) greater than 2–ft thick are shown in the cross–section
LOWER SAND:	Gray/brown very fine to coarse sand.
PRESUMPSCOT CLAY:	Gray silty clay to clayey silt (rock flour), medium to high plasticity, very soft.
BEDROCK:	Highly foliated, mica (biotite) schist; higher metamorphic grade quartz, garnet and biotite also common; slightly to moderately weathered, not fractured



Contract No.	N624R–02–D–0810
Description	Cross–sections in Mere Brook at NASB, ME
Coordinate system	NAD 1983, UTM, Zone 19N in feet
Notes	1. Soil and bedrock contacts are inferred below and between locations. Dashed line indicates contacts are less precisely located. 2. The geologic unit depths shown are based on soil boring records 3. Ground elevations along cross section line from USGS Quad
Sources	1. Soil boring logs

Date	16–JAN–2008	Rev.	Date	App. By
DB	J. Gatherer			
CB	J. Donovan			
AB				

0 100 200 400

GRAPHIC SCALE IN FEET
VERTICAL EXAGGERATION = 5X

INDEX MAP

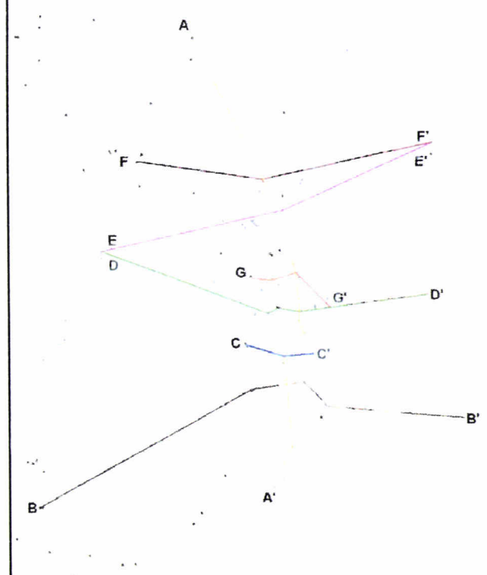
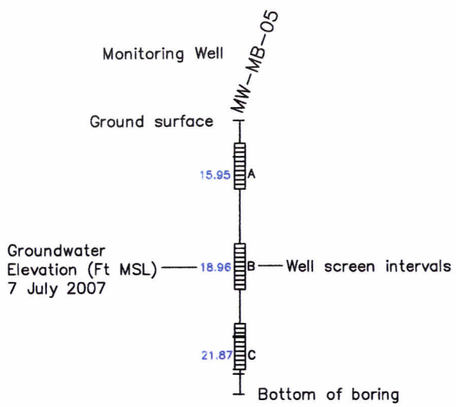


FIGURE 9

CROSS–SECTION E–E'
MERE BROOK, EASTERN PLUME

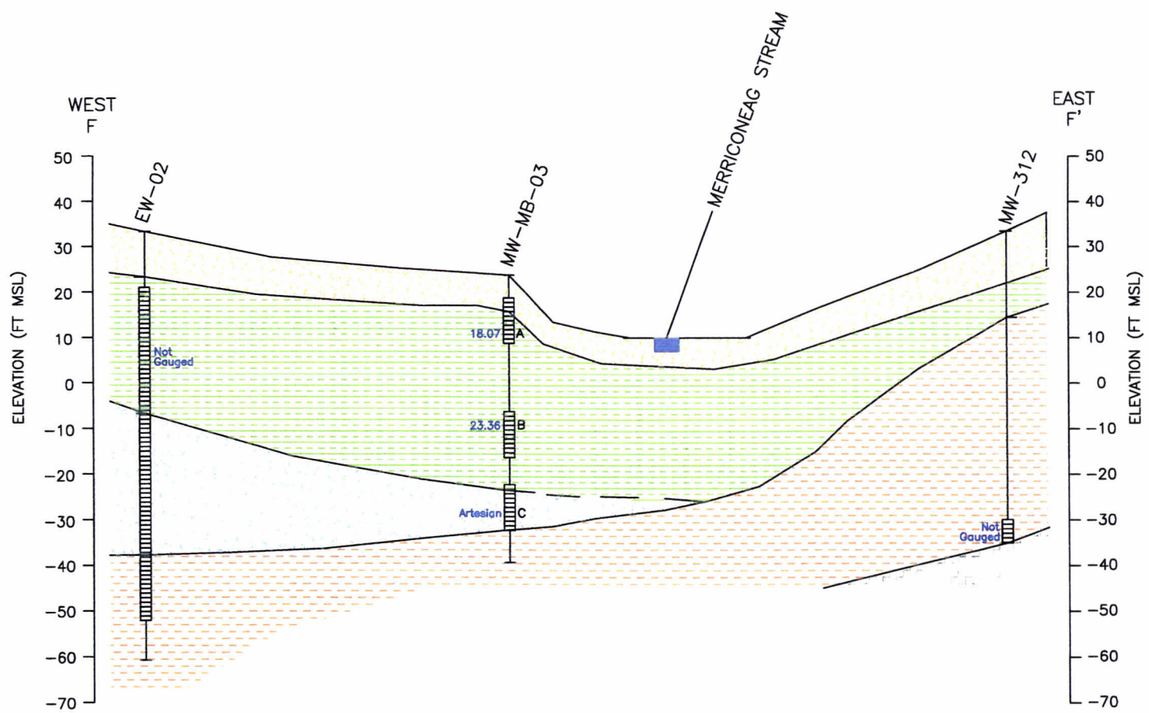
NAVAL AIR STATION
BRUNSWICK, MAINE

LEGEND



SOIL DESCRIPTION

UPPER SAND:	Tan to light brown fine sand, trace to little medium sand and silt, poorly graded, loose to moderately dense, slightly stratified with depth.
TRANSITION:	Predominantly gray very fine to medium sand (coarser in isolated areas) in layers ranging from less than 0.5-ft up to 8-ft thick, interbedded with silt, silty sands, silty clay, or clay in layers ranging from fine lamina fractions of an inch thick up to 5.5-ft thick. Significant sand interbeds (confirmed in soil boring logs) greater than 2-ft thick are shown in the cross-section
LOWER SAND:	Gray/brown very fine to coarse sand.
PRESUMPSCOT CLAY:	Gray silty clay to clayey silt (rock flour), medium to high plasticity, very soft.
BEDROCK:	Highly foliated, mica (biotite) schist; higher metamorphic grade quartz, garnet and biotite also common; slightly to moderately weathered, not fractured



Contract No.	N624R-02-D-0810
Description	Cross-sections in Mere Brook at NASB, ME
Coordinate system	NAD 1983, UTM, Zone 19N in feet
Notes	1. Soil and bedrock contacts are inferred below and between locations. Dashed line indicates contacts are less precisely located. 2. The geologic unit depths shown are based on soil boring records 3. Ground elevations along cross section line from USGS Quad
Sources	1. Soil boring logs

Date	16-JAN-2008	Rev.	Date	App. By
DB	J. Gatherer			
CB	J. Donovan			
AB				

0 100 200 400

GRAPHIC SCALE IN FEET
VERTICAL EXAGGERATION = 5X

INDEX MAP

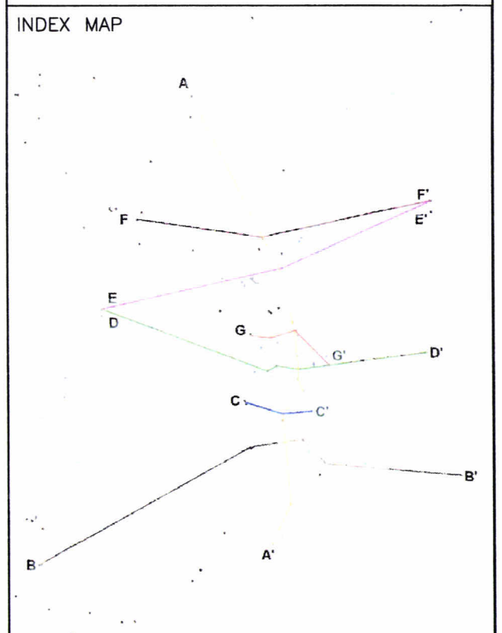


FIGURE 10

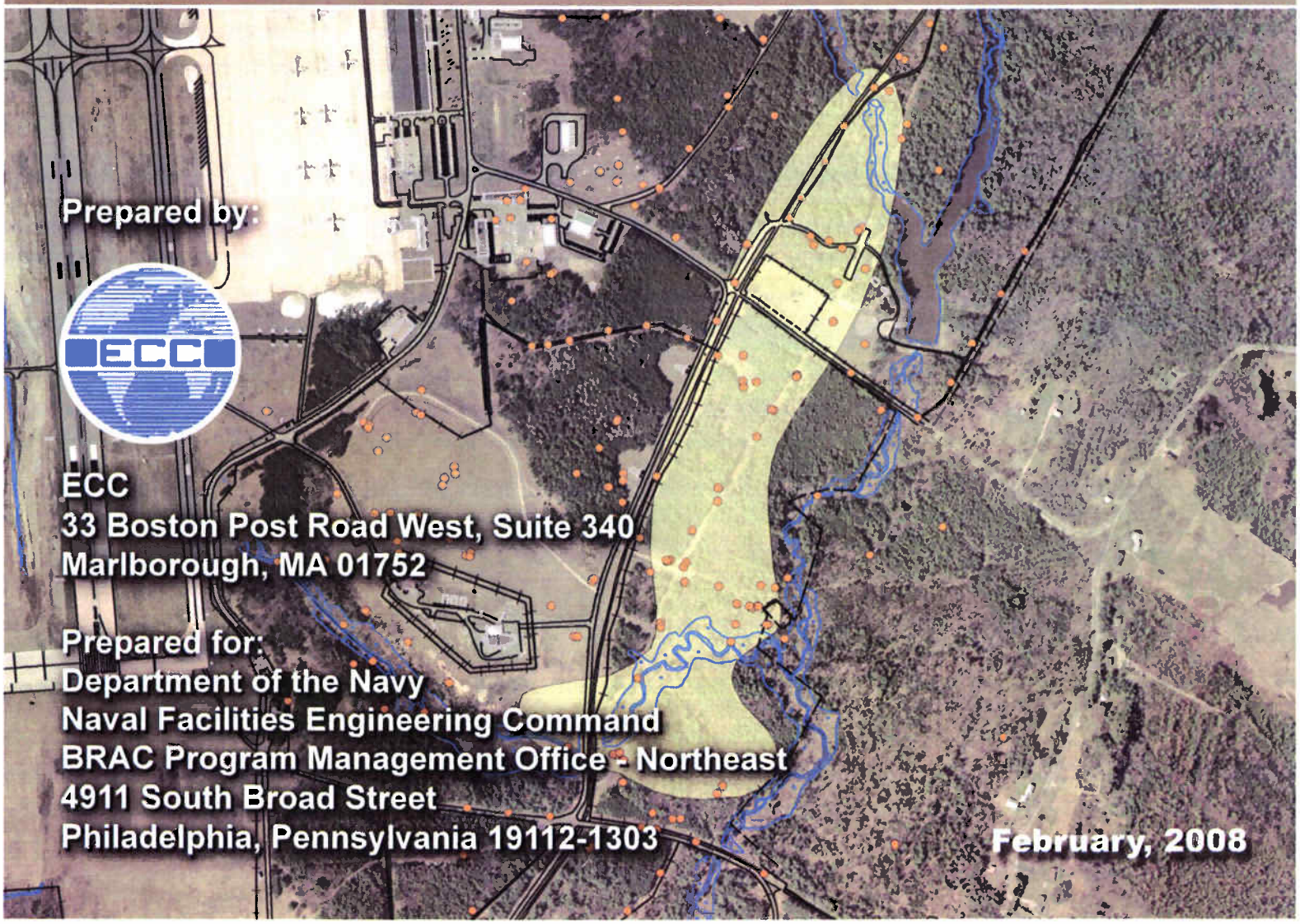
CROSS-SECTION F-F'
MERE BROOK, EASTERN PLUME

NAVAL AIR STATION
BRUNSWICK, MAINE

Draft
Sites 1, 3 & Eastern Plume Monitoring Event 31 Report September 2007

Contract No. N62472-02-D-0810
Contract Task Order No. 007

Naval Air Station Brunswick, Maine

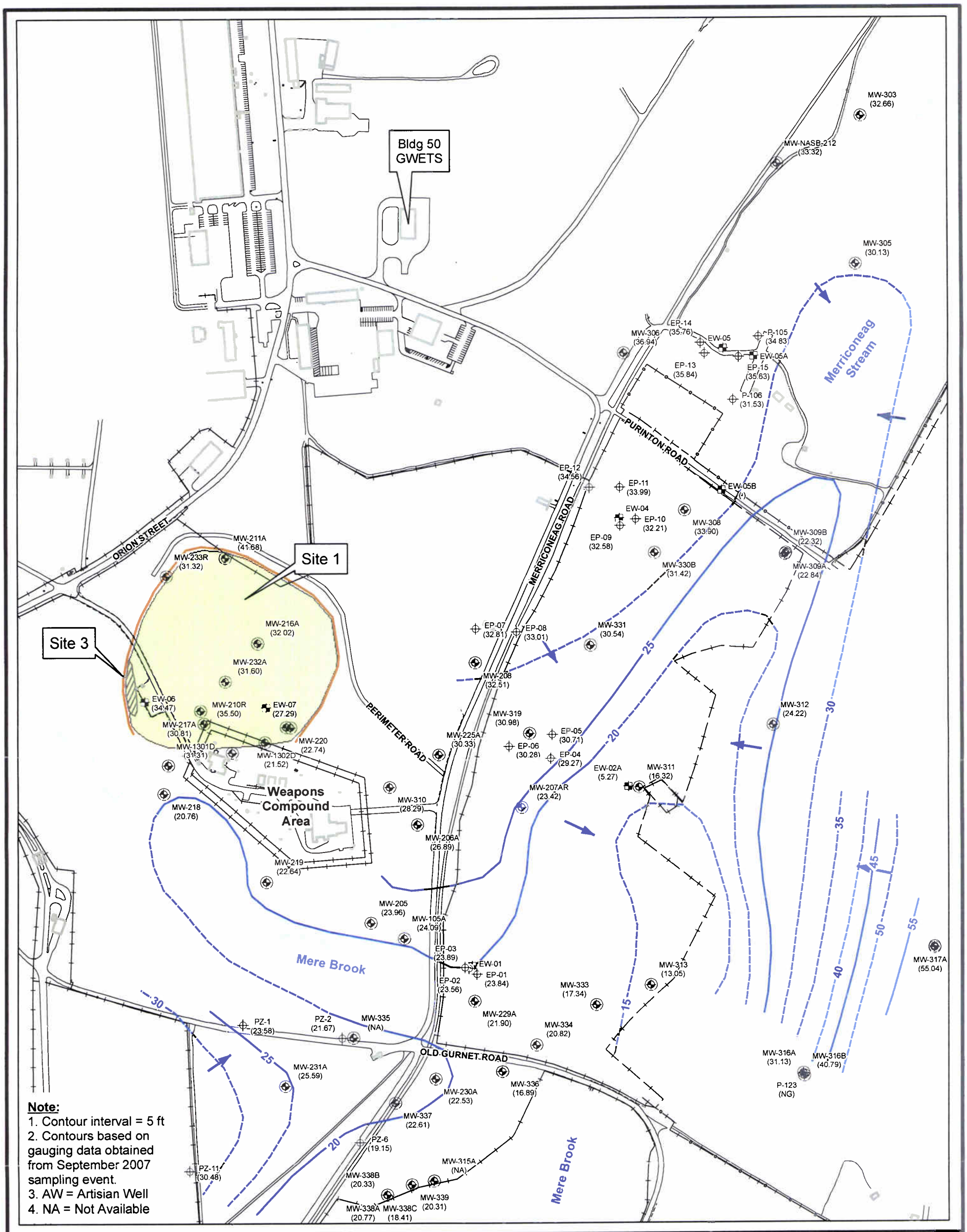


Prepared by:

ECC
33 Boston Post Road West, Suite 340
Marlborough, MA 01752

Prepared for:
Department of the Navy
Naval Facilities Engineering Command
BRAC Program Management Office - Northeast
4911 South Broad Street
Philadelphia, Pennsylvania 19112-1303

February, 2008

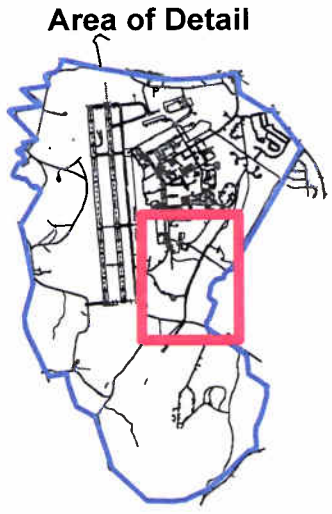


Note:
1. Contour interval = 5 ft
2. Contours based on gauging data obtained from September 2007 sampling event.
3. AW = Artisian Well
4. NA = Not Available

Contract No.	5700.007
Description	NAS Brunswick, ME
Coordinate system	NAD 1983, UTM, Zone 19N in meters
Sources	Naval Base Boundary provided by Navy.

Date	05-FEB-2008	Rev.	Date	App. By
DB	C. Guido			
CB	G. Calderone			
AB				

Area of Detail



Legend

- MW-315A (ND) — Deep Monitoring Well (Groundwater Elevation, ft MSL)
- P-106 (32.29) — Deep Piezometer (Groundwater Elevation, ft MSL)
- EW-02A (0.41) — Extraction Well (Groundwater Elevation, ft MSL)
- Groundwater Contour (ft MSL)
- Inferred Groundwater Contour (ft MSL)
- Groundwater Flow Direction
- Approx. Location of Slurry Wall
- Approx. Limits of Sites 1 and 3
- GWETS Infiltration Gallery

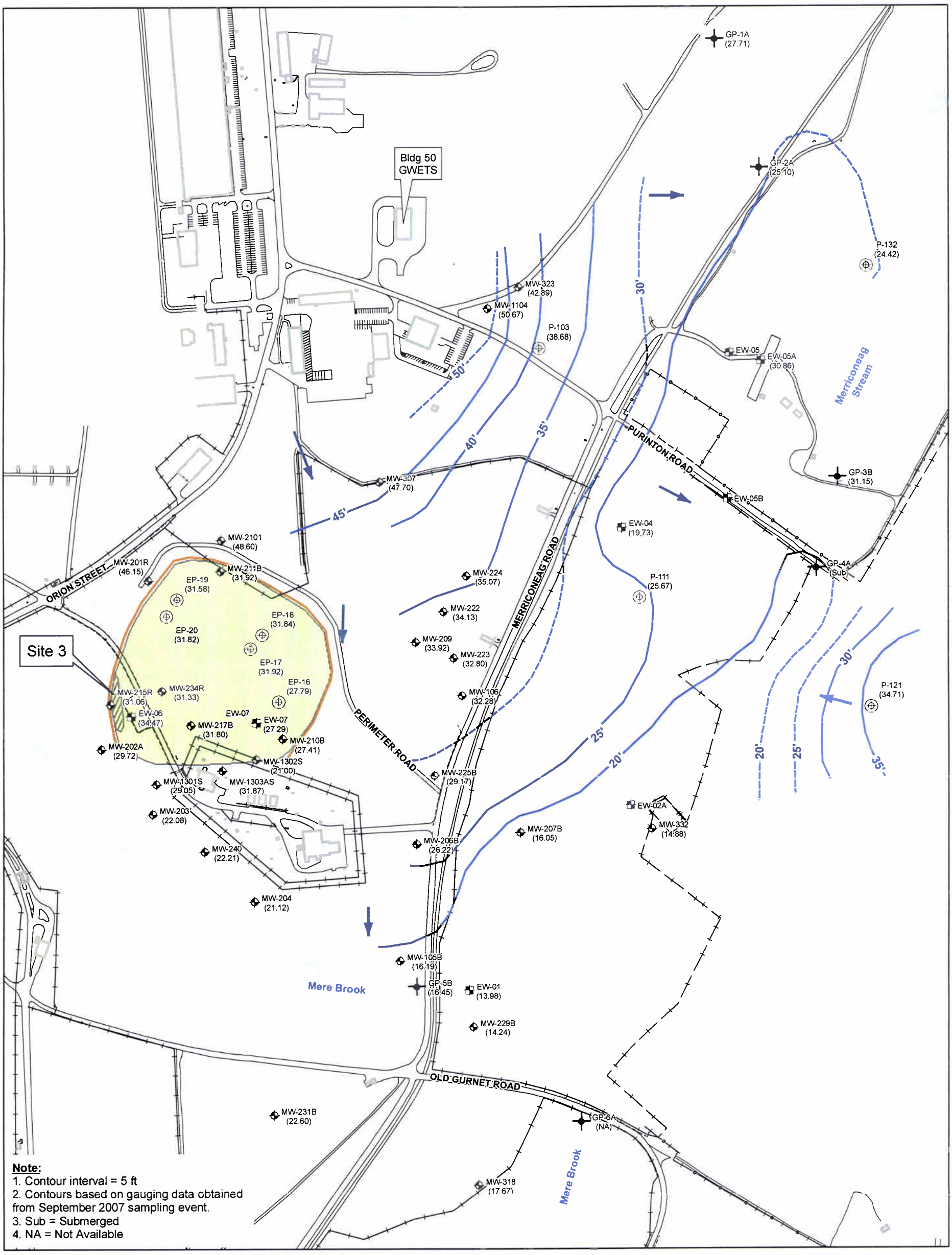
Figure 1-5

Interpreted Deep Groundwater Surface Contour Map (September 2007)

Naval Air Station
Brunswick, Maine

GIS Server
C:\NAVY_GIS\TO07_Brunswick\Site1and3\MapDocuments\EP_Sept07\EP_Sep07_Fig1-5_DeepGWContours.mxd

0 150 300 600 Feet

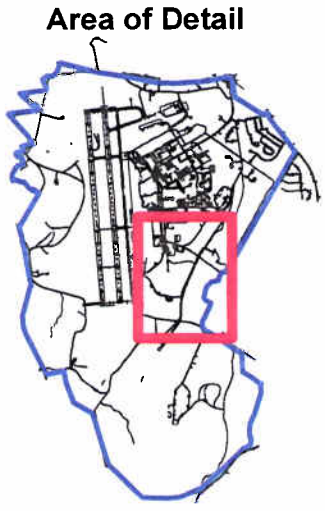


Note:
1. Contour interval = 5 ft
2. Contours based on gauging data obtained from September 2007 sampling event.
3. Sub = Submerged
4. NA = Not Available

Contract No.	5700.007
Description	NAS Brunswick, ME
Coordinate system	NAD 1983, UTM, Zone 19N in meters
Sources	Naval Base Boundary provided by Navy.

Date	05-FEB-2008	Rev.	Date	App. By
DB	C. Guido			
CB	G. Calderone			
AB				

Area of Detail



Legend

- MW-318 (19.46) — Shallow Monitoring Well (Groundwater Elevation, ft MSL)
- GP-1A (28.05) — Surface Water Piezometer (Groundwater Elevation, ft MSL)
- EW-04 (22.14) — Extraction Well (Groundwater Elevation, ft MSL)
- P-111 (27.07) — Shallow Piezometer (Groundwater Elevation, ft MSL)
- Groundwater Flow Direction
- Groundwater Contour (ft MSL)
- Inferred Groundwater Contour (ft MSL)
- Approx. Location of Slurry Wall
- Approx. Limits of Sites 1 and 3
- GWETS Infiltration Gallery

Figure 1-4

Interpreted Shallow Groundwater Surface Contour Map (September 2007)

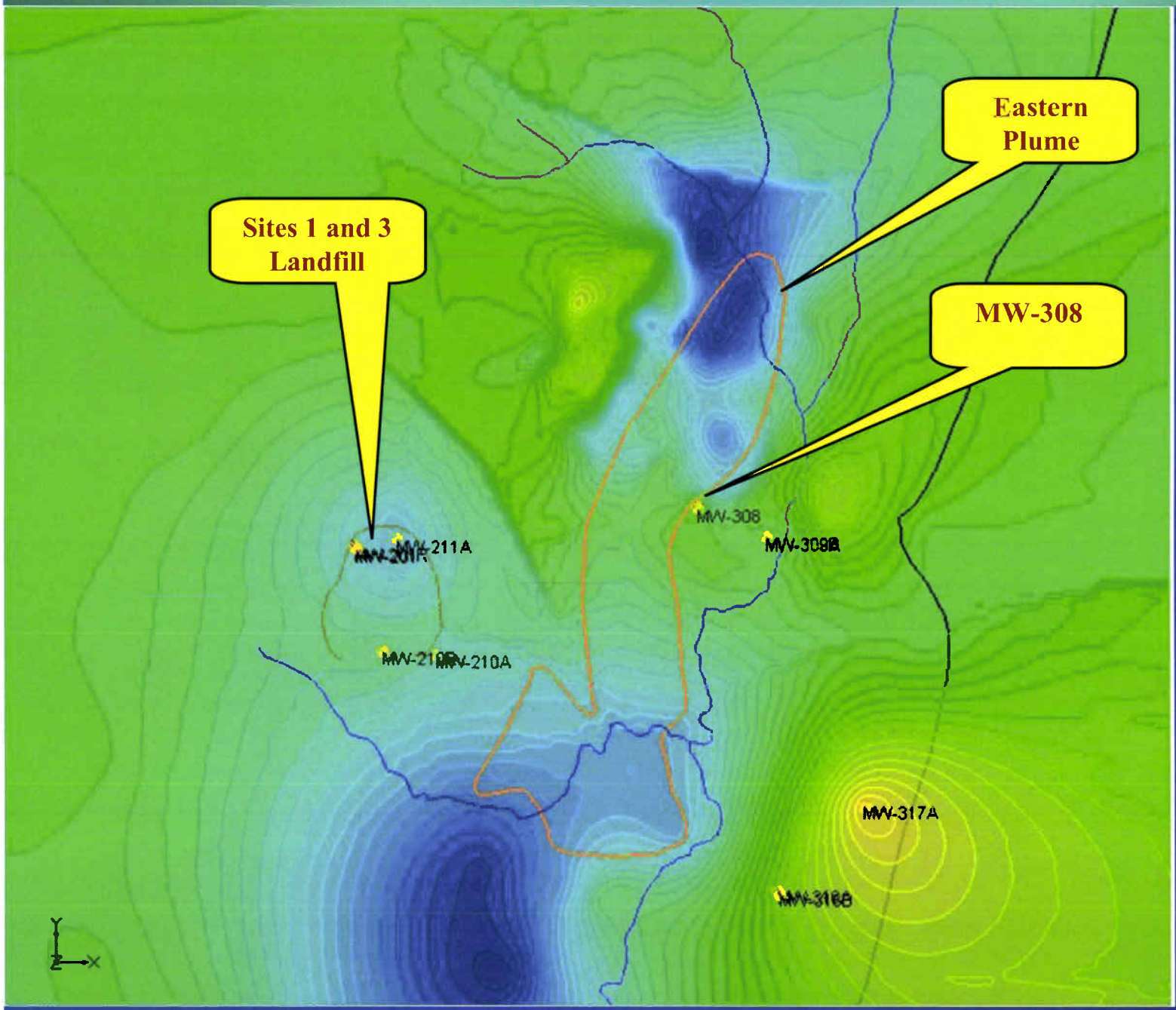
Naval Air Station
Brunswick, Maine

GIS Server
C:\NAVY_GIS\TO07_Brunswick\Site1and3\MapDocuments\EP_April07\EP_April07_Fig1-4_ShallowGWContours.mxd

0 150 300 600 Feet

NAS Brunswick
Technical Meeting
28 February 2008

Overview of Bedrock Character and Bedrock Monitoring at Naval Air Station Brunswick, Maine

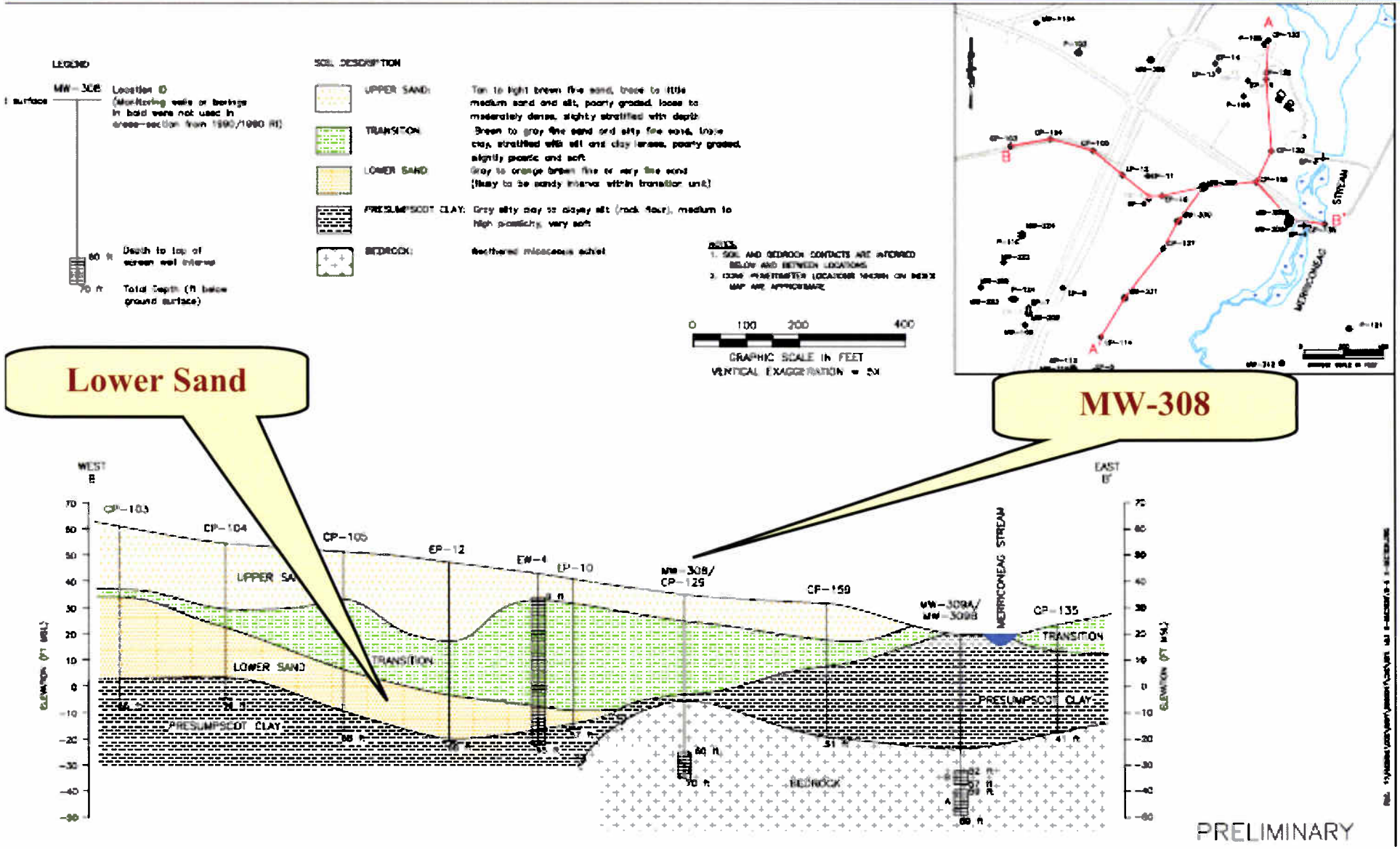


Bedrock Surface Contour Map

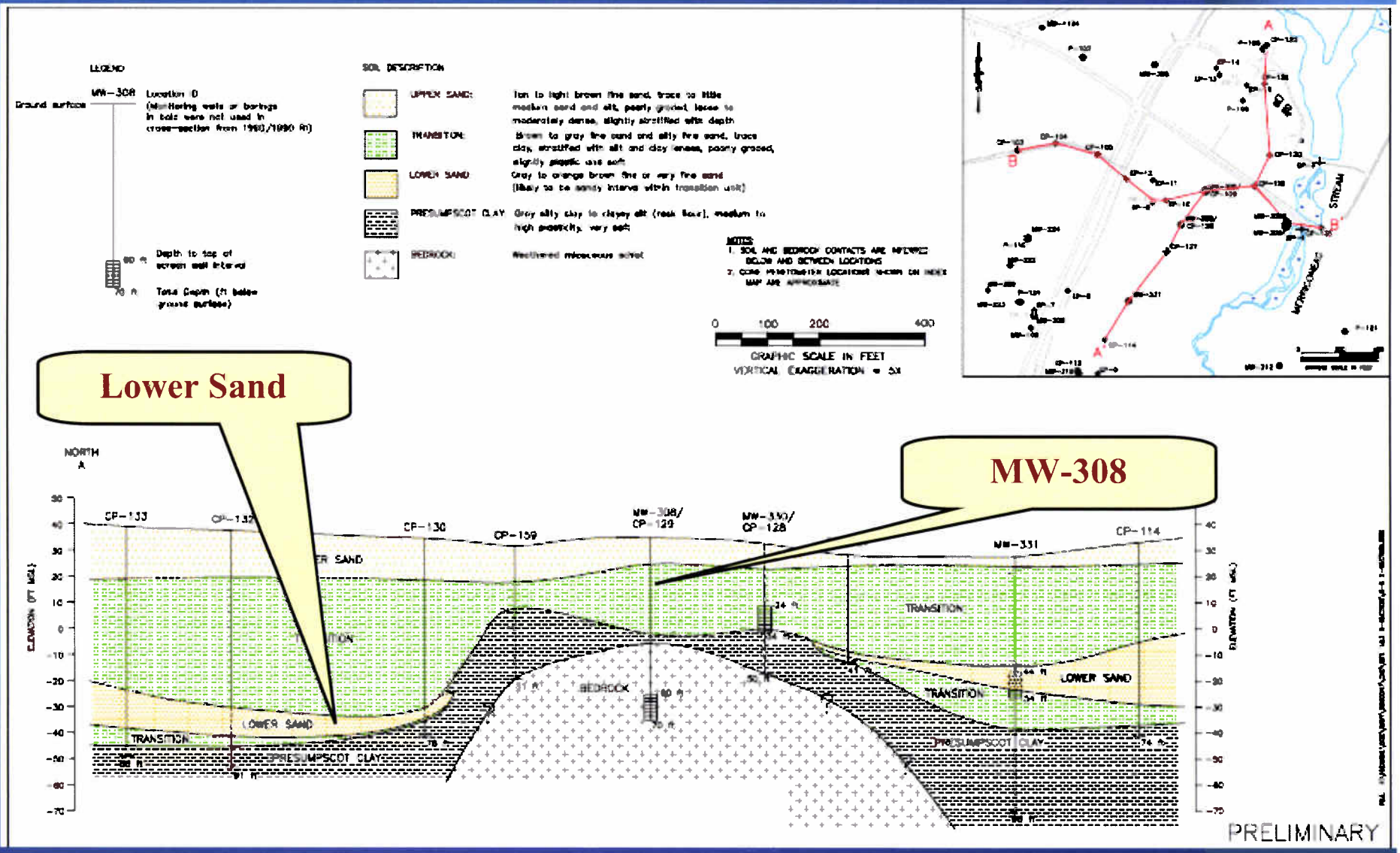
Source: ECC; based on geophysical data and soil boring boring logs

Ft MSL

West-East Cross-Section of Eastern Plume near MW-308



North-South Cross-Section of Eastern Plume near MW-308



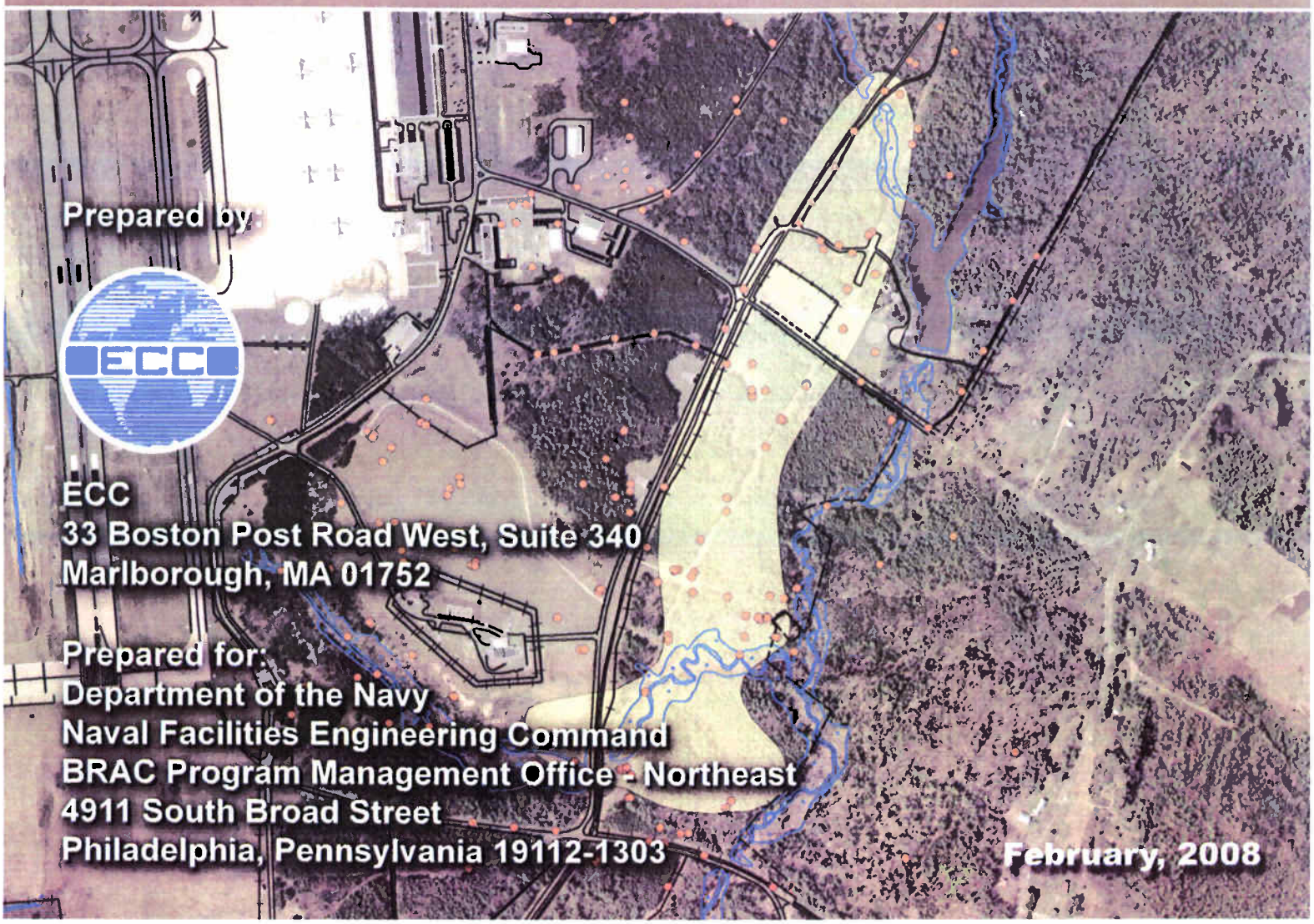
Appendix C-3

Historical 1,4-Dioxane Area Nature and Extent of Contamination

Draft
Sites 1, 3 & Eastern Plume Monitoring Event 31 Report September 2007

Contract No. N62472-02-D-0810
Contract Task Order No. 007

Naval Air Station Brunswick, Maine

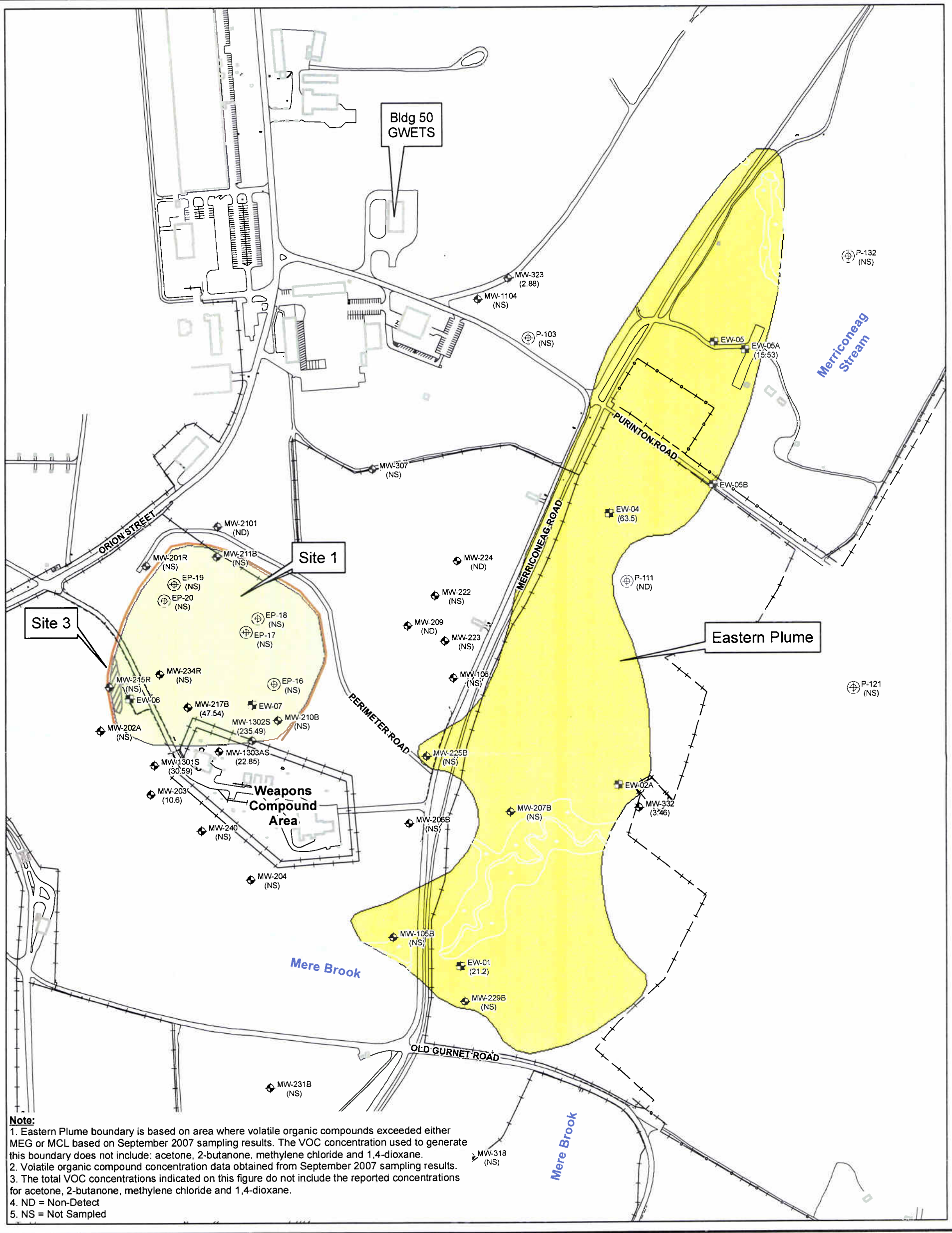


Prepared by:

ECC
33 Boston Post Road West, Suite 340
Marlborough, MA 01752

Prepared for:
Department of the Navy
Naval Facilities Engineering Command
BRAC Program Management Office - Northeast
4911 South Broad Street
Philadelphia, Pennsylvania 19112-1303

February, 2008

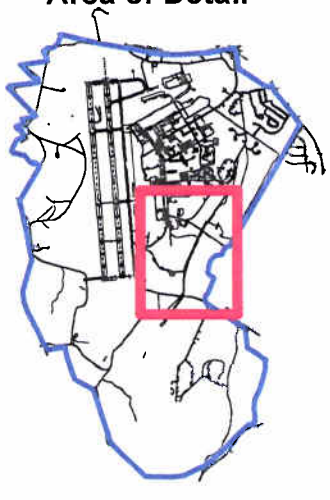


Note:
1. Eastern Plume boundary is based on area where volatile organic compounds exceeded either MEG or MCL based on September 2007 sampling results. The VOC concentration used to generate this boundary does not include: acetone, 2-butanone, methylene chloride and 1,4-dioxane.
2. Volatile organic compound concentration data obtained from September 2007 sampling results.
3. The total VOC concentrations indicated on this figure do not include the reported concentrations for acetone, 2-butanone, methylene chloride and 1,4-dioxane.
4. ND = Non-Detect
5. NS = Not Sampled

Contract No.	5700.007
Description	NAS Brunswick, ME
Coordinate system	NAD 1983, UTM, Zone 19N in meters
Sources	Naval Base Boundary provided by Navy.

Date	04-FEB-2008	Rev.	Date	App. By
DB	C. Guido			
CB	G. Calderone			
AB				

Area of Detail



Legend

- MW-318 (ND) — Shallow Monitoring Well (Total Volatile Organic Compund, ug/L)
- EW-04 (88.72) — Extraction Well (Total Volatile Organic Compund, ug/L)
- P-111 (1.0) — Shallow Piezometer (Total Volatile Organic Compund, ug/L)
- Approx. Location of Slurry Wall
- Approx. Limits of Sites 1 and 3
- GWETS Infiltration Gallery
- Inferred Extent of Eastern Plume

Figure 2-1

Total Volatile Organic Compound Concentration Map Shallow Wells Monitoring Event 31

Naval Air Station
Brunswick, Maine

GIS Server
C:\NAVY_GIS\TO07_Brunswick\Site1and3\MapDocuments\EP_Sept07\EP_Sep07_Fig2-1_ShallowVOC.mxd

0 150 300 600 Feet

Bldg 50
GWETS

Site 1

Site 3

Eastern Plume

Weapons Compound Area

Weapons Compound Area

Merriconeag Stream

Mere Brook

Mere Brook

ORION STREET

PURINTON ROAD

MERRICONEAG ROAD

PERIMETER ROAD

OLD GURNET ROAD

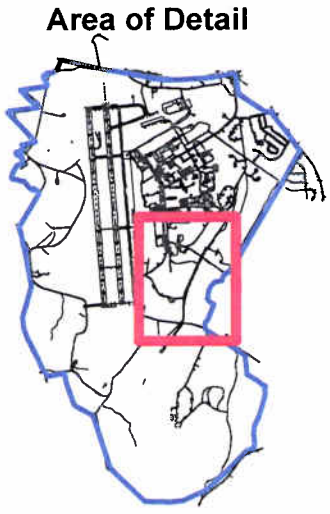
MW-303 (NS)
MW-NASB-212 (7.3)
MW-305 (NS)
EP-14 (NS)
P-105 (NS)
MW-306 (NS)
EW-05
EW-05A
EP-13 (NS)
EP-15 (NS)
P-106 (500.7)
EP-12 (NS)
EP-11 (NS)
EW-05B (1105.92)
EW-04
EP-10 (NS)
MW-308 (45.6)
EP-09 (NS)
MW-330B (ND)
MW-309B (NS)
MW-309A (NS)
MW-211A (NS)
MW-233R (NS)
EP-07 (NS)
EP-08 (NS)
MW-331 (423.7)
MW-208 (NS)
MW-216A (54.93)
MW-232A (NS)
EW-06
MW-217A (NS)
EW-07
MW-210A (NS)
MW-220 (NS)
MW-210R (NS)
MW-1303AD (25.4)
MW-1301D (61.95)
MW-218 (0.61)
MW-319 (29.3)
EP-05 (NS)
EP-06 (NS)
EP-04 (NS)
MW-225A (20.94)
EW-02A (89.4)
MW-311 (38.2)
MW-312 (NS)
MW-310 (NS)
MW-207AR (17.02)
MW-206A (NS)
MW-219 (NS)
MW-205 (30.08)
MW-105A (NS)
EP-03 (NS)
EW-01
EP-01 (NS)
EP-02 (NS)
MW-229A (15.22)
MW-313 (23.73)
MW-333 (19.2)
MW-317B (NS)
MW-317A (NS)
PZ-1 (NS)
PZ-2 (NS)
MW-335 (NS)
MW-334 (7.04)
MW-316A (NS)
MW-316B (NS)
P-123 (NS)
MW-231A (NS)
MW-230A (0.38)
MW-336 (ND)
MW-337 (1.3)
PZ-6 (NS)
MW-339 (0.94)
MW-338A (ND)
MW-315A (ND)
MW-338B (1.4)
MW-338C (ND)

Note:
1. Eastern Plume boundary is based on area where volatile organic compounds exceeded either MEG or MCL based on September 2007 sampling results. The VOC Concentration used to generate this boundary does not include: acetone, 2-butanone, methylene chloride and 1,4-dioxane.
2. Volatile organic compound concentration data obtained from September 2007 sampling results.
3. The total VOC concentrations indicated on this figure do not include the reported concentrations for acetone, 2-butanone, methylene chloride and 1,4-dioxane.
4. ND = Non-Detect
5. NS = Not Sampled

Contract No.	5700.007
Description	NAS Brunswick, ME
Coordinate system	NAD 1983, UTM, Zone 19N in meters
Sources	Naval Base Boundary provided by Navy.

Date	14-JAN-2008	Rev.	Date	App. By
DB	J. Kim			
CB	G. Calderone			
AB				

Area of Detail



Legend

- MW-315A (ND) — Deep Monitoring Well (Total Volatile Organic Compund, ug/L)
- EW-02A (57.9) — Extraction Well (Total Volatile Organic Compund, ug/L)
- P-106 (516.2) — Deep Piezometer (Total Volatile Organic Compund, ug/L)
- Approx. Location of Slurry Wall
- Approx. Limits of Sites 1 and 3
- GWETS Infiltration Gallery
- Inferred Extent of Eastern Plume

Figure 2-2

Total Volatile Organic Compound Concentration Map Deep Wells Monitoring Event 31

Naval Air Station
Brunswick, Maine

GIS Server
C:\NAVY_GIS\TO07_Brunswick\Site1and3\MapDocuments\EP_Sep07\EP_Sep07_Fig2-2_DeepVOC.mxd

0 150 300 600 Feet

Appendix C-4

February 2008 DQO Meeting Day 2 DQOs

SAP Worksheet #10 -- Problem Definition

(UFP-SAP Manual Section 2.5.2)

REMEDIATION SYSTEM DESIGN CONSIDERATIONS (DAY 2 DQOs)

DQO Step 1: State the Problem (Remediation System Design Considerations)

Background

The background and CSM are presented in Step 1 of the Day 1 DQOs. In general, there is an extensive area of 1,4-dioxane in groundwater to be addressed, which extends further than the initial sampling area. The extent of contaminated groundwater has not been identified. Additional unknown factors that affect remediation include groundwater flow paths and velocities and 1,4-dioxane concentrations across the site. In 1995, a groundwater extraction and treatment system (GWETS) was installed featuring an advanced ultraviolet (UV)/oxidation unit. As a result of a 1999 optimization study, this advanced UV/oxidation unit was replaced in 2000 with air stripping and granular activated carbon (GAC) units. In 2002, the GWETS discharge was directed to an infiltration gallery that is currently in use.

Tetra Tech, on behalf of the Navy presented the team with a preliminary list of remedial technologies that they had recently evaluated. Many potential technologies were eliminated as not feasible. The retained technologies were combined to form various remedial alternatives consisting of variations of advanced oxidation technologies (both in-situ and ex-situ) combined with the existing GWETS and with various extraction piping schemes. The team agreed that pumping with ex-situ treatment would be a viable alternative. Also, the team agreed that an extraction system transferring groundwater over an extensive area could be a disadvantage for a single-point treatment design. In-situ treatment consisting of advanced oxidation via injection was also retained. The team discussed a different type of in-situ treatment, a permeable reactive barrier, that had been eliminated during a previous evaluation; ultimately, the team agreed that this treatment was not likely to be feasible based on the potentially extensive lateral and vertical extent of contamination, lack of established reactive media for 1,4-dioxane, and hydrology/hydrogeology complexities. Based on the recent Navy/ECC investigation results, phytoremediation was identified as a potentially viable treatment technology in select areas where upwelling of groundwater in the floodplain has recently been identified.

Problem Definition

According to the interim Record of Decision (June 1992), the extraction system was to have been constructed within the plume to maximize the collection of contaminated groundwater. Wells in the southern end of the plume were to contain the plume, and wells in the northern end were to collect contaminated groundwater. At one time, the conceptual site model (CSM) indicated that the groundwater chlorinated solvent plume would migrate to Harpswell Cove; however, this CSM has since changed. Additionally, the Navy actions are still governed by the Record of Decision (ROD), which requires well placement to be determined during the design of a remediation system. In addition to chlorinated VOCs previously identified in the ROD, the emerging contaminant 1,4-dioxane, which was identified after the ROD was finalized, is now to be added as a contaminant of concern (COC).

The purpose of any project treatment system is to be protective of Mere Brook and groundwater receptors, and it should be designed to reduce 1,4-dioxane concentrations to applicable regulatory limits for surface water and groundwater, respectively. Although 1,4-dioxane is completely miscible with water and therefore difficult to purge from water, the current air stripping and GAC treatment is expected to have some limited effectiveness based on literature (although not evidenced by comparing current-day GWETS influent and effluent concentrations). The capacity of the current infiltration gallery is unknown, but the system has successfully accepted approximately 50 gallons per minute of flow. The Navy is tasked with determining the design capacity of the existing infiltration gallery to evaluate whether an expanded system is necessary for the added capacity. Furthermore, the water discharged from the GWETS needs to be controlled to meet applicable State of Maine discharge requirements.

Another potential remedial alternative utilizes advanced oxidation of the 1,4-dioxane, combined with in-situ phytoremediation in the floodplain where upwelling of groundwater occurs. A summary of the preliminary listing of remedial alternatives for future consideration is provided in Table 10.1.

Because of the 1,4-dioxane concentration exceedance (169 ug/L) of the MEG (32 ug/L) in new extraction well EW-05B, the team agreed to move as expeditiously as possible toward an effective treatment system design. Nevertheless, it was agreed that the design should be well thought out, which means that the contaminant distributions across the site should be understood sufficiently to design an effective permanent solution that successfully remediates the plume to the extent practical rather than executing a "piecemeal" temporary approach.

Overall, the team agreed that the treatment alternatives for the plume should focus on remediation of 1,4-dioxane where it exceeds 32 ug/L and should focus on protection of Mere Brook and Merriconeag Stream.

TABLE 10.1
PRELIMINARY LIST OF GROUNDWATER REMEDIAL ALTERNATIVES
EASTERN PLUME 1,4-DIOXANE CONTAMINATION
NAVAL AIR STATION BRUNSWICK, MAINE

Alternative No.		Alternative Name/Description
Alternative 1		No Action
Alternative 2		• In-Situ Treatment (Advanced Oxidation) • Phytoremediation at Floodplain • Land Use Controls (LUCs) • Monitoring
Alternative 3		• Extraction • Phytoremediation at Floodplain • Ex-Situ Treatment of 1,4-Dioxane groundwater in separate system by Advanced Oxidation (per 3A, 3B, or 3C, below) at GWETS followed by GAC Adsorption (Existing) • Disposal via existing infiltration gallery • LUCs • Monitoring
	3A	• Advanced Oxidation by UV/hydrogen peroxide using mothballed unit
	3B	• Advanced Oxidation by new UV/hydrogen peroxide unit
	3C	• Advanced Oxidation by new ozone/hydrogen unit
Alternative 4		• Extraction • Phytoremediation at Floodplain • Ex-Situ Treatment of 1,4-Dioxane groundwater combined with other Eastern Plume water by Advanced Oxidation (per 4A, 4B, or 4C, below) at GWETS followed by GAC Adsorption (Existing) • Disposal via existing infiltration gallery • LUCs • Monitoring
	4A	• Advanced Oxidation by UV/hydrogen peroxide using mothballed unit
	4B	• Advanced Oxidation by new UV/hydrogen peroxide unit
	4C	• Advanced Oxidation by new ozone/hydrogen unit
Alternative 5		• Extraction • Phytoremediation at Floodplain • Ex-Situ Treatment of 1,4-Dioxane groundwater by Advanced Oxidation (per 5A, 5B, or 5C, below) in separate system near southern end of plume • Discharge of treated water into existing extraction piping for GAC Adsorption (Existing at GWETS) • Disposal via existing infiltration gallery • LUCs • Monitoring
	5A	• Advanced Oxidation by UV/hydrogen peroxide using relocated mothballed unit
	5B	• Advanced Oxidation by new UV/hydrogen peroxide unit
	5C	• Advanced Oxidation by new ozone/hydrogen unit

Notes:

1. It was assumed that the existing UV/hydrogen peroxide treatment system could not be retrofitted with an ozone injection system because of limitations of existing control system.
2. Disposal of treated water through POTW was not evaluated further because of availability of the infiltration gallery and because of the cost associated with POTW discharge.

SAP Worksheet #11 -- Project Quality Objectives/Systematic Planning Process Statements

(UFP-SAP Manual Section 2.6.1)

REMEDIATION SYSTEM DESIGN CONSIDERATIONS
(DAY 2 DQOs)

DATA USERS AND USES:

The Navy and regulatory agencies (EPA and MEDEP) will use the data collected from this effort to Design a groundwater remediation system for reducing 1,4-dioxane and other VOC concentrations to acceptable levels. .The remediation system design will occur later under a different project. The DQO planning for Day 2 was for consideration of potential remedial alternatives.

Data will be reported, managed and archived as described throughout the remainder of this SAP. The amount of data and other pertinent Project Quality Objectives (PQOs) are described throughout the remainder of this worksheet and on other worksheets in this SAP.

Data Quality Objectives Step 2: Identify the Goals of the Study
(Remediation System Design Considerations)

Based on the conceptual model and concerns presented on Worksheet #10, the following decision statement was developed:

Decision Statement:

Determine whether 1,4-dioxane groundwater concentrations across the site are less than 32 ug/L (and incremental lifetime cancer risk is less than 1E-5 and hazard index is less than 1 from exposures to groundwater.) If this condition is satisfied, stop treatment, otherwise, take one of the following actions:

- Continue treatment
- Re-evaluate the remedy
- Redesign the treatment system to be more effective
- Establish technical impracticability
- Other actions as may be appropriate based on site-specific conditions

The team acknowledged the existence of Navy policy and guidance on groundwater treatment system optimization. They also indicated that a ROD and LTMP for NAS Brunswick are in place and that these

documents comply with State and Federal rules and regulations. These documents will govern optimization of the treatment system. The LTMP has specific requirements for determining when conditions have met the established remediation goals.

DQO Step 3: Data Quality Objectives Step 3: Identify Information Inputs (Remediation System Design Considerations)

The following data must be collected to support remedial system design. Other data needs are identified in the Day 1 DQOs (Step 3):

- Horizontal and vertical extent of 1,4-dioxane and chlorinated VOC contamination exceeding clean-up criteria via isoconcentration contouring that will include additional contours identifying each order of magnitude exceedance of clean-up criteria
- Groundwater and contaminant flow rates and paths
- Groundwater chemistry conditions (same as Day 1 DQOs) including concentrations of metals such as iron that could affect the performance of a remediation system
- Mass removal rates and time necessary to achieve clean-up goals, as established via groundwater modeling (potential models include MT3D, ModFlow, Visual ModFlow, and ModPath)
- Remediation costs

Methodologies for collecting data on these parameters will be the same as recorded in the Day 1 DQOs, including the potential to use the EPA mobile laboratory for VOC and 1,4-dioxane analyses.

DQO Step 4: Define the Boundaries of the Study (Remediation System Design Considerations)

Both short-term and long-term considerations are involved in developing a remedy to address 1,4-dioxane groundwater contamination. Long-term considerations were discussed during DQO Step 1 (see Worksheet #10). The Navy cannot transfer property until the remedy in place for the Eastern Plume is operating properly and successfully.

Short-term considerations and concerns were voiced at the meeting, resulting from the need to connect new extraction well EW-5B to the GWETS and the presence of 1,4-dioxane in groundwater in excess of the MEG at this location. As a basis of design, a flow rate of 8 to 12 gallons per minute (gpm) flow rate is expected from well EW-5B; current concentrations of 1,4-dioxane in groundwater at EW-5B may not be sustainable with pumping. The group agreed that the treatment remedy for 1,4-dioxane would have to be

different from treatment for the chlorinated VOCs present at EW-5B. An assumption was made that a short-term remedy for treatment of EW-05B extracted groundwater would help alleviate an immediate concern to start pumping at this new location while aquifer characterization work occurs concurrently.

To address the short-term concerns, the team discussed at length the potential for conducting an advanced oxidation pilot study using the "mothballed" chemical oxidation unit located at the GWETS to treat groundwater from extraction well EW-5B. The team agreed it was not likely to be a viable long-term solution because the unit is old and inefficient and because lamps and spare parts are difficult to find; moreover, any reconfiguration of the "mothballed" unit would likely result in significant changes from the original operating and design specifications. Use of the "mothballed" unit as a short-term solution is possible; however, at this time, understanding of the extent of contamination is not adequate and expediting design of a temporary system might not be effective if it does not comprehensively address the problem.

Another option to address short-term concerns would be to bring an advanced oxidation skid-mounted unit on site for treatment of groundwater from extraction well EW-5B. Additionally, having extra capacity for the unit would be helpful if the skid was to be used to address contaminated groundwater from other areas of the plume in the future. This could possibly address some of the short-term concerns while additionally providing critical design information for a permanent remedy. Discharge of the treated water from a skid-mounted unit would be directed to the existing GWETS. A bench-scale test was discussed to select the appropriate type of advanced oxidation; however, the team decided that a bench study was unnecessary. Areas to be investigated spatially were described in the Day 1 DQOs.

DQO Step 5: Develop the Analytic Approach
(Remediation System Design Considerations)

If during monitoring, the appropriate criteria are met for 1,4-dioxane in every sample from every well, for at least four consecutive sampling rounds spaced about 6 months apart (except for sentinel wells), then stop treatment; otherwise, take one of the following actions:

- Continue treatment
- Re-evaluate the remedy
- Redesign the treatment system to be more effective
- Establish technical impracticability
- Other actions as appropriate based on site-specific conditions

The team agreed that not every existing or new groundwater monitoring well would have to be monitored during every sampling round. Instead, wells to be monitored would be selected based on site conditions and the locations.

DQO Step 6: Specify Performance or Acceptance Criteria
(Remediation System Design Considerations)

Similar to the Day 1 DQOs, statistical computation of the minimum number of samples is not feasible for this problem. The team agreed that well selection and installation of new wells would follow a strategy similar to that used previously.

DQO Step 7: Develop the Plan for Obtaining Data
(Remediation System Design Considerations)

Design of the remediation system cannot occur until characterization of the aquifer is completed. The area(s) with 1,4-dioxane concentrations greater than 32 ug/L will be targeted for treatment because that will result in the greatest benefit. The team agreed that it is better to expend characterization funds than to spend those funds on short- term treatment that might not contribute to the permanent treatment system design, but to move forward expeditiously. The team agreed to proceed concurrently with performing a pump test and characterizing the aquifer. The team also agreed that it might be possible to try an interim action such as phytoremediation in the floodplain; additional research would be needed as to the viability of this approach and the selection of appropriate vegetation .

Regardless of the prioritization, the Navy will expedite the schedule as much as possible to offset some of the past delays. The MEDEP reminded the Navy that the next five-year review will occur in 2010 and that only conditional approval on the last five-year review was granted. In light of the recent 1,4-dioxane data, it is unknown whether the next five-year review report will be able to demonstrate adequate protection of human health and the environment.

Also, a planned pump test associated with recently installed extraction well EW-5B will aid future remedial design. Chemistry data for 1,4-dioxane, chlorinated VOCs, and select inorganic chemicals (e.g., pH, ferrous iron) will be obtained at the beginning, end, and one or two interim time points during the EW-5B pump test. This will allow a trend line to be developed to understand groundwater chemistry changes with pumping considering that select inorganics may affect performance of a groundwater remediation system and considering that pumping could significantly decrease 1,4-dioxane concentrations if concentrations are not sustainable.

APPENDIX D

FIELD STANDARD OPERATING PROCEDURES

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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.

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 scares and patches, and topographical depressions. Note the location of any emergency shut off switches. Any additional information regarding utility

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.

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 though conductive materials, tools, or equipment.

The following table provides the required minimum clearances for working in proximity to overhead power lines.

Nominal Voltage	Minimum Clearance
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.

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

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.

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

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

Alabama
Alabama One-Call
1-800-292-8525

Alaska
Locate Call Center of Alaska, Inc.
1-800-478-3121

Arizona
Arizona Blue Stake
1-800-782-5348

Arkansas
Arkansas One Call System, Inc.
1-800-482-8998

California
Underground Service Alert North
1-800-227-2600
Underground Service Alert of Southern California
1-800-227-2600

Colorado
Utility Notification Center of Colorado
1-800-922-1987

Connecticut
Call Before You Dig
1-800-922-4455

Delaware
Miss Utility of Delmarva
1-800-282-8555

Florida
Sunshine State One-Call of Florida, Inc.
1-800-432-4770

Georgia
Underground Protection Center, Inc.
1-800-282-7411

Hawaii
Underground Service Alert North
1-800-227-2600

Idaho
Dig Line Inc.
1-800-342-1585
Kootenai County One-Call
1-800-428-4950
Shoshone - Benewah One-Call
1-800-398-3285

Illinois
JULIE, Inc.
1-800-892-0123
Digger (Chicago Utility Alert Network)
312-744-7000

Indiana
Indiana Underground Plant Protection Service
1-800-382-5544

Iowa
Iowa One-Call
1-800-292-8989

Kansas
Kansas One-Call System, Inc.
1-800-344-7233

Kentucky
Kentucky Underground Protection Inc.
1-800-752-6007

Louisiana
Louisiana One Call System, Inc.
1-800-272-3020

Maine
Dig Safe System, Inc.
1-888-344-7233

Maryland
Miss Utility
1-800-257-7777
Miss Utility of Delmarva
1-800-282-8555

Massachusetts
Dig Safe System, Inc.
1-888-344-7233

Michigan
Miss Dig System, Inc.
1-800-482-7171

Minnesota
Gopher State One Call
1-800-252-1168

Mississippi
Mississippi One-Call System, Inc
1-800-227-6477

Missouri
Missouri One-Call System, Inc.
1-800-344-7483

Montana
Utilities Underground Protection Center
1-800-424-5555
Montana One Call Center
1-800-551-8344

Nebraska
Diggers Hotline of Nebraska
1-800-331-5666

Nevada
Underground Service Alert North
1-800-227-2600

New Hampshire
Dig Safe System, Inc.
1-888-344-7233

New Jersey
New Jersey One Call
1-800-272-1000

New Mexico
New Mexico One Call System, Inc.
1-800-321-2537
Las Cruces- Dona Ana Blue Stakes
1-888-526-0400

New York
Dig Safely New York
1-800-962-7962
New York City- Long Island One Call Center
1-800-272-4480

North Carolina
The North Carolina One-Call Center, Inc.
1-800-632-4949

North Dakota
North Dakota One-Call
1-800-795-0555

Ohio
Ohio Utilities Protection Service
1-800-362-2764
Oil & Gas Producers Underground Protect'n Svc
1-800-925-0988

Oklahoma
Call Okie
1-800-522-6543

Oregon
Oregon Utility Notification Center/One Call Concepts
1-800-332-2344

Pennsylvania
Pennsylvania One Call System, Inc.
1-800-242-1776

Rhode Island
Dig Safe System, Inc.
1-888-344-7233

South Carolina
Palmetto Utility Protection Service Inc.
1-888-721-7877

South Dakota
South Dakota One Call
1-800-781-7474

Tennessee
Tennessee One-Call System, Inc.
1-800-351-1111

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

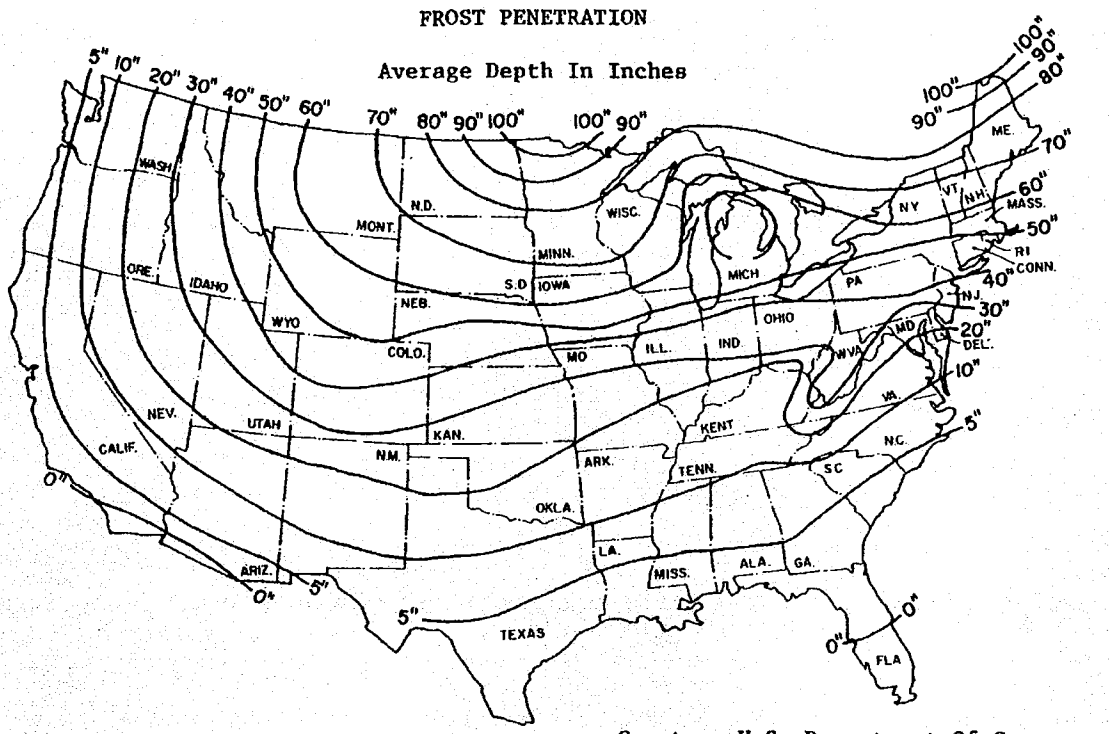
British Columbia
BC One Call
1-800-474-6886

Ontario
Ontario One-Call System
1-800-400-2255

Quebec
Info-Excavation
1-800-663-9228

ATTACHMENT 2

FROST LINE PENETRATION DEPTHS BY GEOGRAPHIC LOCATION



Courtesy U.S. Department Of Commerce

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 Name _____ Telephone: _____ Date: _____	yes no N/A
g)	State One-Call agency called? Caller: _____ Ticket Number: _____ Date: _____	yes no N/A
h)	Geophysical survey performed? Survey performed by: _____ Method: _____ Date: _____	yes no N/A
i)	Hand excavation performed (with concurrent use of utility detection device)? Completed by: _____ Total depth: _____ feet Date: _____	yes no N/A
j)	Trench/excavation probed? Probing completed by: _____ Depth/frequency: _____ Date: _____	yes no N/A

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

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:

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.

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.

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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 alpha-numeric 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

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

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

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".

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.

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1.0 PURPOSE

The purpose of this document is to specify a consistent procedure for the quality assurance review of electronic and hard copy databases. This SOP outlines the requirements for establishment of a Database Record File, Quality Assurance review procedures, and documentation of the Quality Assurance Review Process.

2.0 SCOPE

The methods described in this Standard Operating Procedure (SOP) shall be used consistently for all projects managed by Tetra Tech NUS (TtNUS).

3.0 GLOSSARY

Chain-of-Custody Form - A Chain-of-Custody Form is a printed form that accompanies a sample or a group of samples from the time of sample collection to the laboratory. The Chain-of-Custody Form is retained with the samples during transfer of samples from one custodian to another. The Chain-of-Custody Form is a controlled document that becomes part of the permanent project file. Chain-of-Custody and field documentation requirements are addressed in SOP SA-6.1.

Electronic Database - A database provided on a compact laser disk (CD). Such electronic databases will generally be prepared using public domain software such as DBase, RBase, Oracle, Visual FoxPro, Microsoft Access, Paradox, etc.

Hardcopy Database - A printed copy of a database prepared using the software discussed under the definition of an electronic database.

Form I - A printed copy of the analytical results for each sample.

Sample Tracking Summary - A printed record of sample information including the date the samples were collected, the number of samples collected, the sample matrix, the laboratory to which the samples were shipped, the associated analytical requirements for the samples, the date the analytical data were received from the laboratory, and the date that validation of the sample data was completed.

4.0 RESPONSIBILITIES

Database Records Custodian - It shall be the responsibility of the Database Records Custodian to update and file the Sample Tracking Summaries for all active projects on a weekly basis. It shall be the responsibility of the Database Records Custodian to ensure that the most recent copies of the Sample Tracking Summaries are placed in the Database Records file. It shall be the responsibility of the Database Records Custodian to ensure that a copy of all validation deliverables is provided to the Project Manager (for placement in the project file). It shall be the responsibility of the Database Records Custodian to ensure that photocopies of all validation deliverables and historical data and reports (as applicable) are placed in the Database Records file.

Data Validation Coordinator - It shall be the responsibility of the Data Validation Coordinator (or designee) to ensure that the Sample Tracking Summaries are maintained by the Database Records Custodian. It shall be the responsibility of the Data Validation Coordinator (or designee) to ensure that photocopies of all data validation deliverables are placed in the applicable Database Records file by the Database Records Custodian.

Earth Sciences Department Manager - It shall be the responsibility of the Earth Sciences Department Manager (or equivalent) to ensure that all field personnel are familiar with the requirements of this Standard Operating Procedure (specifically Section 5.5).

FOL - It shall be the responsibility of the FOL (FOL) of each project to ensure that all field technicians or sampling personnel are thoroughly familiar with this SOP, specifically regarding provision of the Chain-of-Custody Forms to the Database Records Custodian. Other responsibilities of the FOL are described in Sections 5.4 and 5.5.

Management Information Systems (MIS) Manager - It shall be the responsibility of the MIS Manager to ensure that copies of original electronic deliverables (CDs) are placed in both the project files and the Database Records File. It shall be the responsibility of the MIS Manager (or designee) to verify the completeness of the database (presence of all samples) in both electronic and hardcopy form in the Database Records File. It shall be the responsibility of the MIS Manager to ensure that Quality Assurance Reviews are completed and are attested to by Quality Assurance Reviewers. It shall be the responsibility of the MIS Manager to ensure that records of the Quality Assurance review process are placed in the Database Records File. It shall be the responsibility of the MIS Manager to ensure that both electronic and hardcopy forms of the final database are placed in both the project and the Database Record File. It shall be the responsibility of the MIS Manager to ensure that data validation qualifiers are entered in the database.

Furthermore, it shall be the responsibility of the MIS Manager to participate in project planning at the request of the Project Manager, specifically with respect to the generation of level of effort and schedule estimates. To support the project planning effort, the MIS Manager shall provide a copy of the MIS Request From included as Attachment A to the project manager. It shall be the responsibility of the MIS Manager to generate level of effort and budget estimates at the time database support is requested if a budget does not exist at the time of the request. The MIS Request Form shall be provided to the Project Manager at the time of any such requests. It shall be the responsibility of the MIS Manager to notify the Project Manager of any anticipated level of effort overruns or schedule noncompliances as soon as such problems arise along with full justification for any deviations from the budget estimates (provided they were generated by the MIS Manager). It shall be the responsibility of the MIS Manager to document any changes to the scope of work dictated by the Project Manager, along with an estimate of the impact of the change on the level of effort and the schedule.

Program/Department Managers - It shall be the responsibility of the Department and/or Program Managers (or designees) to inform their respective department's Project Managers of the existence and requirements of this SOP.

Project Manager - It shall be the responsibility of each Project Manager to determine the applicability of this SOP 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 FOL is familiar with the requirements regarding Chain-of-Custody Form provision to the Database Records Custodian. It shall be the responsibility of the Project Manager (or designee) to determine which, if any, historical data are relevant and to ensure that such data (including all relevant information such as originating entity, sample locations, sampling dates, etc.) are provided to the Database Records Custodian for inclusion in the Database Records File. It shall be the responsibility of the Project Manager to obtain project planning input regarding the level of effort and schedule from the MIS Manager. It shall be the responsibility of the Project Manager to complete the database checklist (Attachment A) to support the level of effort and schedule estimate and to facilitate database preparation and subroutine execution.

Risk Assessment Department Manager - It shall be the responsibility of the Risk Assessment Department Manager to monitor compliance with this Standard Operating Procedure, to modify this SOP as necessary, and to take corrective action if necessary. Monitoring of the process shall be completed on a quarterly basis.

Quality Assurance Reviewers - It shall be the responsibility of the Quality Assurance Reviewers to verify the completeness of the sample results via review of the Chain-of-Custody Forms and Sample Tracking Summaries. It shall be the responsibility of the Quality Assurance Reviewers to ensure the correctness of the database via direct comparison of the hardcopy printout of the database and the hardcopy summaries of the original analytical data (e.g., Form Is provided in data validation deliverables). Correctness includes the presence of all relevant sample information (all sample information fields), agreement of the laboratory and database analytical results, and the presence of data validation qualifiers.

Quality Manager - It shall be the responsibility of the Quality Manager to monitor compliance with this Standard Operating Procedure via routine audits.

5.0 PROCEDURES

5.1 Introduction

Verification of the accuracy and completeness of an electronic database can only be accomplished via comparison of a hardcopy of the database with hardcopy of all relevant sample information. The primary purposes of this SOP are to ensure that 1) all necessary hardcopy information is readily available to Quality Assurance Reviewers; 2) ensure that the Quality Assurance review is completed in a consistent and comprehensive manner, and; 3) ensure that documentation of the Quality Assurance review process is maintained in the project file.

5.2 File Establishment

A Database Record file shall be established for a specific project at the discretion of the Project Manager. Initiation of the filing procedure will commence upon receipt of the first set of Chain-of-Custody documents from a FOL or sampling technician. The Database Record Custodian shall establish a project-specific file for placement in the Database Record File. Each file in the Database Record File shall consist of standard components placed in the file as the project progresses. Each file shall be clearly labeled with the project number, which shall be placed on the front of the file drawer and on each and every hanging file folder relevant to the project. The following constitute the minimum components of a completed file:

- Electronic Deliverables
- Sample Tracking Forms
- Chain-of-Custody Forms
- Data Validation Letters
- Quality Assurance Records

5.3 Electronic Deliverables

The format of electronic deliverables shall be specified in the laboratory procurement specification and shall be provided by the laboratory. The integrity of all original electronic data deliverables shall be maintained. This shall be accomplished via the generation of copies of each electronic deliverable provided by the laboratory. The original electronic deliverable shall be provided to the project manager for inclusion in the project file. A copy of the original electronic deliverable shall be placed in the Database Record File. The second copy shall be maintained by the MIS Manager (or designee) to be used as a working copy.

5.4 Sample Tracking Forms

Updated versions of the sample tracking form for each relevant project shall be maintained by the Database Record Custodian. The Sample Tracking Forms shall be updated any time additional Chain-of-Custody Forms are received from a FOL or sampling technician, or at any time that data are received from a laboratory, or at any time that validation of a given data package (sample delivery group) is completed. The Data Validation Coordinator shall inform the Database Record Custodian of the receipt of any data packages from the laboratory and of completion of validation of a given data package to facilitate updating of the Sample Tracking Form. The Database Record Custodian shall place a revised copy of the Sample Tracking Form in the Database Record File anytime it has been updated. Copies of the updated Sample Tracking Form shall also be provided to the project manager to apprise the project manager of sample package receipt, completion of validation, etc.

5.5 Chain-of-Custody Forms

The Chain-of-Custody Forms for all sampling efforts will be used as the basis for (1) updating the Sample Tracking Form, and (2) confirming that all required samples and associated analyses have been completed. It shall be the responsibility of the FOL (or sample technician) to provide a photocopy of all Chain-of-Custody Forms to the Database Record Custodian immediately upon completion of a sampling effort. The Database Record Custodian shall then place the copies of the Chain-of-Custody Form(s) in the Database Record File. Upon receipt of a sample data package from an analytical laboratory, the Data Validation Coordinator shall provide a copy of the laboratory Chain-of-Custody Form to the Database Record Custodian. The Database Record Custodian shall use this copy to update the Sample Tracking Summary and shall place the copy of the laboratory-provided Chain-of-Custody Form in the Database Record File. The photocopy of the laboratory-provided Chain-of Custody Form shall be stapled to the previously filed field copy. Upon receipt of all analytical data, two copies of the Chain-of-Custody will therefore be in the file. Review of the Chain-of-Custody Forms will therefore be a simple mechanism to determine if all data have been received. Chain-of-Custody is addressed in SOP SA-6.1.

5.6 Data Validation Letters

All data validation deliverables (or raw data summaries if validation is not conducted) shall be provided for inclusion in both the Database Record File and the project file. If USEPA regional- or client-specific requirements are such that Form Is (or similar analytical results) need not be provided with the validation deliverable, copies of such results must be appended to the deliverable. It is preferable, although not essential that the validation qualifiers be hand-written directly on the data summary forms. The data validation deliverables (and attendant analytical summaries) will provide the basis for direct comparison of the database printout and the raw data and qualifiers.

5.7 Historical Data

At the direction of the Project Manager, historical data may also be included in a project-specific analytical database. In the event that historical data are germane to the project, hardcopy of the historical data must be included in the Database Record File. Historical data may be maintained in the form of final reports or as raw data. The information contained in the historical data file must be sufficient to identify its origin, its collection date, the sample location, the matrix, and any and all other pertinent information. All available analytical data, Chain-of-Custody Forms, boring logs, well construction logs, sample location maps, shall be photocopied by the Project Manager (or designee) and placed in one or more 3-ring binders. All information shall be organized chronologically by matrix. It shall be the responsibility of the Project Manager (or designee) to ensure that all inconsistencies between analytical data, Chain-of-Custody Forms, boring logs, sample log sheets, and field logbooks are identified and corrected. The Project Manager (or designee) shall decide which nomenclature is appropriate and edit, initial and date all relevant forms. Data entry may only be performed on information that has undergone the aforementioned

editing process, thereby having a direct correlation between hardcopy information and what will become the electronic database.

6.0 RECORDS

Records regarding database preparation and quality assurance review include all those identified in the previous section. Upon completion of the database task, records from the file will be forwarded to the Project Manager for inclusion in the project file, or will be placed in bankers boxes (or equivalent) for storage. The final records for storage shall include the following minimum information on placards placed on both the top and end of the storage box:

Database Record File
PROJECT NUMBER: ____
SITE NAME: _____
DATE FILED: __/__/__
SUMMARY OF CONTENTS ENCLOSED
BOX _ OF _

Project- or program-specific record keeping requirements shall take precedence over the record keeping requirements of this SOP.

ATTACHMENT A

MIS REQUEST FORM

Tetra Tech NUS, Inc.

Project Name:
CTO:
Project Manager:
Requestor:
Program/Client:
State/EPA Region:

Request Date:
Date Data Available for Production:
Request in Support of:
Database Lead:
GIS Lead:
Statistics Lead:
Risk Lead:

Site Name(s) (Area, OU, etc.):
Sampling Date(s):
Matrix: ☐ GW ☐ SO ☐ SD ☐ SW ☐ Other:

Labels: ☐ Labels needed for an upcoming sampling event _____ Total # of Samples
_____ Estimated Hours
_____ Due Date
_____ Complete ETS Charge No.
_____ FOL
Additional Instructions:

Data Entry:
☐ Chemical data needs to be entered from hardcopy _____ Estimated # of Samples
☐ Chemical data needs to be formated electronically
☐ Field analytical data needs to be entered from hardcopy
☐ Geologic data needs to be entered from hardcopy
☐ Hydrology data needs to be entered from hardcopy
_____ Estimated Hours
_____ Due Date
_____ Complete ETS Charge No.
Additional Instructions:

Tables:
☐ Full Data Printout
☐ Summary of Positive Hits
☐ Occurance and Distribution ☐ with criteria
☐ Sampling Analytical Summary
☐ Other:
_____ Estimated Hours
_____ Due Date
_____ Complete ETS Charge No.
Additional Instructions:

GIS:
☐ General Facility Location
☐ Site Location
☐ Potentiometric Contours/Groundwater Flow
☐ Sample Location Proposed
☐ Sample Location Existing
☐ Tag Map Single Round
☐ Tag Map Multiple Round
☐ Isoconcentrations
☐ Chart Map
☐ 3D Visualization
☐ EGIS CD
☐ Other:
_____ Estimated Hours
_____ Due Date
_____ Complete ETS Charge No.
Additional Instructions:

Statistics: ☐ Yes
_____ Estimated Hours
_____ Due Date
_____ Complete ETS Charge No.
Additional Instructions:

Geostatistics: ☐ Yes
_____ Estimated Hours
_____ Due Date
_____ Complete ETS Charge No.
Additional Instructions:

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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.

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

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.

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.

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 "plopping" or "popping" 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.

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.

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'*

ATTACHMENT B
GROUNDWATER LEVEL MESUREMENT SHEET

Tetra Tech NUS, Inc.

GROUNDWATER LEVEL MEASUREMENT SHEET

Project Name: _____ **Project No.:** _____
Location: _____ **Personnel:** _____
Weather Conditions: _____ **Measuring Device:** _____
Tidally Influenced: Yes ___ No ___ **Remarks:** _____

Well or Piezometer Number	Date	Time	Elevation of Reference Point (feet)*	Water Level Indicator Reading (feet)*	Thickness of Free Product (feet)*	Comments

* All measurements to the nearest 0.01 foot, from surveyed mark (unless noted)

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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.

Geoprobe7 - Geoprobe7 is a manufacturer of a hydraulically-powered, percussion/probing machines utilizing DPT to collect subsurface environmental samples. Geoprobe7 relies on a relatively small amount of static weight (vehicle) combined with percussion as the energy for advancement of a tool string. The Geoprobe7 equipment can be mounted in a multitude of vehicles for access to all types of environmental sites.

HydroPunch9 - HydroPunch9 is a manufacturer of stainless steel and Teflon7 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. HydroPunch9 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.

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.

- 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

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.

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 ☐ Escape Pack ☐
Half face APR ☐ SCBA ☐
SKA-PAC SAR ☐ Bottle Trailer ☐
Skid Rig ☐ 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	☒ Yes	☐ No	Hearing Protection (Plugs/Muffs)	☒ Yes	☐ No
Safety Glasses	☒ Yes	☐ No	Safety belt/harness	☐ Yes	☒ No
Chemical/splash goggles	☐ Yes	☒ No	Radio	☐ Yes	☒ No
Splash Shield	☐ Yes	☒ No	Barricades	☒ Yes	☐ No
Splash suits/coveralls	☐ Yes	☐ No	Gloves (Type - _____)	☐ Yes	☐ No
Steel toe Work shoes or boots	☒ Yes	☐ No	Work/warming regimen	☐ Yes	☐ No

Modifications/Exceptions: Reflective vests for high traffic areas.

VIII. Procedure review with permit acceptors

	Yes	NA		Yes	NA
Safety shower/eyewash (Location & Use)	☐	☒	Emergency alarms	☐	☐
Daily tail gate meetings	☒	☐	Evacuation routes	☐	☐
Contractor tools/equipment/PPE inspected	☐	☐	Assembly points	☐	☐

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

	Yes	NA
Equipment drained/depressurized	☐	☒
Equipment purged/cleaned	☐	☒
Isolation checklist completed	☐	☒
Electrical lockout required/field switch tested	☐	☒
Blinds/misalignments/blocks & bleeds in place	☐	☒
Hazardous materials on walls/behind liners considered	☐	☒

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: _____

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ATTACHMENT

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FIGURE

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1.0 PURPOSE

The purpose of this procedure is to describe the methods and equipment necessary to perform soil and rock borings and identify the equipment, sequence of events, and appropriate methods necessary to obtain soil, both surface and subsurface, and rock samples during field sampling activities.

2.0 SCOPE

This guideline addresses most of the accepted and standard drilling techniques, their benefits, and drawbacks. It should be used generally to determine what type of drilling techniques would be most successful depending on site-specific geologic conditions and the type of sampling required.

The sampling methods described within this procedure are applicable to collecting surface and subsurface soil samples, and obtaining rock core samples for lithologic and hydrogeologic evaluation, excavation/foundation design, remedial alternative design and related civil engineering purposes.

3.0 GLOSSARY

Rock Coring - A method in which a continuous solid cylindrical sample of rock or compact rock-like soil is obtained by the use of a double tube core barrel that is equipped with an appropriate diamond-studded drill bit which is advanced with a hydraulic rotary drilling machine.

Wire-Line Coring - As an alternative to conventional coring, this technique is valuable in deep hole drilling, since this method eliminates trips in and out of the hole with the coring equipment. With this technique, the core barrel becomes an integral part of the drill rod string. The drill rod serves as both a coring device and casing.

4.0 RESPONSIBILITIES

Project Manager - In consultation with the project geologist, the Project Manager is responsible for evaluating the drilling requirements for the site and specifying drilling techniques that will be successful given the study objectives and the known or suspected geologic conditions at the site. The Project Manager also determines the disposal methods for products generated by drilling, such as drill cuttings and well development water, as well as any specialized supplies or logistical support required for the drilling operations.

Field Operations Leader (FOL) - The FOL is responsible for the overall supervision and scheduling of drilling activities, and is strongly supported by the project geologist.

Project Geologist - The project geologist is responsible for ensuring that standard and approved drilling procedures are followed. The geologist will generate a detailed boring log for each test hole. This log shall include a description of materials, samples, method of sampling, blow counts, and other pertinent drilling and testing information that may be obtained during drilling (see SOPs SA-6.3 and GH-1.5). Often this position for inspecting the drilling operations may be filled by other geotechnical personnel, such as soils and foundation engineers, civil engineers, etc.

Determination of the exact location for borings is the responsibility of the site geologist. The final location for drilling must be properly documented on the boring log. The general area in which the borings are to be located will be shown on a site map included in the Work Plan and/or Sampling and Analysis Plan.

Drilling Subcontractor - Operates under the supervision of the FOL. Responsible for obtaining all drilling permits and clearances, and supplying all services (including labor), equipment and material required to perform the drilling, testing, and well installation program, as well as maintenance and quality control of such required equipment except as stated in signed and approved subcontracts.

The driller must report any major technical or analytical problems encountered in the field to the FOL within 24 hours of determination, and must provide advance written notification of any changes in field procedures, describing and justifying such changes. No such changes shall be made unless requested and authorized in writing by the FOL (with the concurrence of the Project Manager). Depending on the subcontract, the Project Manager may need to obtain written authorization from appropriate administrative personnel before approving any changes.

The drilling subcontractor is responsible for following decontamination procedures specified in the project plan documents. Upon completion of the work, the driller is responsible for demobilizing all equipment, cleaning up any materials deposited on site during drilling operations, and properly backfilling any open borings.

5.0 PROCEDURES

5.1 General

The purpose of drilling boreholes is:

- To determine the type, thickness, and certain physical and chemical properties of the soil, water and rock strata which underlie the site.
- To install monitoring wells or piezometers.

All drilling and sampling equipment will be cleaned between samples and borings using appropriate decontamination procedures as outlined in SOP SA-7.1. Unless otherwise specified, it is generally advisable to drill borings at "clean" locations first, and at the most contaminated locations last, to reduce the risk of spreading contamination between locations. All borings must be logged by the site geologist as they proceed (see SOPs SA-6.3 and GH-1.5). Situations where logging would not be required would include installation of multiple well points within a small area, or a "second attempt" boring adjacent to a boring that could not be continued through resistant material. In the latter case, the boring log can be resumed 5 feet above the depth at which the initial boring was abandoned, although the site geologist should still confirm that the stratigraphy at the redrilled location conforms essentially with that encountered at the original location. If significant differences are seen, each hole should be logged separately.

5.2 Drilling Methods

The selected drilling methods described below apply to drilling in subsurface materials, including, but not limited to, sand, gravel, clay, silt, cobbles, boulders, rock and man-made fill. Drilling methods should be selected after studying the site geology and terrain, the waste conditions at the site, and reviewing the purpose of drilling and the overall subsurface investigation program proposed for the site. The full range of different drilling methods applicable to the proposed program should be identified with final selection based on relative cost, availability, time constraints, and how well each method meets the sampling and testing requirements of the individual drilling program.

5.2.1 Continuous-Flight Hollow-Stem Auger Drilling

This method of drilling consists of rotating augers with a hollow stem into the ground. Cuttings are brought to the surface by the rotating action of the auger. This method is relatively quick and inexpensive. Advantages of this type of drilling include:

- Samples can be obtained without pulling the augers out of the hole. However, this is a poor method for obtaining grab samples from thin, discrete formations because of mixing of soils which occurs as the material is brought to the surface. Sampling of such formations requires the use of split-barrel or thin-wall tube samplers advanced through the hollow core of the auger.
- No drilling fluids are required.
- A well can be installed inside the auger stem and backfilled as the augers are withdrawn.

Disadvantages and limitations of this method of drilling include:

- Augering can only be done in unconsolidated materials.

- The inside diameter of hollow stem augers used for well installation should be at least 4 inches greater than the well casing. Use of such large-diameter hollow-stem augers is more expensive than the use of small-diameter augers in boreholes not used for well installation. Furthermore, the density of unconsolidated materials and depths become more of a limiting factor. More friction is produced with the larger diameter auger and subsequently greater torque is needed to advance the boring.

- The maximum effective depth for drilling is 150 feet or less, depending on site conditions and the size of augers used.

- In augering through clean sand formations below the water table, the sand will tend to flow into the hollow stem when the plug is removed for soil sampling or well installation. If the condition of "running" or "flowing" sands is persistent at a site, an alternative method of drilling is recommended, in particular for wells or boreholes deeper than 25 feet.

Hollow-stem auger drilling is the preferred method of drilling. Most alternative methods require the introduction of water or mud downhole (air rotary is the exception) to maintain the open borehole. With these other methods, great care must be taken to ensure that the method does not interfere with the collection of a representative sample (which may be the prime objective of the borehole construction). With this in mind, the preferred order of choice of drilling method after hollow-stem augering (HSA) is:

- Cable tool
- Casing drive (air)
- Air rotary
- Mud rotary
- Rotosonic
- Drive and wash
- Jetting

However, the use of any method will also depend on efficiency and cost-effectiveness. In many cases, mud rotary is the only feasible alternative to hollow-stem augering. Thus, mud rotary drilling is generally acceptable as a first substitute for HSA.

The procedures for sampling soils through holes drilled by hollow-stem auger shall conform with the applicable ASTM Standards: D1587-83 and D1586-84. The guidelines established in SOP SA-1.3 shall

also be followed. The hollow-stem auger may be advanced by any power-operated drilling machine having sufficient torque and ram range to rotate and force the auger to the desired depth. The machine must, however, be equipped with the accessory equipment needed to perform required sampling, or rock coring.

The hollow-stem auger may be used without the plug when boring for geotechnical examination or for well installation. However, when drilling below the water table, specially designed plugs which allow passage of formation water but not solid material shall be used (see Reference 1 of this guideline). This drilling configuration method also prevents blow back and plugging of the auger when the plug is removed for sampling.

Alternately, it may be necessary to keep the hollow stem full of water, at least to the level of the water table, to prevent blowback and plugging of the auger. If water is added to the hole, it must be sampled and analyzed to determine if it is free from contaminants prior to use. In addition, the amount of water introduced, the amount recovered upon attainment of depth, and the amount of water extracted during well development must be carefully logged in order to ensure that a representative sample of the formation water can be obtained. Well development should occur as soon after well completion as practicable (see SOP GH-2.8 for well development procedures). If gravelly or hard material is encountered which prevents advancing the auger to the desired depth, augering should be halted and either driven casing or hydraulic rotary methods should be attempted. If the depth to the bedrock/soil interface and bedrock lithology must be determined, then a 5-foot confirmatory core run should be conducted (see Section 5.2.9).

At the option of the Field Operations Leader (in communication with the Project Manager), when resistant materials prevent the advancement of the auger, a new boring can be attempted. The original boring must be properly backfilled and the new boring started a short distance away at a location determined by the site geologist. If multiple water bearing strata were encountered, the original boring must be grouted. In some formations, it may be prudent to also grout borings which penetrate only the water table aquifer, since loose soil backfill in the boring may still provide a preferred pathway for surface liquids to reach the water table. Backfilling requirements may also be driven by state or local regulations.

5.2.2 Continuous-Flight Solid-Stem Auger Drilling

This drilling method is similar to hollow-stem augering. Practical application of this method is severely restricted compared to use of hollow-stem augers. Split-barrel (split-spoon) sampling cannot be performed without pulling the augers out, which may allow the hole to collapse. The continuous-flight solid-stem auger drilling method is therefore very time consuming and is not cost effective. Also, augers would have to be withdrawn before installing a monitoring well, which again, may allow the hole to collapse. Furthermore, geologic logging by examining the soils brought to the surface is unreliable, and depth to water may be difficult to determine while drilling.

There would be very few situations where use of a solid-stem auger would be preferable to other drilling methods. The only practical applications of this method would be to drill boreholes for well installation where no lithologic information is desired and the soils are such that the borehole can be expected to remain open after the augers are withdrawn. Alternatively, this technique can be used to find depth to bedrock in an area when no other information is required from drilling.

5.2.3 Rotary Drilling

Direct rotary drilling includes air-rotary and fluid-rotary drilling. For air or fluid-rotary drilling, the rotary drill may be advanced to the desired depth by any power-operated drilling machine having sufficient torque

and ram range to rotate and force the bit to the desired depth. The drilling machine must, however, be equipped with any accessory equipment needed to perform required sampling, or coring. Prior to sampling, any settled drill cuttings in the borehole must be removed.

Air-rotary drilling is a method of drilling where the drill rig simultaneously turns and exerts a downward pressure on the drilling rods and bit while circulating compressed air down the inside of the drill rods, around the bit, and out the annulus of the borehole. Air circulation serves to both cool the bit and remove the cuttings from the borehole. Advantages of this method include:

- The drilling rate is high (even in rock).
- The cost per foot of drilling is relatively low.
- Air-rotary rigs are common in most areas.
- No drilling fluid is required (except when water is injected to keep down dust).
- The borehole diameter is large, to allow room for proper well installation procedures.

Disadvantages to using this method include:

- Formations must be logged from the cuttings that are blown to the surface and thus the depths of materials logged are approximate.
- Air blown into the formation during drilling may "bind" the formation and impede well development and natural groundwater flow.
- In-situ samples cannot be taken, unless the hole is cased.
- Casing must generally be used in unconsolidated materials.
- Air-rotary drill rigs are large and heavy.
- Large amounts of Investigation Derived Waste (IDW) may be generated which may require containerization, sampling, and off-site disposal.

A variation of the typical air-rotary drill bit is a down hole hammer which hammers the drill bit down as it drills. This makes drilling in hard rock faster. Air-rotary drills can also be adapted to use for rock coring although they are generally slower than other types of core drills. A major application of the air-rotary drilling method would be to drill holes in rock for well installation.

Fluid-Rotary drilling operates in a similar manner to air-rotary drilling except that a drilling fluid ("mud") or clean water is used in place of air to cool the drill bit and remove cuttings. There are a variety of fluids that can be used with this drilling method, including bentonite slurry and synthetic slurries. If a drilling fluid other than water/cuttings is used, it must be a natural clay (i.e., bentonite) and a "background" sample of the fluid should be taken for analysis of possible organic or inorganic contaminants.

Advantages to the fluid-rotary drilling method include:

- The ability to drill in many types of formations.
- Relatively quick and inexpensive.
- Split-barrel (split-spoon) or thin-wall (Shelby) tube samples can be obtained without removing drill rods if the appropriate size drill rods and bits (i.e., fish-tail or drag bit) are used.

- In some borings temporary casing may not be needed as the drilling fluids may keep the borehole open.
- Drill rigs are readily available in most areas.

Disadvantages to this method include:

- Formation logging is not as accurate as with hollow-stem auger method if split-barrel (split-spoon) samples are not taken (i.e., the depths of materials logged from cuttings delivered to the surface are approximate).
- Drilling fluids reduce permeability of the formation adjacent to the boring to some degree, and require more extensive well development than "dry" techniques (augering, air-rotary).
- No information on depth to water is obtainable while drilling.
- Fluids are needed for drilling, and there is some question about the effects of the drilling fluids on subsequent water samples obtained. For this reason as well, extensive well development may be required.
- In very porous materials (i.e., rubble fill, boulders, coarse gravel) drilling fluids may be continuously lost into the formation. This requires either constant replenishment of the drilling fluid, or the use of casing through this formation.
- Drill rigs are large and heavy, and must be supported with supplied water.
- Groundwater samples can be potentially diluted with drilling fluid.

The procedures for performing direct rotary soil investigations and sampling shall conform with the applicable ASTM standards: D2113-83, D1587-83, and D1586-84.

Soil samples shall be taken as specified by project plan documents, or more frequently, if requested by the project geologist. Any required sampling shall be performed by rotation, pressing, or driving in accordance with the standard or approved method governing use of the particular sampling tool.

When field conditions prevent the advancement of the hole to the desired depth, a new boring may be drilled at the request of the Field Operations Leader. The original boring shall be backfilled using methods and materials appropriate for the given site and a new boring started a short distance away at a location determined by the project geologist.

5.2.4 Rotosonic Drilling

The Rotosonic drilling method employs a high frequency vibrational and low speed rotational motion coupled with down pressure to advance the cutting edge of a drill string. This produces a uniform borehole while providing a continuous, undisturbed core sample of both unconsolidated and most bedrock formations. Rotosonic drilling advances a 4-inch diameter to 12-inch diameter core barrel for sampling and can advance up to a 12-inch diameter outer casing for the construction of standard and telescoped monitoring wells. During drilling, the core barrel is advanced ahead of the outer barrel in increments as determined by the site geologist and depending upon type of material, degree of subsurface contamination and sampling objectives.

The outer casing can be advanced at the same time as the inner drill string and core barrel, or advanced down over the inner drill rods and core barrel, or after the core barrel has moved ahead to collect the undisturbed sample and has been pulled out of the borehole. The outer casing can be advanced dry in most cases, or can be advanced with water or air depending upon the formations being drilled, the depth and diameter of the hole, or requirements of the project.

Advantages of this method include:

- Sampling and well installation are faster as compared to other drilling methods.
- Continuous sampling, with larger sample volume as compared to split-spoon sampling.
- The ability to drill through difficult formations such as cobbles or boulders, hard till and bedrock.
- Reduction of IDW by an average of 70 to 80 percent.
- Well installations are quick and controlled by elimination of potential bridging of annular materials during well installation, due to the ability to vibrate the outer casing during removal.

Disadvantages include:

- The cost for Rotosonic drilling as compared to other methods are generally higher. However, the net result can be a significant savings considering reduced IDW and shortened project duration.
- Rotosonic drill rigs are large and need ample room to drill, however, Rotosonic units can be placed on the ground or placed on an ATV.
- There are a limited number of Rotosonic drilling contractors at the present time.

5.2.5 Reverse Circulation Rotary Drilling

The common reverse-circulation rig is a water or mud-rotary rig with a large-diameter drill pipe which circulates the drilling water down the annulus and up the inside of the drill pipe (reverse flow direction from direct mud-rotary). This type of rig is used for the construction of large-capacity production water wells and is not suited for small, water quality sampling wells because of the use of drilling muds and the large-diameter hole which is created. A few special reverse-circulation rotary rigs are made with double-wall drill pipe. The drilling water or air is circulated down the annulus between the drill pipes and up inside the inner pipe.

Advantages of the latter method include:

- The formation water is not contaminated by the drilling water.
- Formation samples can be obtained, from known depths.
- When drilling with air, immediate information is available regarding the water-bearing properties of formations penetrated.
- Collapsing of the hole in unconsolidated formations is not as great a problem as when drilling with the normal air-rotary rig.

Disadvantages include:

- Double-wall, reverse-circulation drill rigs are rare and expensive to operate.
- Placing cement grout around the outside of the well casing above a well screen often is difficult, especially when the screen and casing are placed down through the inner drill pipe before the drill pipe is pulled out.

5.2.6 Drill-through Casing Driver

The driven-casing method consists of alternately driving casing (fitted with a sharp, hardened casing shoe) into the ground using a hammer lifted and dropped by the drill rig (or an air-hammer) and cleaning out the casing using a rotary chopping bit and air or water to flush out the materials. The casing is driven down in stages (usually 5 feet per stage); a continuous record is kept of the blows per foot in driving the casing (see SOP GH-1.5). The casing is normally advanced by a 300-pound hammer falling freely through a height of 30 inches. Simultaneous washing and driving of the casing is not recommended. If this procedure is used, the elevations within which wash water is used and in which the casing is driven must be clearly recorded.

The driven casing method is used in unconsolidated formations only. When the boring is to be used for later well installation, the driven casing used should be at least 4 inches larger in diameter than the well casing to be installed. Advantages to this method of drilling include:

- Split-barrel (split-spoon) sampling can be conducted while drilling.
- Well installation is easily accomplished.
- Drill rigs used are relatively small and mobile.
- The use of casing minimizes flow into the hole from upper water-bearing layers; therefore, multiple aquifers can be penetrated and sampled for rough field determinations of some water quality parameters.

Some of the disadvantages include:

- This method can only be used in unconsolidated formations.
- The method is slower than other methods (average drilling progress is 30 to 50 feet per day).
- Maximum depth of the borehole varies with the size of the drill rig and casing diameter used, and the nature of the formations drilled.
- The cost per hour or per foot of drilling may be substantially higher than other drilling methods.
- It is difficult and time consuming to pull back the casing if it has been driven very deep (deeper than 50 feet in many formations).

5.2.7 Cable Tool Drilling

A cable tool rig uses a heavy, solid-steel, chisel-type drill bit ("tool") suspended on a steel cable, which when raised and dropped, chisels or pounds a hole through the soils and rock. Drilling progress may be

expedited by the use of "slip-jars" which serve as a cable-activated down hole percussion device to hammer the bit ahead.

When drilling through the unsaturated zone, some water must be added to the hole. The cuttings are suspended in the water and then bailed out periodically. Below the water table, after sufficient ground water enters the borehole to replace the water removed by bailing, no further water needs to be added. When soft caving formations are encountered, it is usually necessary to drive casing as the hole is advanced to prevent collapse of the hole. Often the drilling can be only a few feet below the bottom of the casing. Because the drill bit is lowered through the casing, the hole created by the bit is smaller than the casing. Therefore, the casing (with a sharp, hardened casing shoe on the bottom) must be driven into the hole (see Section 5.2.5 of this guideline).

Advantages of the cable-tool method include the following:

- Information regarding water-bearing zones is readily available during the drilling. Even relative permeabilities and rough water quality data from different zones penetrated can be obtained by skilled operators.
- The cable-tool rig can operate satisfactorily in all formations, but is best suited for caving, boulder, cobble or coarse gravel type formations (e.g., glacial till) or formations with large cavities above the water table (such as limestones).
- When casing is used, the casing seals formation water out of the hole, preventing down hole contamination and allowing sampling of deeper aquifers for field-measurable water quality parameters.
- Split-barrel (split-spoon) or thin-wall (Shelby) tube samples can be collected through the casing.

Disadvantages include:

- Drilling is slow compared with rotary rigs.
- The necessity of driving the casing in unconsolidated formations requires that the casing be pulled back if exposure of selected water-bearing zones is desired. This process complicates the well completion process and often increases costs. There is also a chance that the casing may become stuck in the hole.
- The relatively large diameters required (minimum of 4-inch casing) plus the cost of steel casing result in higher costs compared to rotary drilling methods where casing is not required (e.g., such use of a hollow-stem auger).
- Cable-tool rigs have largely been replaced by rotary rigs. In some parts of the U.S., availability may be difficult.

5.2.8 Jet Drilling (Washing)

Jet drilling, which should be used only for piezometer or vadose zone sampler installation, consists of pumping water or drilling mud down through a small diameter (1/2- to 2-inch) standard pipe (steel or PVC). The pipe may be fitted with a chisel bit or a special jetting screen. Formation materials dislodged by the bit and jetting action of the water are brought to the surface through the annulus around the pipe. As the pipe is jetted deeper, additional lengths of pipe may be added at the surface.

Jet percussion is a variation of the jetting method, in which the casing is driven with a drive weight. Normally, this method is used to place 2-inch-diameter casing in shallow, unconsolidated sand formations, but this method has also been used to install 3- to 4-inch-diameter casings to a depth of 200 feet.

Jetting is acceptable in very soft formations, usually for shallow sampling, and when introduction of drilling water to the formation is acceptable. Such conditions would occur during rough stratigraphic investigation or installation of piezometers for water level measurement. Advantages of this method include:

- Jetting is fast and inexpensive.
- Because of the small amount of equipment required, jetting can be accomplished in locations where access by a normal drilling rig would be very difficult. For example, it would be possible to jet down a well point in the center of a lagoon at a fraction of the cost of using a drill rig.
- Jetting numerous well points just into a shallow water table is an inexpensive method for determining the water table contours, hence flow direction.

Disadvantages include the following:

- A large amount of foreign water or drilling mud is introduced above and into the formation to be sampled.
- Jetting is usually done in very soft formations which are subject to caving. Because of this caving, it is often not possible to place a grout seal above the screen to assure that water in the well is only from the screened interval.
- The diameter of the casing is usually limited to 2 inches.
- Jetting is only possible in very soft formations that do not contain boulders or coarse gravel, and the depth limitation is shallow (about 30 feet without jet percussion equipment).
- Large quantities of water are often needed.

5.2.9 Drilling with a Hand Auger

This method is applicable wherever the formation, total depth of sampling, and the site and groundwater conditions are such as to allow hand auger drilling. Hand augering can also be considered at locations where drill rig access is not possible. All hand auger borings will be performed according to ASTM D1452-80.

Samples should be taken continuously unless otherwise specified by the project plan documents. Any required sampling is performed by rotation, pressing, or driving in accordance with the standard or approved method governing use of the particular sampling tool. Typical equipment used for sampling and advancing shallow "hand auger" holes are Iwan samplers (which are rotated) or post hole diggers (which are operated like tongs). These techniques are slow but effective where larger pieces of equipment do not have access, and where very shallow holes are desired (less than 15 feet). Surficial soils must be composed of relatively soft and non-cemented formations to allow penetration by the auger.

5.2.10 Rock Drilling and Coring

When soil borings cannot be continued using augers or rotary methods due to the hardness of the soil or when rock or large boulders are encountered, drilling and sampling can be performed using a diamond bit corer in accordance with ASTM D2113.

Drilling is done by rotating and applying downward pressure to the drill rods and drill bit. The drill bit is a circular, hollow, diamond-studded bit attached to the outer core barrel in a double-tube core barrel. The use of single-tube core barrels is not recommended, as the rotation of the barrel erodes the sample and limits its use for detailed geological evaluation. Water or air is circulated down through the drill rods and annular space between the core barrel tubes to cool the bit and remove the cuttings. The bit cuts a core out of the rock which rises into an inner barrel mounted inside the outer barrel. The inner core barrel and rock core are removed by lowering a wire line with a coupling into the drill rods, latching onto the inner barrel and withdrawing the inner barrel. A less efficient variation of this method utilizes a core barrel that cannot be removed without pulling all of the drill rods. This variation is practical only if less than 50 feet of core is required.

Core borings are made through the casing used for the soil borings. The casing must be driven and sealed into the rock formation to prevent seepage from the overburden into the hole to be cored (see Section 5.3 of this guideline). A double-tube core barrel with a diamond bit and reaming shell or equivalent should be used to recover rock cores of a size specified in the project plans. The most common core barrel diameters are listed in Attachment A.

Soft or decomposed rock should be sampled with a driven split-barrel whenever possible or cored with a Denison or Pitcher sampler.

When coring rock, including shale and claystone, the speed of the drill and the drilling pressure, amount and pressure of water, and length of run can be varied to give the maximum recovery from the rock being drilled. Should any rock formation be so soft or broken that the pieces continually fall into the hole causing unsatisfactory coring, the hole should be reamed and a flush-joint casing installed to a point below the broken formation. The size of the flush-joint casing must permit securing the core size specified. When soft or broken rock is anticipated, the length of core runs should be reduced to less than 5 feet to avoid core loss and minimize core disturbance.

Advantages of core drilling include:

- Undisturbed rock cores can be recovered for examination and/or testing.
- In formations in which the cored hole will remain open without casing, water from the rock fractures may be recovered from the well without the installation of a well screen and gravel pack.
- Formation logging is extremely accurate.
- Drill rigs are relatively small and mobile.

Disadvantages include:

- Water or air is needed for drilling.
- Coring is slower than rotary drilling (and more expensive).
- Depth to water cannot accurately be determined if water is used for drilling.
- The size of the borehole is limited.

This drilling method is useful if accurate determinations of rock lithology are desired or if open wells are to be installed into bedrock. To install larger diameter wells in coreholes, the hole must be reamed out to the proper size after boring, using air or mud rotary drilling methods.

5.2.11 Drilling & Support Vehicles

In addition to the drilling method required to accomplish the objectives of the field program, the type of vehicle carrying the drill rig and/or support equipment and its suitability for the site terrain, will often be an additional deciding factor in planning the drilling program. The types of vehicles available are extensive, and depend upon the particular drilling subcontractor's fleet. Most large drilling subcontractors will have a wide variety of vehicle and drill types suited for most drilling assignments in their particular region, while smaller drilling subcontractors will usually have a fleet of much more limited diversity. The weight, size, and means of locomotion (tires, tracks, etc.) of the drill rig must be selected to be compatible with the site terrain to assure adequate mobility between borehole locations. Such considerations also apply to necessary support vehicles used to transport water and/or drilling materials to the drill rigs at the borehole locations. When the drill rigs or support vehicles do not have adequate mobility to easily traverse the site, provisions must be made for assisting equipment, such as bulldozers, winches, timber planking, etc., to maintain adequate progress during the drilling program.

Some of the typical vehicles which are usually available for drill rigs and support equipment are:

- Totally portable drilling/sampling equipment, where all necessary components (tripods, samplers, hammers, catheads, etc.) may be hand carried to the borehole site. Drilling/sampling methods used with such equipment include:

  - Hand augers and lightweight motorized augers.
  - Retractable plug samplers--driven by hand (hammer).
  - Motorized cathead - a lightweight aluminum tripod with a small gas-engine cathead mounted on one leg, used to install small-diameter cased borings. This rig is sometimes called a "monkey on a stick."

- Skid-mounted drilling equipment containing a rotary drill or engine-driven cathead (to lift hammers and drill string), a pump, and a dismounted tripod. The skid is pushed, dragged, or winched (using the cathead drum) between boring locations.

- Small truck-mounted drilling equipment using a Jeep, stake body or other light truck (4 to 6 wheels), upon which are mounted the drill and/or a cathead, a pump, and a tripod or small drilling derrick. On some rigs, the drill and/or a cathead are driven by a power take-off from the truck, instead of by a separate engine.

- Track-mounted drilling equipment is similar to truck-mounted rigs, except that the vehicle used has wide bulldozer tracks for traversing soft ground. Sometimes a continuous-track "all terrain vehicle" is also modified for this purpose. Some types of tracked drill rigs are called "bombardier" or "weasel" rigs.

- Heavy truck-mounted drilling equipment is mounted on tandem or dual tandem trucks to transport the drill, derrick, winches, and pumps or compressors. The drill may be provided with a separate engine or may use a power take-off from the truck engine. Large augers, hydraulic rotary and reverse circulation rotary drilling equipment are usually mounted on such heavy duty trucks. For soft-ground sites, the drilling equipment is sometimes mounted on vehicles having low pressure, very wide diameter tires and capable of floating; these vehicles are called "swamp buggy" rigs.

- Marine drilling equipment is mounted on various floating equipment for drilling borings in lakes, estuaries and other bodies of water. The floating equipment varies, and is often manufactured or customized by the drilling subcontractor to suit specific drilling requirements. Typically, the range of flotation vehicles include:

  - Barrel-float rigs - a drill rig mounted on a timber platform buoyed by empty 55-gallon drums or similar flotation units.

  - Barge-mounted drill rigs.

  - Jack-up platforms - drilling equipment mounted on a floating platform having retractable legs to support the unit on the sea or lake bed when the platform is jacked up out of the water.

  - Drill ships - for deep ocean drilling.

In addition to the mobility for the drilling equipment, similar consideration must be given for equipment to support the drilling operations. Such vehicles or floating equipment are needed to transport drill water, drilling supplies and equipment, samples, drilling personnel, etc. to and/or from various boring locations.

5.2.12 Equipment Sizes

In planning subsurface exploration programs, care must be taken in specifying the various drilling components, so that they will fit properly in the boring or well.

For drilling open boreholes using rotary drilling equipment, tri-cone drill bits are employed with air, water or drilling mud to remove cuttings and cool the bit. Tri-cone bits are slightly smaller than the holes they drill (i.e., 5-7/8-inch or 7-7/8-inch bits will nominally drill 6-inch and 8-inch holes, respectively).

For obtaining split-barrel samples of a formation, samplers are commonly manufactured in sizes ranging from 2 inches to 3-1/2 inches in outside diameter. However, the most commonly used size is the 2-inch O.D., 1-3/8-inch I.D. split-barrel sampler. When this sampler is used and driven by a 140-pound (± 2-pound) hammer dropping 30 inches (± 1 inch), the procedure is called a Standard Penetration Test, and the blows per foot required to advance the sampler into the formation can be correlated to the formation's density or strength.

In planning the drilling of boreholes using hollow-stem augers or casing, in which thin-wall tube samples or diamond core drilling will be performed, refer to the various sizes and clearances provided in Attachment A of this guideline. Sizes selected must be stated in the project plan documents.

5.2.13 Estimated Drilling Progress

To estimate the anticipated rates of drilling progress for a site, the following must be considered:

- The speed of the drilling method employed.

- Applicable site conditions (e.g., terrain, mobility between borings, difficult drilling conditions in bouldery soils, rubble fill or broken rock, etc.).

- Project-imposed restrictions (e.g., drilling while wearing personal protective equipment, decontamination of drilling equipment, etc.).

Based on recent experience in drilling average soil conditions (no boulders) and taking samples at 5-foot intervals, for moderate depth (30 feet to 50 feet) boreholes (not including installation or development of wells), the following daily rates of total drilling progress may be anticipated for the following drilling methods:

Drilling Method	Average Daily Progress (linear feet)
Hollow-stem augers	75'
Solid-stem augers	50'
Mud-Rotary Drilling	100' (cuttings samples)
Rotosonic Drilling	100'-160' (continuous core)
Reverse-Circulation Rotary	100' (cuttings samples)
Skid-Rig with driven casing	30'
Rotary with driven casing	50'
Cable Tool	30'
Hand Auger	Varies
Continuous Rock Coring	50'

5.3 Prevention of Cross-Contamination

A telescoping or multiple casing technique minimizes the potential for the migration of contaminated groundwater to lower strata below a confining layer. The telescoping technique consists of drilling to a confining layer utilizing a spun casing method with a diamond cutting or augering shoe (a method similar to the rock coring method described in Section 5.2.10, except that larger casing is used) or by using a driven-casing method (see Section 5.2.6 of this guideline) and installing a specified diameter steel well casing. The operation consists of three separate steps. Initially, a drilling casing (usually of 8-inch diameter) is installed followed by installation of the well casing (6-inch-diameter is common for 2-inch wells). This well casing is driven into the confining layer to ensure a tight seal at the bottom of the hole. The well casing is sealed at the bottom with a bentonite-cement slurry. The remaining depth of the boring is drilled utilizing a narrower diameter spun or driven casing technique within the outer well casing. A smaller diameter well casing with an appropriate length of slotted screen on the lower end, is installed to the surface.

Clean sand is placed in the annulus around and to a point of about 2 feet above the screen prior to withdrawal of the drilling casing. The annular space above the screen and to a point 2 feet above the bottom of the outer well casing is sealed with a tremied cement-bentonite slurry which is pressure-grouted or displacement-grouted into the hole. The remaining casing annulus is backfilled with clean material and grouted at the surface, or it is grouted all the way to the surface.

5.4 Cleanout of Casing Prior to Sampling

The boring hole must be completely cleaned of disturbed soil, segregated coarse material and clay adhering to the inside walls of the casing. The cleaning must extend to the bottom edge of the casing and, if possible, a short distance further (1 or 2 inches) to bypass disturbed soil resulting from the advancement of the casing. Loss of wash water during cleaning should be recorded.

For disturbed samples both above and below the water table and where introduction of relatively large volumes of wash water is permissible, the cleaning operation is usually performed by washing the material out of the casing with water; however, the cleaning should never be accomplished with a strong, downward-directed jet which will disturb the underlying soil. When clean out has reached the bottom of the casing or slightly below (as specified above), the string of tools should be lifted one foot off the bottom with the water still flowing, until the wash water coming out of the casing is clear of granular soil particles. In formations where the cuttings contain gravel and other larger particles, it is often useful to repeatedly raise and lower the drill rods and wash bit while washing out the hole, to surge these large particles upward out of the hole. As a time saver, the drilling contractor may be permitted to use a split-barrel (split-spoon) sampler with the ball check valve removed as the clean-out tool, provided the material below the spoon is not disturbed and the shoe of the spoon is not damaged. However, because the ball check valve has been removed, in some formations it may be necessary to install a flap valve or spring sample retainer in the split-spoon bit, to prevent the sample from falling out as the sampler is withdrawn from the hole. The use of jet-type chopping bits is discouraged except where large boulders and cobbles or hard-cemented soils are encountered. If water markedly softens the soils above the water table, clean out should be performed dry with an auger.

For undisturbed samples below the water table, or where wash water must be minimized, clean out is usually accomplished with an appropriate diameter clean out auger. This auger has cutting blades at the bottom to carry loose material up into the auger, and up-turned water jets just above the cutting blades to carry the removed soil to the surface. In this manner, there is a minimum of disturbance at the top of the material to be sampled. If any gravel material washes down into the casing and cannot be removed by the clean out auger, a split-barrel sample can be taken to remove it; bailers and sandpumps should not be used. For undisturbed samples above the groundwater table, all operations must be performed in a dry manner.

If all of the cuttings created by drilling through the overlying formations are not cleaned from the borehole prior to sampling, some of the problems which may be encountered during sampling include:

- When sampling is attempted through the cuttings remaining in the borehole, all or part of the sampler may become filled with the cuttings. This limits the amount of sample from the underlying formation which can enter and be retained in the sampler, and also raises questions as to the validity of the sample.

- If the cuttings remaining in the borehole contain coarse gravel and/or other large particles, these may block the bit of the sampler and prevent any materials from the underlying formation from entering the sampler when the sampler is advanced.

- In cased borings, should sampling be attempted through cuttings which remain in the lower portion of the casing, these cuttings could cause the sampler to become bound into the casing, such that it becomes very difficult to either advance or retract the sampler.

- When sampler blow counts are used to estimate the density or strength of the formation being sampled, the presence of cuttings in the borehole will usually give erroneously high sample blow counts.

To confirm that all cuttings have been removed from the borehole prior to attempting sampling, it is important that the site geologist measure the "stickup" of the drill string. This is accomplished by measuring the assembled length of all drill rods and bits or samplers (the drill string) as they are lowered to the bottom of the hole, below some convenient reference point of the drill string, then measuring the height of this reference point above the ground surface. The difference of these measurements is the

depth of the drill string (lower end of the bit or sampler) below the ground surface, which must then be compared with the depth of sampling required (installed depth of casing or depth of borehole drilled). If the length of drill string below grade is more than the drilled or casing depth, the borehole has been cleaned too deeply, and this deeper depth of sampling must be recorded on the log. If the length of drill string below grade is less than the drilled or casing depth, the difference represents the thickness of cuttings which remain in the borehole. In most cases, an inch or two of cuttings may be left in the borehole with little or no problem. However, if more than a few inches of cuttings are encountered, the borehole must be recleaned prior to attempting sampling.

5.5 Materials of Construction

The effects of monitoring well construction materials on specific chemical analytical parameters are described and/or referenced in SOP GH-2.8. However, there are several materials used during drilling, particularly drilling fluids and lubricants, which must be used with care to avoid compromising the representativeness of soil and ground water samples.

The use of synthetic or organic polymer slurries is not permitted at any location where soil samples for chemical analysis are to be collected. These slurry materials could be used for installation of long-term monitoring wells, but the early time data in time series collection of ground water data may then be suspect. If synthetic or organic polymer muds are proposed for use at a given site, a complete written justification including methods and procedures for their use must be provided by the site geologist and approved by the Project Manager. The specific slurry composition and the concentration of suspected contaminants for each site must be known.

For many drilling operations, potable water is an adequate lubricant for drill stem and drilling tool connections. However, there are instances, such as drilling in tight clayey formations or in loose gravels, when threaded couplings must be lubricated to avoid binding. In these instances, to be determined in the field by the judgment of the site geologist and noted in the site logbook, and only after approval by the Project Manager, a vegetable oil or silicone-based lubricant should be used. Petroleum based greases, etc. will not be permitted. Samples of lubricants used must be provided and analyzed for chemical parameters appropriate to the given site.

5.6 Subsurface Soil Samples

Subsurface soil samples are used to characterize subsurface stratigraphy. This characterization can indicate the potential for migration of chemical contaminants in the subsurface. In addition, definition of the actual migration of contaminants can be obtained through chemical analysis of the soil samples. Where the remedial activities may include in-situ treatment or excavation and removal of the contaminated soil, the depth and areal extent of contamination must be known as accurately as possible.

Engineering and physical properties of soil may also be of interest should site construction activities be planned. Soil types, grain size distribution, shear strength, compressibility, permeability, plasticity, unit weight, and moisture content are some of the physical characteristics that may be determined for soil samples.

Penetration tests are also described in this procedure. The tests can be used to estimate various physical and engineering parameters such as relative density, unconfined compressive strength, and consolidation characteristics of soils.

Surface protocols for various soil sampling techniques are discussed in SOP SA-1.3. Continuous-core soil sampling and rock coring are discussed below. The procedures described here are representative of

a larger number of possible drilling and sampling techniques. The choice of techniques is based on a large number of variables such as cost, local geology, etc. The final choice of methods must be made with the assistance of drilling subcontractors familiar with the local geologic conditions. Alternative techniques must be based upon the underlying principles of quality assurance implicit in the following procedures.

The CME continuous sample tube system provides a method of sampling soil continuously during hollow-stem augering. The 5-foot sample barrel fits within the lead auger of a hollow-auger column. The sampling system can be used with a wide range of I.D. hollow-stem augers (from 3-1/4-inch to 8-1/4-inch I.D.). This method has been used to sample many different materials such as glacial drift, hard clays and shales, mine tailings, etc. This method is particularly used when SPT samples are not required and a large volume of material is needed. Also, this method is useful when a visual description of the subsurface lithology is required. Rotosonic drilling methods also provide a continuous soil sample.

5.7 Rock Sampling (Coring) (ASTM D2113-83)

Rock coring enables a detailed assessment of borehole conditions to be made, showing precisely all lithologic changes and characteristics. Because coring is an expensive drilling method, it is commonly used for shallow studies of 500 feet or less, or for specific intervals in the drill hole that require detailed logging and/or analyzing. Rock coring can, however, proceed for thousands of feet continuously, depending on the size of the drill rig, and yields better quality data than air-rotary drilling, although at a substantially reduced drilling rate. Rate of drilling varies widely, depending on the characteristics of lithologies encountered, drilling methods, depth of drilling, and condition of drilling equipment. Average output in a 10-hour day ranges from 40 to over 200 feet. Down hole geophysical logging or television camera monitoring is sometimes used to complement the data generated by coring.

Borehole diameter can be drilled to various sizes, depending on the information needed. Standard sizes of core barrels (showing core diameter) and casing are shown in Figure 1.

Core drilling is used when formations are too hard to be sampled by soil sampling methods and a continuous solid sample is desired. Usually, soil samples are used for overburden, and coring begins in sound bedrock. Casing is set into bedrock before coring begins to prevent loose material from entering the borehole, to prevent loss of drilling fluid, and to prevent cross-contamination of aquifers.

Drilling through bedrock is initiated by using a diamond-tipped core bit threaded to a drill rod (outer core barrel) with a rate of drilling determined by the downward pressure, rotation speed of drill rods, drilling fluid pressure in the borehole, and the characteristics of the rock (mineralogy, cementation, weathering).

FIGURE 1

STANDARD SIZES OF CORE BARRELS AND CASING

Coring Bit Size	Nominal*		Set Size*	
	O.D.	I.D.	O.D.	I.D.
RWT	1 5/32	3/4	1.160	0.735
EWT	1 1/2	29/32	1.470	0.905
EX, EXL, EWG, EWM	1 1/2	13/16	1.470	0.845
AWT	1 7/8	1 9/32	1.875	1.281
AX, AXL, AWG, AWM	1 7/8	1 3/16	1.875	1.185
BWT	2 3/8	1 3/4	2.345	1.750
BX, BXL, BWG, BWM	2 3/8	1 5/8	2.345	1.655
NWT	3	2 5/16	2.965	2.313
NX, NXL, NWG, NWM	3	2 1/8	2.965	2.155
HWT	3 29/32	3 3/16	3.889	3.187
HWG	3 29/32	3	3.889	3.000
2 3/4 x 3 7/8	3 7/8	2 3/4	3.840	2.690
4 x 5 1/2	5 1/2	4	5.435	3.970
6 x 7 3/4	7 3/4	6	7.655	5.970
AX Wire line ___|___/	1 7/8	1	1.875	1.000
BX Wire line ___|___/	2 3/8	1 7/16	2.345	1.437
NX Wire line ___|___/	3	1 15/16	2.965	1.937

* All dimensions are in inches; to convert to millimeters, multiply by 25.4.

___|___/ Wire line dimensions and designations may vary according to manufacturer.

FIGURE 1
STANDARD SIZES OF CORE BARRELS AND CASING
PAGE TWO

Size Designations		Casing O.D., Inches	Casing Coupling		Casing bit O.D., Inches	Core barrel bit O.D., Inches*	Drill rod O.D., Inches	Approximate Core Diameter	
Casing; Casing coupling; Casing bits; Core barrel bits	Rod; rod couplings		O.D., Inches	I.D., Inches				Normal, Inches	Thinwall, Inches
RX	RW	1.437	1.437	1.188	1.485	1.160	1.094	—	0.735
EX	E	1.812	1.812	1.500	1.875	1.470	1.313	0.845	0.905
AX	A	2.250	2.250	1.906	2.345	1.875	1.625	1.185	1.281
BX	B	2.875	2.875	2.375	2.965	2.345	1.906	1.655	1.750
NX	N	3.500	3.500	3.000	3.615	2.965	2.375	2.155	2.313
HX	HW	4.500	4.500	3.938	4.625	3.890	3.500	3.000	3.187
RW	RW	1.437	Flush Joint	No Coupling	1.485	1.160	1.094	—	0.735
EW	EW	1.812			1.875	1.470	1.375	0.845	0.905
AW	AW	2.250			2.345	1.875	1.750	1.185	1.281
BW	BW	2.875			2.965	2.345	2.125	1.655	1.750
NW	NW	3.500			3.615	2.965	2.625	2.155	2.313
HW	HW	4.500			4.625	3.890	3.500	3.000	3.187
PW	—	5.500			5.650	—	—	—	—
SW	—	6.625			6.790	—	—	—	—
UW	—	7.625			7.800	—	—	—	—
ZW	—	8.625			8.810	—	—	—	—
—	AX __|__\	—	—	—	—	1.875	1.750	1.000	—
—	BX __|__\	—	—	—	—	2.345	2.250	1.437	—
—	NX __|__\	—	—	—	—	2.965	2.813	1.937	—

* All dimensions are in inches; to convert to millimeters, multiply by 25.4.

__|__/ Wire line dimensions and designations may vary according to manufacturer.

NOMINAL DIMENSIONS FOR DRILL CASINGS AND ACCESSORIES. (DIAMOND CORE DRILL MANUFACTURERS ASSOCIATION). 288-D-2889

5.7.1 Diamond Core Drilling

A penetration of typically less than 6 inches per 50 blows using a 140-lb. hammer dropping 30 inches with a 2-inch split-barrel sampler shall be considered an indication that soil sampling methods may not be applicable and that coring may be necessary to obtain samples.

When formations are encountered that are too hard to be sampled by soil sampling methods, the following diamond core drilling procedure may be used:

- Firmly seat a casing into the bedrock or the hard material to prevent loose materials from entering the hole and to prevent the loss of drilling fluid return. Level the surface of the rock or hard material when necessary by the use of a fishtail or other bits. If the drill hole can be retained open without the casing and if cross-contamination of aquifers in the unconsolidated materials is unlikely, leveling may be omitted.

- Begin the core drilling using a double-tube swivel-core barrel of the desired size. After drilling no more than 10 feet (3 m), remove the core barrel from the hole and take out the core. If the core blocks the flow of the drilling fluid during drilling, remove the core barrel immediately. In soft materials, a large starting size may be specified for the coring tools; where local experience indicates satisfactory core recovery or where hard, sound materials are anticipated, a smaller size or the single-tube type may be specified and longer runs may be drilled. NX/NW size coring equipment is the most commonly used size.

- When soft materials are encountered that produce less than 50 percent recovery, stop the core drilling. If soil samples are desired, secure such samples in accordance with the procedures described in ASTM Method D 1586 (Split-barrel Sampling) or in Method D 1587 (Thin-Walled Tube Sampling); sample soils per SOP SA-1.3. Resume diamond core drilling when refusal materials are again encountered.

- Since rock structures and the occurrence of seams, fissures, cavities, and broken areas are among the most important items to be detected and described, take special care to obtain and record these features. If such broken zones or cavities prevent further advance of the boring, one of the following three steps shall be taken: (1) cement the hole; (2) ream and case; or (3) case and advance with the next smaller size core barrel, as conditions warrant.

- In soft, seamy, or otherwise unsound rock, where core recovery may be difficult, M-design core barrels may be used. In hard, sound rock where a high percentage of core recovery is anticipated, the single-tube core barrel may be employed.

5.7.2 Rock Sample Preparation and Documentation

Once the rock coring has been completed and the core recovered, the rock core shall be carefully removed from the barrel, placed in a core tray (previously labeled "top" and "bottom" to avoid confusion), classified, and measured for percentage of recovery as well as the rock quality designation (RQD). Each core shall be described, classified, and logged using a uniform system as presented in SOP GH-1.5. If moisture content will be determined or if it is desirable to prevent drying (e.g., to prevent shrinkage of clay formations) or oxidation of the core, the core shall be wrapped in plastic sleeves immediately after logging. Each plastic sleeve shall be labeled with indelible ink. The boring number, run number, and the footage represented in each sleeve shall be included, as well as designating the top and bottom of the core run.

After sampling, rock cores shall be placed in the sequence of recovery in well-constructed wooden boxes provided by the drilling contractor. Rock cores from two different borings shall not be placed in the same core box unless accepted by the Project Geologist. The core boxes shall be constructed to accommodate at least 20 linear feet of core in rows of approximately 5 feet each and shall be constructed with hinged tops secured with screws, and a latch (usually a hook and eye) to keep the top securely fastened down. Wood partitions shall be placed at the end of each core run and between rows.

The depth from the surface of the boring to the top and bottom of the drill run and run number shall be marked on the wooden partitions with indelible ink. A wooden partition (wooden block) shall be placed at the end of each run with the depth of the bottom of the run written on the block. These blocks will serve to separate successive core runs and indicate depth intervals for each run. The order of placing cores shall be the same in all core boxes. Rock core shall be placed in the box so that, when the box is open, with the inside of the lid facing the observer, the top of the cored interval contained within the box is in the upper left corner of the box, and the bottom of the cored interval is in the lower right corner of the box. The top and bottom of each core obtained and its true depth shall be clearly and permanently marked on each box. The width of each row must be compatible with the core diameter to prevent lateral movement of the core in the box. Similarly, an empty space in a row shall be filled with an appropriate filler material or spacers to prevent longitudinal movement of the core in the box.

The inside and outside of the core-box lid shall be marked by indelible ink to show all pertinent data on the box's contents. At a minimum, the following information shall be included:

- Project name.
- Project number.
- Boring number.
- Run numbers.
- Footage (depths).
- Recovery.
- RQD (%).
- Box number and total number of boxes for that boring (Example: Box 5 of 7).

For easy retrieval when core boxes are stacked, the sides and ends of the box shall also be labeled and include project number, boring number, top and bottom depths of core and box number.

Prior to final closing of the core box, a photograph of the recovered core and the labeling on the inside cover shall be taken. If moisture content is not critical, the core shall be wetted and wiped clean for the photograph. (This will help to show true colors and bedding features in the cores).

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ATTACHMENT A

DRILLING EQUIPMENT SIZES

Drilling Component	Designation or Hole Size (Inches)	O.D. (Inches)	I.D. (Inches)	Coupling I.D. (Inches)
Hollow-stem augers (Ref. 7)	6 1/4	5	2 1/4	
	6 3/4	5 3/4	2 3/4	---
	7 1/4	6 1/4	3 1/4	---
	13 1/4	12	6	---
Thin Wall Tube Samplers (Ref. 7)	---	2	1 7/8	---
	---	2 1/2	2 3/8	---
	---	3	2 7/8	---
	---	3 1/2	3 3/8	---
	---	4 1/2	4 3/8	---
	---	5	4 3/4	---
Drill Rods (Ref. 7)	RW	1 3/32	23/32	13/32
	EW	1 3/8	15/16	7/16
	AW	1 3/4	1 1/4	5/8
	BW	2 1/8	1 3/4	3/4
	NW	2 5/8	2 1/4	1 3/8
	HW	3 1/2	3 1/16	2 3/8
	E	1 5/16	7/8	7/16
	A	1 5/8	1 1/8	9/16
	B	1 7/8	1 1/4	5/8
	N	2 3/8	2	1
				Wall Thickness (Inches)
Driven External Coupled Extra Strong Steel* Casing (Ref. 8)	2 1/2	2.875	2.323	0.276
	3	3.5	2.9	0.300
	3 1/2	4.0	3.364	0.318
	4	4.5	3.826	0.337
	5	5.63	4.813	0.375
	6	6.625	5.761	0.432
	8	8.625	7.625	0.500
	10	10.750	9.750	0.500
	12	12.750	11.750	0.500

* Add twice the casing wall thickness to casing O.D. to obtain the approximate O.D. of the external pipe couplings.

ATTACHMENT A
DRILLING EQUIPMENT SIZES
PAGE TWO

Drilling Component	Designation or Hole Size (Inches)	O.D. (Inches)	I.D. (Inches)	Coupling I.D. (Inches)
Flush Coupled Casing (Ref. 7)	RX	1 7/16	1 3/16	1 3/16
	EX	1 13/16	1 5/8	1 1/2
	AX	2 1/4	2	1 29/32
	BX	2 7/8	2 9/16	2 3/8
	NX	3 1/2	3 3/16	3
	HX	4 1/2	4 1/8	3 15/16
Flush Joint Casing (Ref. 7)	RW	1 7/16	1 3/16	
	EW	1 13/16	1 1/2	
	AW	2 1/4	1 29/32	
	BW	2 7/8	2 3/8	
	NW	3 1/2	3	
	HW	4 1/2	4	
	PW	5 1/2	5	
	SW	6 5/8	6	
	UW	7 5/8	7	
	ZW	8 5/8	8	
Diamond Core Barrels (Ref. 7)	EWM	1 1/2	7/8**	
	AWM	1 7/8	1 1/8**	
	BWM	2 3/8	1 5/8**	
	NWM	3	2 1/8	
	HWG	3 7/8	3	
	2 3/4 x 3 7/8	3 7/8	2 11/16	
	4 x 5 1/2	5 1/2	3 15/16	
	6 x 7 3/4	7 3/4	5 15/16	
	AQ (wireline)	1 57/64	1 1/16**	
	BQ (wireline)	2 23/64	1 7/16**	
	NQ (wireline)	2 63/64	1 7/8	
	HQ (wireline)	3 25/32	2 1/2	

** Because of the fragile nature of the core and the difficulty to identify rock details, use of small-diameter core (1 3/8") is not recommended.

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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
BORING LOG (EXAMPLE)

BORING LOG

Page ___ of ___

PROJECT NAME: _____ BORING NUMBER: _____
PROJECT NUMBER: _____ DATE: _____
DRILLING COMPANY: _____ GEOLOGIST: _____
DRILLING RIG: _____ DRILLER: _____

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: Soil Density/ Consistency or Rock Hardness	MATERIAL DESCRIPTION: Color	MATERIAL DESCRIPTION: Material Classification	USCS*	Remarks	PID/FID Reading (ppm): Sample	PID/FID Reading (ppm): Sampler BZ	PID/FID Reading (ppm): Borehole**	PID/FID Reading (ppm): Driller BZ**

* When rock coring, enter rock brokeness.

** Include monitor reading in 6 foot intervals @ borehole. Increase reading frequency if elevated response-read.

Remarks: _____

Drilling Area Background (ppm): _____

Converted to Well: Yes _____ No _____ Well I.D. #: _____

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 Particles Larger Than 3 Inches and Basing Fractions on Estimated Weights)			GROUP SYMBOL	TYPICAL NAMES	FIELD IDENTIFICATION PROCEDURES (Excluding Particles Larger Than 3 Inches and Basing Fractions on Estimated Weights)				GROUP SYMBOL	TYPICAL NAMES
					Identification Procedures on Fraction Smaller Than No. 40 Sieve Size					
						DRY STRENGTH (Crushing Characteristics)	DILATANCY (Reaction to Shaking)	TOUGHNESS (Consistency Near Plastic Limit)		
GRAVELS (More than 50% of Coarse Fraction Retained on No. 4 Sieve)	CLEAN GRAVELS (Less Than 5% Fines)	Wide range in grain size and substantial amounts of all intermediate particle sizes.	GW	Well graded gravels, gravel-sand mixtures, little or no fines.	SILTS AND CLAYS Liquid Limit Less Than 50	None to Slight	Quick to Slow	None	ML	Inorganic silts and very fine sands, rock flour, silty or clayey fine sands with slight plasticity.
		Predominantly one size or a range of sizes with some intermediate sizes missing.	GP	Poorly graded gravels, gravel-sand mixtures, little 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.
	GRAVELS WITH FINES (More Than 12% Fines)	Non-plastic fines (for identification procedures, see ML)	GM	Silty gravels, poorly graded gravel-sand-silt mixtures.		Slight to Medium	Slow	Slight	OL	Organic silts and organic silt-clays of low plasticity.
		Plastic fines (for identification procedures, see CL)	GC	Clayey gravels, poorly graded gravel-sand-clay mixtures.	SILTS AND CLAYS Liquid Limit Greater Than 50	Slight to Medium	Slow to None	Slight to Medium	MH	Inorganic silts, micaceous or diatomaceous fine sandy or silty soils, elastic silts.
SANDS (More Than 50% of Coarse Fraction Passes No. 4 Sieve)	CLEAN SANDS (Less Than 5% Fines)	Wide range in grain size and substantial amounts of all intermediate particle sizes.	SW	Well graded sand, gravelly sands, little or no fines.		High to Very High	None	High	CH	Inorganic clays of high plasticity, fat clays.
		Predominantly one size or a range of sizes with some intermediate sizes missing.	SP	Poorly graded sands, gravelly sands, little or no fines.		Medium to High	None to Very Slow	Slight to Medium	OH	Organic clays of medium to high plasticity.
	SANDS WITH FINES (More Than 12% Fines)	Non-plastic fines (for identification procedures, see ML)	SM	Silty sands, poorly graded sand-silt mixtures.	HIGHLY ORGANIC SOILS	Readily identified by color, odor, spongy feel and frequently by fibrous texture.			Pt	Peat and other organic soils
		Plastic fines (for identification procedures, see CL)	SC	Clayey sands, poorly graded sand-clay mixtures.						

Boundary classifications: 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 sieve sizes on this chart are U.S. Standard.

DENSITY OF GRANULAR SOILS

DESIGNATION	STANDARD PENETRATION RESISTANCE - BLOWS/FOOT
Very Loose	0-4
Loose	5-9
Medium Loose	10-24
Dense	25-49
Very Dense	Over 50

CONSISTENCY OF COHESIVE SOILS

CONSISTENCY	UNCONFINED COMPRESSIVE STRENGTH (TONS/SQ. FT.)	STANDARD PENETRATION RESISTANCE - BLOWS/FOOT	FIELD IDENTIFICATION METHODS
Very Soft	Less Than 0.25	0 to 2	Easily penetrated several inches by fist.
Soft	0.25 to 0.50	2 to 4	Easily penetrated several inches by thumb.
Medium Stiff	0.50 to 1.0	4 to 8	Can be penetrated several inches by thumb.
Stiff	1.0 to 2.0	8 to 15	Readily indented by thumb.
Very Stiff	2.0 to 4.0	15 to 30	Readily indented by thumbnail.
Hard	More Than 4.0	Over 30	Indented with difficulty by thumbnail.

ROCK TERMS

ROCK HARDNESS (FROM CORE SAMPLES)

Descriptive Terms	Scratch or Knife Effects	Hammer Effects
Soft	Easily Gouged	Crushes when pressed with hammer
Medium Soft	Can be Gouged	Breaks (one blow); crumbly edges
Medium Hard	Can be scratched	Breaks (one blow); sharp edges
Hard	Cannot be scratched	Breaks considerably (several blows); sharp edges

ROCK BROKENNESS

Descriptive Terms	Abbreviation	Spacing
Very Broken	(V. Br.)	0-2"
Broken	(Br.)	2"-1'
Blocky	(Bl.)	1'-3'
Massive	(Ma.)	3'-10'

LEGEND :

SOIL SAMPLES - TYPES

1-4" Split Barrel Sample

3.25" O.D. Undisturbed Sample

O - Other Samples, Specify in Remarks

ROCK SAMPLES - TYPES

X-NX (Conventional) Core (2.1-1.5" O.D.)

O-NQ (Diamond) Core (2.1-1.5" O.D.)

Z - Other Core Sizes, Specify in Remarks

LINE LEVELS

W.L. Initial Level w/Date & Depth

W.L. Stabilized Level w/Date & Depth

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 inchΦ-1/2 inchΦ)" 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

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)

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

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 works "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

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.

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.

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

Page 1 of 1

PROJECT NAME: NSB- SITE
PROJECT NUMBER: 9594
DRILLING COMPANY: SOILTEST CO.
DRILLING RIG: CME-55
BORING NUMBER: SB/MW 1
DATE: 3/8/96
GEOLOGIST: SJ CONTI
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: Soil Density/ Consistency or Rock Hardness	Color	Material Classification	USCS*	Remarks	PID/FID Reading (ppm): Sample	Sampler BZ	Borehole**	Driller BZ**
S-1 @ 0800	0.0	7/6	1.5/2.0		M DENSE	BRN TO BLK	SILTY SAND - SOME	SM	MOIST SL. ORG. ODOR	5	0	0	0
	2.0	9/10					ROCK FR.: TR BRICKS (FILL)		FILL TO 4'±				
	4.0			4.0									
S-2 @ 0810		5/7	2.0/2.0		M DENSE	BRN	SILTY SAND - TR FINE GRAVEL	SM	MOIST - W ODOR	10	0	–	–
	6.0	9/8							NAT. MATL. TOOK SAMPLE SB01-0406 FOR ANALYSIS				
	•			7'± ▽									
	8.0			8.0									
S-3 @ 0820		6/8	1.9/2.0		DENSE	TAN BRN	FINE TO COARSE SAND	SW	WET	0	0	0	0
	10.0	17/16					TR. F. GRAVEL		HIT WATER: 7'±				
	12.0			12.0									
S-4 @ 0830		7/6	1.6/2.0		STIFF	GRAY	SILTY CLAY	CL	MOIST → WET	0	.5	–	–
	14.0	5/8											
	15.0			15.0					AUGER REF @ 15'				
				LIKELIHOOD 16	M HARD	BRN	SILTSTONE	VBR	WEATHERED				
									LO ∡JNTS @ 15.5				
9/5	①		4.0/5.0						WATER STAINS @ 16.5, 17.1, 17.5	0	0	0	0
				19'					LOSING SOME				
	20.0				HARD	GRAY	SANDSTONE - SOME SILTSTONE	BR	DRILL H2O @ 17'±				
									SET TEMP 6" CAS TO 15.5				
4.9/5.0	②		5.0/5.0						SET 2"Φ PVC SCREEN 16-25	0	0	0	0
									SAND 14-25				
	25.0			25					PELLETS 12-14				

* When rock coring, enter rock brokeness.
** Include monitor reading in 6 foot intervals @ borehole. Increase reading frequency if elevated response read.

• 1-20% 1-80%

Drilling Area Background (ppm): 0

Remarks: CME-55 R16, 4¼" ID HSA - 9" OD±
2" SPLIT SPOONS - 140 LB HAMMER - 30" DROP
NX CORE IN BEDROCK RUN ① = 25 min, RUN ② = 15 min

Converted to Well: Yes ✓ No _____ Well I.D. #: MW-1

- 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).

- 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.

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.

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A HYDRAULIC CONDUCTIVITY TESTING DATA SHEET 7

1.0 PURPOSE

This guideline is intended to describe procedures for performing in-situ hydraulic conductivity testing (slug testing) in boreholes and monitoring wells, and provide a short description of commonly used evaluation techniques for the data generated. Slug tests are used to provide data regarding the hydraulic properties of the formation tested. A variation of the slug test, called a constant-head test, is also briefly described.

2.0 SCOPE

Slug tests are short-term tests designed to provide approximate hydraulic conductivity values for the portion of a formation immediately surrounding the screened/open interval of a well or boring. These tests are much less accurate than pumping tests, as a much more localized area is involved. Therefore, a number of slug tests are typically performed and averaged to determine a representative hydraulic conductivity value for the formation tested. Performance of slug tests may be preferable to pumping tests in situations where handling of large volumes of contaminated water is a concern or when time/budget constraints preclude the more expensive and time-consuming setup and performance of a pumping test.

Constant-head tests also are used to determine hydraulic conductivity values and are similar to slug tests with regard to the quality of data obtained and time/cost considerations. A disadvantage of constant-head tests is that a significant volume of water may be added to high-permeability formations, potentially affecting short-term water quality.

3.0 GLOSSARY

Hydraulic Conductivity (K) - A quantitative measure of the ability of a porous material to transmit water, defined as the volume of water that will flow through a unit cross-sectional area of porous material per unit time under a head gradient of 1. Hydraulic conductivity is dependent upon properties of the medium and fluid. Common units of expression include centimeters per second (cm/sec), feet per day (ft/day), and gallons per day per foot^2 (gpd/ft^2).

Transmissivity (T) - A quantitative measure of the ability of an aquifer to transmit water. The product of the hydraulic conductivity times the saturated thickness.

Slug Test - A rising head or falling head test used to measure hydraulic conductivity. A slug test consists of instantaneously changing the water level within a well and measuring the rate of recovery of the water level to equilibrium conditions. Slug tests are performed by either withdrawing a slug of water (rising head test) or adding a slug of water (falling head test), then measuring recovery over time. A solid slug of known volume can be used to displace a volume of water, thereby simulating the addition or removal of water.

4.0 RESPONSIBILITIES

Project Hydrogeologist - The project hydrogeologist, in conjunction with the Project Manager, shall evaluate the type(s) and extent of hydraulic testing required for a given project during the planning process, and design the field program accordingly. The project hydrogeologist also shall ensure that field personnel have the necessary training and guidance to properly perform the tests, and shall oversee data reduction activities, including selecting the appropriate evaluation techniques and checking calculations for accuracy.

Field Geologist - The field geologist is responsible for performing the planned field tests as specified in the project planning documents, (or approved modifications thereto). The field geologist also generally assists in the data evaluation process. The field geologist shall be knowledgeable in the testing methodologies used and is responsible for obtaining the necessary support equipment required to perform the field tests. All applicable data regarding testing procedures, equipment used, well construction, and geologic/hydrogeologic conditions shall be recorded by the field geologist. The field geologist shall be familiar enough with testing procedures/requirements to be able to recommend changes in methodology, should unanticipated field conditions be encountered.

5.0 PROCEDURES

5.1 In-situ Hydraulic Conductivity Testing in Wells

Slug tests are commonly performed in completed wells. Prior to testing, the well shall be thoroughly developed and allowed to stabilize, in order to obtain accurate results. Once the water level within the well has stabilized at its static level, it shall be quickly raised or lowered and the rate of recovery measured.

One of the basic assumptions of slug testing is that the initial change in water level is instantaneous; therefore, an effort shall be made to minimize the time involved in raising or lowering the water level initially. Various methods can be used to induce instantaneous (or nearly instantaneous) changes in water level within the well. A rise in water levels can be induced by pouring water into the well. A solid slug of known volume, quickly lowered below the water level within the well, will displace an equivalent volume of water and raise the water level within the well. The slug can be left in place until the water level restabilizes at the static water level, then suddenly removed to create a drop in water level within the well. An advantage of using a solid cylinder of known volume (slug) to change the water level is that no water is removed or added to the monitoring well. This eliminates the need to dispose of contaminated water and/or add water to the system. A bailer or pump can be used to withdraw water from the well. If a pump is used, pumping shall not continue for more than several seconds so that a cone of depression is not created which would adversely impact testing results. The pump hose shall also be removed from the well during the recovery period, as data analysis techniques involve volume of recovery versus time, and leaving the hose within the well would distort the calculated testing results by altering the apparent volume of recovery. Falling head slug tests should only be performed in wells with fully submerged screens, while rising head slug tests can be performed in wells with either partially or fully submerged screens/open intervals.

Other methods that can be used to change water levels within a well include creating a vacuum or a high pressure environment within the well. The vacuum method will raise water levels within the well, while the pressure method will depress the water level in the well. These methods are particularly useful in highly permeable formations where other methods are ineffective in creating measurable changes in water levels. Both of these methods are limited to wells which have completely submerged screens.

Rate of recovery measurements shall be obtained from time zero (maximum change in water level) until water level recovery exceeds 90 percent of the initial change in water level. In low permeability formations, the test may be cut-off short of 90 percent recovery due to time constraints. Time intervals between water level readings will vary according to the rate of recovery of the well. For a moderately fast recovering well, water level readings at 0, 0.1, 0.2, 0.3, 0.4, 0.5, 0.75, 1.0, 1.25, 1.5, 2.0, 2.5, 3.0, 4.0, . . . minutes may be required. With practice, readings at down to 0.05-minute (3 seconds) time intervals can be obtained with reasonable accuracy, using a pressure transducer and hand held readout. For wells which recover very fast, a pressure transducer and data logger may be required to obtain representative data. Time intervals between measurements can be extended for slow recovering wells. A typical

schedule for measurements for a slow recovering well would be 0, 0.25, 0.5, 0.75, 1.0, 1.5, 2.0, 3.0, 4.0, 6.0, 8.0, 10.0, 15.0, 20.0, 30.0, . . . minutes from the beginning the test. Measurements shall be taken from the top of the well casing.

Water level measurements can be obtained using an electric water level indicator, popper, or pressure transducer. Steel tape coated with chalk or water sensitive paste although very accurate, is a slower method of obtaining water levels and is generally not recommended for use due to the frequency at which water level measurements need to be obtained during the performance of a slug test.

Time/recovery should be field-plotted on semilog graph paper to determine the data quality. The data set should plot along a sloped, straight line. If excessive data scatter is observed, the test should be rerun until acceptable results are obtained.

The following data shall be recorded when performing slug tests in wells or borings:

- Well/boring ID number
- Total depth of well/boring
- Screened/open interval depth and length
- Gravel pack interval depth and length
- Well stickup above ground surface
- Gravel pack radius
- Static water level
- Aquifer thickness
- Depth to confining layer
- Time/recovery data

A variation of the slug test, called a constant-head test, is a test in which water is added to the well at a measured rate sufficient to maintain the water level in the well at a constant height above the static water level. Once a stable elevated water level has been achieved, discharge (pumping) rate measurements are recorded in place of time/recovery data for approximately 10 to 20 minutes. The hydraulic conductivity is then calculated from this information. The constant-head test is generally not recommended for monitoring wells as large volumes of water may be introduced into the screened formation, potentially impacting later sampling events.

5.2 In-situ Hydraulic Conductivity Testing in Borings

Slug tests can be performed in borings while the boring is being advanced. This permits testing of formations at different depths throughout the drilling process. Boreholes to be tested shall be drilled using casing, so that discrete depths may be investigated. Various tests and testing methods are described below. The most appropriate test and testing method to be used in a situation varies and shall be selected after a careful evaluation of drilling, geologic, and general site conditions.

Rising head or falling head slug tests can be performed in saturated and unsaturated formations during drilling. There are two ways that the tests can be performed. One way entails setting the casing flush with the bottom of the boring when the desired testing depth has been reached. The hole is then cleaned out to remove loose materials, the drill bit and rods are carefully withdrawn from the boring, and a few feet of sand (of higher permeability than the surrounding formation) is added to the bottom of the boring. After the water level in the boring has stabilized (for saturated formations), the static water level is measured and recorded. The water level is then raised (falling head test) or lowered (rising head test) and the change in water level is measured at time intervals determined by the field hydrogeologist. Only falling head tests can be performed for depth intervals within the unsaturated (vadose) zone. As described for

wells, time intervals for water level measurements will vary according to the formation's hydraulic conductivity. The faster the rate of recovery expected, the shorter the time intervals between measurements shall be. The rate of change of water level will be used to calculate hydraulic conductivity. The test is to be conducted until the water level again stabilizes, or for a minimum of 20 minutes. In low permeability formations, it is not always practical to run the test until the water level stabilizes, as it may take a long time to do so. The top of the casing shall be used as the reference point for all water level measurements.

The second method for conducting a slug test during drilling consists of placing a temporary well with a short screen into the cleaned-out boring, pulling the drilling casing back to expose the screen, allowing the formation to collapse around the screen (or placing a sand/gravel pack around the screen), and performing the appropriate hydraulic conductivity test in the well, as described for the first method. Again, the test shall be conducted until the water level stabilizes or for a minimum of 20 minutes. This method allows for testing a larger section of the formation and results in more reliable hydraulic conductivity estimates.

Constant-head tests may also be performed in borings. As described for monitoring wells, once a stable elevated level has been achieved, the discharge rate into the boring is measured for a period of time, usually 10 to 20 minutes, and the hydraulic conductivity is calculated from this. This method is the most accurate method depicted in this section, and shall be given preference over others if the materials are available to perform the test and the addition of water to the boring does not adversely impact project objectives. Once the test is over, additional information can be gathered by measuring the rate of the drop in water level in the boring (for saturated formations). A limitation of the constant-head test is that foreign water is introduced into the formation which must be removed from the well area by natural or artificial means, before a representative groundwater sample can be obtained.

Detailed descriptions regarding the performance of borehole hydraulic conductivity tests and subsequent data analysis techniques are provided in Ground Water Manual (1981).

5.3 Data Analysis

There are a number of data analysis methods available to reduce and evaluate slug testing data. The determination of which method is most appropriate shall be made based on the testing conditions (including physical setup of the well/boring tested, hydrogeologic conditions, and testing methodology) and the limitations of each test analysis method. Well construction details, aquifer type (confined or unconfined), and screened/open interval (fully or partially penetrating the aquifer) shall be taken into account in selecting an analysis method. Cooper, et al. (1967), and Papadapulos, et al. (1973) have developed test interpretation procedures for fully penetrating wells in confined aquifers. Hvorslev (1951) developed a relatively simple analytical procedure for point piezometers in an infinite isotropic medium. In Cedergren (1967), Hvorslev presents a number of analytical procedures which cover a wide variety of hydrogeologic conditions, testing procedures, and well/boring/piezometer configurations. Bouwer and Rice (1976) developed an analytical technique applicable to both unconfined and confined conditions, which factors in partial/full penetration and discusses well screen gravel pack considerations. The Ground Water Manual (1981) presents a number of testing and test analysis procedures for wells and borings open above or below the water table, and for both falling head and constant-head tests. The methods described above do not represent a complete listing of test analysis methods available, but are some of the more commonly used and accepted methods. Other methods can be used, at the discretion of the project hydrogeologist and in concurrence with the Project Manager and client.

One consideration to be noted during data analysis is the determination of the screened/open interval of a tested well. If a well with a fully submerged screen is installed in a relatively low permeability formation,

and a gravel pack which is significantly more permeable is installed around the screen, the length of the gravel pack (if longer than the screened interval) should be used as the screened/open length, rather than the screen length itself. In situations where the formation permeability is judged to be comparable to the gravel pack permeability (within about an order of magnitude) this adjustment is not required.

All data analysis applications and calculations shall be reviewed by technical personnel thoroughly familiar with testing and test analysis procedures. Upon approval of the calculations and results, the calculation sheets shall be initialed and dated by the reviewer. Distribution copies shall be supplied to appropriate project personnel and the original copy stored in the project central file.

6.0 REFERENCES

Cedergren, H. R., 1967. Seepage, Drainage, and Flow Nets. John Wiley and Sons Inc., New York, pp. 78-76.

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Hvorslev, M. J., 1951. Time Lag and Soil Permeability in Ground Water Observations. U.S. Army Corps of Engineers, Waterways Experiment Station, Washington, D.C., Bull. No. 36.

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Bouwer, H. and R. C. Rice, 1976. "A Slug Test for Determining Hydraulic Conductivity of Unconfined Aquifers with Completely or Partially Penetrating Wells." Water Resources Research, 12:423-28.

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7.0 RECORDS

Field data shall be recorded on the data sheet included as Attachment A (or equivalent). 1 Any notes regarding testing procedures, problems encountered, and general observations not included on the data sheet shall be noted in the bound site logbook or field notebook. The boring log and well construction diagrams for each well/boring tested shall be used as references during testing and data analysis activities. Original data sheets shall be placed in the project file, along with the logbook/notebook.

1 If an automated data recorder is used, the data may be displayed using the printer output from the unit. Such printouts should be annoted to include the relevant data form, or attached to the form shown as Attachment A.

ATTACHMENT A
EXAMPLE HYDRAULIC CONDUCTIVITY TESTING DATA SHEET

HYDRAULIC CONDUCTIVITY TESTING DATA SHEET

PROJECT NAME: ..... WELL/BORING NO.: .....

PROJECT NO.: ..... GEOLOGIST: .....

WELL DIAMETER: ..... SCREEN LENGTH/DEPTH: ..... TEST NO.: .....

STATIC WATER LEVEL (Depth/Elevation): ..... DATE: .....

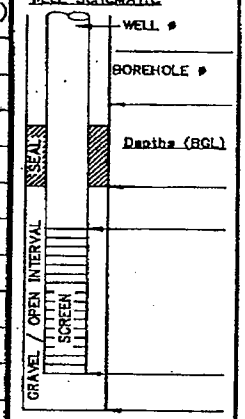
TEST TYPE (Rising/Falling/Constant Head): ..... CHECKED: .....

METHOD OF INDUCING WATER LEVEL CHANGE: ..... PAGE ..... OF .....

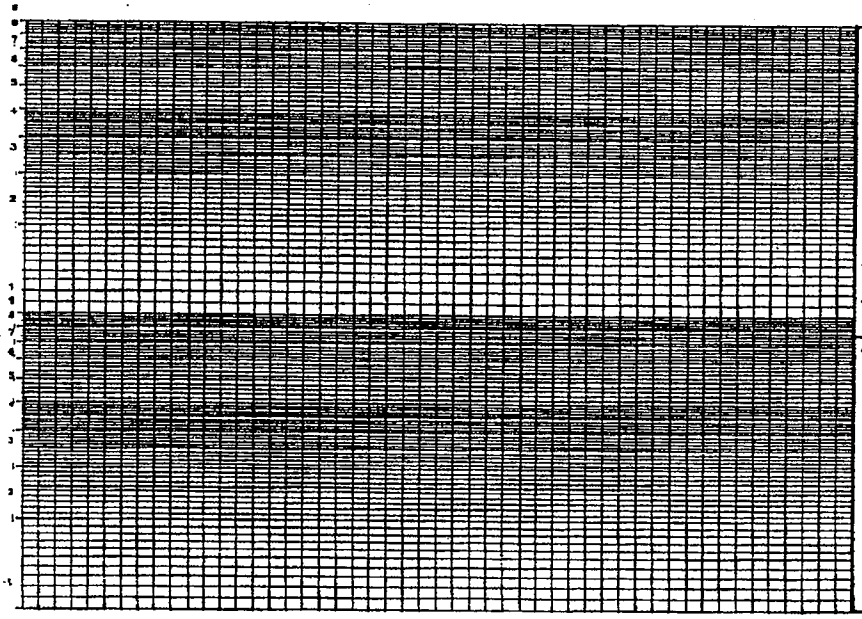
REFERENCE PT. FOR WL MEAS. (Top of Casing, Transducer, etc.): .....

ELAPSED TIME (min. or sec.)	MEASURED WATER LEVEL (feet)	DRAWDOWN OR HEAD (ΔH) (feet)	ELAPSED TIME (min. or sec.)	MEASURED WATER LEVEL (feet)	DRAWDOWN OR HEAD (ΔH) (feet)

WELL SCHEMATIC



▽ Indicate SWL Depth on Drawing



REMARKS:

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CALCS, SKETCH MAPS, ETC.:

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1.0 PURPOSE

The purpose of this procedure is to provide a basic understanding of developing contour maps and the approaches used to identify and quantify the direction and rate of groundwater flow and contaminant plume movement.

2.0 SCOPE

This procedure provides only a general overview of the field techniques, mathematical and physical relationships and data handling procedures used for determining groundwater flow direction and rate. The references identified herein can provide a more complete explanation of particular methods cited, as well as a more comprehensive discussion on the interpretation of hydrogeologic data.

3.0 GLOSSARY

Aquifer - A geologic formation capable of transmitting usable quantities of groundwater to a well or other discharge point.

Aquitard - A geologic formation which retards the flow of groundwater due to its low permeability.

Confined Aquifer - An aquifer that is overlain and underlain by zones of lower permeability (aquitards). If the aquifer is "artesian," the potentiometric head of the aquifer at a given point is higher than the top of the zone comprising the aquifer at that point.

Equipotential Line - A line connecting points of equal elevation of the water table or potentiometric surface. Equipotential lines on the water table are also called water table contour lines.

Flow Line - A flow line indicates the direction of groundwater movement within the saturated zone. Flow lines are drawn perpendicular to equipotential lines.

Flow Net - A diagram of groundwater flow showing flow lines and equipotential lines.

Hydraulic Conductivity (K) - A quantitative measure of the ability of porous material to transmit water. Volume of water that will flow through a unit cross sectional area of porous material per unit time under a head gradient. Hydraulic conductivity is dependent upon properties of the medium and fluid.

Hydraulic Gradient (i) - The rate of change of hydraulic head per unit distance of flow at a given point and in the downgradient direction.

Hydraulic Head - The height to which water will rise inside a well casing, equal to the elevation head plus the pressure head. In a well screened across the water table, hydraulic head equals the elevation head, as the pressure head equals 0. In wells screened below the water table in an unconfined aquifer or screened at any interval within a confined aquifer, the head is the sum of the elevation of the aquifer (the elevation head) and the fluid pressure of the water confined in the aquifer (the pressure head).

Potentiometric (piezometric) Surface - A hypothetical surface that coincides with the static level of the water in an aquifer (i.e., the maximum elevation to which water will rise in a well or piezometer penetrating the aquifer). The term "potentiometric surface" is usually applied to confined aquifers, although the water table is the potentiometric surface of an unconfined aquifer.

Unconfined Aquifer - An aquifer in which the water table forms the upper boundary.

Water Table - The surface in the groundwater system at which the fluid pressure is equal to atmospheric pressure (i.e., the net pressure head is zero) and below which all strata are saturated with water.

4.0 RESPONSIBILITIES

Project Hydrogeologist - The project hydrogeologist has overall responsibility for obtaining water level measurements and developing groundwater contour maps. The hydrogeologist (with the concurrence of 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 of data points needed and which wells shall be used for 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 - All supporting 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.

5.0 PROCEDURES

5.1 Potentiometric Surface Mapping

5.1.1 Selection of Wells

All wells used to prepare a flow net in a plan or map view should represent the same hydrogeologic unit, be it aquifer or aquitard. All water level measurements used shall be collected on the same day, preferably within 2-3 hours. This is especially important when working in an area where groundwater levels are tidally influenced or influenced by pumping.

The recorded water levels, monitoring-well construction data, site geology, and topographic setting must be reviewed to ascertain that the wells are completed in the same hydrogeologic unit and to determine if strong vertical hydraulic gradients may be present. Such conditions will be manifested by a pronounced correlation between well depth and water level, or by a difference in water level between two wells located near each other but set to different depths or having different screen lengths. Professional judgment of the hydrogeologist is important in this determination. If vertical gradients are significant, the data to be used must be limited vertically, and only wells finished in a chosen vertical zone of the hydrogeologic unit can be used.

At least three wells must be used to provide an estimation of the direction of groundwater flow; information from many more wells are needed to provide an accurate contour map. Generally, shallow systems require data from more wells than deep systems for accurate contour mapping. Potentiometric surface mapping for shallow flow systems also requires water level measurements from nearby surface water bodies.

5.1.2 Water Level Measurements

After selection of the wells to be used for mapping, the next step in determining the direction of groundwater flow is to obtain water level elevations from the selected points. In addition, any other readily available wells/surface water bodies should be measured to ensure that sufficient data are available for interpretation purposes.

Elevations are obtained from measurements of the depth to water in a monitoring well or piezometer taken from the top of the well casing (see SOP GH-1.2) and then referencing the elevation of the casing to a chosen and consistent datum point, usually mean sea level. Subtracting the depth to water from the casing elevation provides the elevation of the potentiometric surface. Elevations of points and areas of groundwater discharge or recharge such as springs, seeps, streams, rivers, and lakes also need to be determined, typically through staff gauge measurements. Comparison of these elevations, which represent hydraulic heads, will reveal the direction of flow because groundwater flows from areas of high head to areas of low head.

5.1.3 Construction of Equipotential Lines

Graphical methods available for depicting the flow of groundwater include the use of equipotential lines and flow lines to construct potentiometric surface maps and vertical flow nets. If the hydrogeologic system consists of a water table aquifer and one or more confined aquifers, separate contour maps should be prepared for each aquifer system. Water table maps should be developed using water level measurements obtained from monitoring wells screened at the unsaturated-saturated interface. Water level measurements collected from monitoring wells screened in the deeper portions of an unconfined aquifer should generally be contoured as a separate potentiometric surface map. Surface water discharge or recharge features are contoured in the water table system. Vertical flow nets should be constructed using a cross section aligned parallel to the direction of groundwater flow. All water level measurements along this cross section, both deep and shallow, are used in developing equipotential lines and flow lines for the flow net.

To construct equipotential lines, water level elevations in the chosen wells are plotted on a site map. Other hydrogeologic features associated with the zone of interest -- such as seeps, wetlands, and surface-water bodies -- should also be plotted along with their elevations.

The data should then be contoured, using mathematically valid and generally accepted techniques. Linear interpolation is the most commonly used technique. However, quadratic interpolation or any technique of trend-surface analysis or data smoothing is acceptable. Computer-generated contour maps may be useful rough mapping of large data sets; however, final, detailed mapping must always be performed by hand by an experienced hydrogeologist. Contour lines shall be drawn as smooth, continuous lines which never cross one another.

Inspect the contour map, noting known features, such as pumping wells and site topography. The contour lines must be adjusted utilizing the professional judgment of the hydrogeologist in accordance with these features. Closed contours should be avoided unless a known groundwater sink (i.e., pumping well) or mound exists. Groundwater mounding is common under landfills and lagoons; if the data imply this, the feature must be evident in the contour plot.

5.1.4 Determination of Groundwater-Flow Direction

Flow lines shall be drawn so that they are perpendicular to equipotential lines. Flow lines will begin at high head elevations and end at low head elevations. Closed highs will be the source of additional flow lines. Closed depressions (i.e., wells) will be the termination of some flow lines. Care must be used in areas with significant vertical gradients to avoid erroneous conclusions concerning gradients and flow directions.

5.2 Groundwater Flow Considerations

Groundwater movement is an integral part of the hydrologic cycle. Recharge to the shallow groundwater environment generally occurs by infiltration of precipitation through an upper unsaturated soil zone. Movement is downward under the force of gravity until the water reaches the saturated zone of the water table aquifer. Once water is part of the water table aquifer, movement is controlled by differences in hydraulic head, with movement from areas of high head to areas of low head. Areas of low head include natural discharge areas such as springs, lakes, rivers, and, ultimately, the ocean. These features can be considered as outcrops of the water table. Points of low head also are created by pumping wells.

Local head differences and consequent vertical flow patterns within an aquifer can be detected by well clusters. A well cluster consists of several adjacent wells, generally installed within a few feet of each other, and screened at different depths. Variations in water levels in these closely spaced wells indicates the vertical component of groundwater flow within an aquifer, provided that the wells are all screened within the same aquifer.

The number, location, and extent of geologic units and their properties with regard to aquifer or aquitard characteristics must be understood to properly interpret water level data gathered from the monitoring system. This firm understanding of the hydrogeologic system must be developed through a program of borings, wells, and interpretation of subsurface geology. The adequacy of the positions and depths of borings/wells used to define relevant subsurface hydrogeologic conditions must also be assessed. The location of surface water discharge or recharge points must be considered. Surface water features influence the system, as flow is most likely toward them (if they are discharge points) or away from them (if they are recharge points). Man-made discharge or recharge features such as pumping or injection wells, ditches, and trenches can also affect the flow of groundwater.

5.3 Determination of Flow Rate

Darcy's Law states that the quantity of water flowing through a geologic material is dependent upon the permeability of the material, the hydraulic gradient, and the cross sectional area through which the water flows. This relation is expressed in the equation:

$$Q = KiA$$

where:

Q	=	volume of water flowing through the cross sectional area of the formation (L^3/T).
K	=	hydraulic conductivity (L/T).
i	=	hydraulic gradient (L/L, i.e., dimensionless).
A	=	cross sectional area of formation being considered (L^2).

The relation is similar to one used in stream flow measurements where:

$$Q = VA$$

where:

Q	=	discharge from the cross sectional area of a stream or pipe (L^3/T).
V	=	average velocity of flowing water (L/T).
A	=	cross sectional area through which water flows (L^2).

The velocity of water movement in a geologic formation depends on the specific formation properties and the head differences across the formation. This relation is defined in the equation:

$$V = \frac{Ki}{n}$$

where:

V	=	average linear velocity of groundwater through the formation (L/T)
K	=	hydraulic conductivity (L/T)
i	=	hydraulic gradient (dimensionless)
n	=	porosity (expressed as a fraction).

Values of porosity for several geologic materials are given in Attachment A. More accurate and specific values of porosity can be obtained by laboratory analysis of a formation sample or from an unconfined aquifer pumping test.

Hydraulic conductivity is related to the permeability of the formation and depends on the size and interconnection of the pore spaces. In isotropic and homogeneous formations, the hydraulic conductivity will be the same vertically and horizontally. In anisotropic formations, horizontal and vertical conductivity can be markedly different and the vertical hydraulic conductivity can be up to several orders of magnitude lower than the horizontal hydraulic conductivity. Typically, most formations are anisotropic with horizontal hydraulic conductivities at least several times as high as the vertical hydraulic conductivities.

Generally, hydraulic conductivities are high for sands, gravels, and limestone containing large solution cavities and low for silts, clays, and tightly fractured rock. Attachment A gives values of hydraulic conductivity for several geologic materials. More accurate values can be obtained during field testing of aquifers or from laboratory measurements on undisturbed cores. Results from field testing usually provide higher (and more representative) hydraulic conductivities than laboratory testing because full-scale field testing includes the effects of the formational macrostructure (i.e., secondary permeability due to jointing or fractures) which is not reflected in the testing of a small sample in the laboratory.

The hydraulic gradient, i, is determined from field measurements of hydraulic head obtained from water level measuring points. Do <u>not</u> measure gradient from well to well; measure across equipotential lines that are drawn based on the well (and other) data. Once a potentiometric surface map has been generated using the hydraulic head data, the hydraulic gradient can be calculated using the following formula:

$$i = \frac{dh}{dl}$$

where:

dh	=	change in head (L)
dl	=	distance between equipotential lines (L)

The hydraulic gradient along any flow line can be calculated from a potentiometric surface map by dividing the change in head by the length of the flow line, typically beginning and ending at equipotential lines. The longer the distance over which the head change is measured, the more representative the gradient is of overall conditions.

When chemical solutes are traveling in groundwater, as in cases of groundwater contamination, the calculated groundwater velocity may predict migration rates in excess of what is actually observed. The difference in chemical versus water velocities may be due to attenuation or biodegradation of the chemical species in the aquifer. Attenuation is most often caused by adsorption of the chemical contaminant onto the formation grains or matrix. The result is that the chemical does not appear at the downgradient sampling point as quickly as the velocity calculation predicts. An equation to correct for this attenuation is:

$$V_c = V_w / (1 + K_d P_b / n)$$

where:

V_c	=	velocity of the chemical solute flow (L/T)
V_w	=	velocity of groundwater flow (L/T)
P_b	=	formation mass bulk density (M/L^3)
n	=	formation porosity (expressed as a fraction)
K_d	=	distribution coefficient = (L^3/M)

The K_d is equal to the mass of solute per unit mass of solid phase divided by the concentration of solute in solution. The term in the denominator is known as the retardation factor.

Density and/or viscosity differences between water and contaminants can also cause velocity determination errors. Light hydrocarbons such as gasoline are less dense than water and consequently float on the water table. These contaminants can migrate along the water table surface at rates faster or slower than the rate of groundwater movement, depending on specific conditions, and may also volatilize into unsaturated soil pore spaces. Oils are more viscous than water and will typically migrate more slowly due to the viscosity difference. Contaminants denser than water such as heavy hydrocarbons (e.g., coal tar) or chlorinated compounds (e.g., TCE, PCE) tend to sink to the bottom of an aquifer if present in concentrations exceeding their solubility limit (these chemicals are often referred to as dense, nonaqueous phase liquids, or DNAPLs if present as a separate-phase liquid). Here, the contamination may move at faster or slower rates than the overlying groundwater or may actually move in a direction opposite to that of the groundwater, depending on the geologic characteristics of the aquifer base and direction of dip of the underlying aquitard.

Other factors involving the physicochemical interaction between the chemical and the groundwater, such as dilution (mixing contaminated water or chemicals with additional quantities of groundwater) and dispersion (molecular diffusion of the chemical throughout the groundwater regime), can also affect the observed rates of travel of contaminants in groundwater. In addition to such physicochemical characteristics, all of the aquifer and aquitard properties and groundwater flow characteristics described above must be known so that adequate and accurate estimations of the extent and rate of groundwater contaminant migration can be developed.

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ATTACHMENT A

GENERALIZED POROSITY AND HYDRAULIC CONDUCTIVITY VALUES FOR GEOLOGIC MATERIALS

Material	Porosity Range (%)	Hydraulic Conductivity Range	
		cm/sec	ft/day
Gravel	30-40	10^{-1} to 10^{-2}	280 to 2.8×10^{5}
Coarse sand (clean)	30-40	10^{-1} to 1	280 to 2,800
Medium sand (clean)	35-45	10^{-2} to 10^{-1}	28 to 280
Fine sand (clean)	40-50	5×10^{-4} to 10^{-2}	1.4 to 28
Silty sand	25-40	10^{-5} to 10^{-2}	0.03 to 280
Glacial Till	Variable	10^{-10} to 10^{-4}	3×10^{-7} to 0.3
Unweathered Clay/Shale	45-55 (clay)	10^{-7} to 10^{-4}	3×10^{-4} to 0.3 (horizontal)
		10^{-10} to 10^{-6}	3×10^{-7} to 3×10^{-3} (vertical)
Karst Limestone	---	10^{-4} to 10^{-1}	0.3 to 2,800
Fractured Igneous/Metamorphic Rocks	---	10^{-6} to 10^{-1}	3×10^{-3} to 280
Sandstone	5-30	10^{-8} to 10^{-4}	3×10^{-5} to 0.3

Source: References 1 and 2

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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.

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.

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.

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.

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.

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

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

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

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

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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®
2 Stainless Steel 316
3. Stainless Steel 304
4 PVC 1
5 Lo-Carbon Steel
6 Galvanized Steel
7 Carbon Steel

* 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®
2 Polypropylene (PP)
3. PVC Flexible/PE Linear
4 Viton®
5 PE Conventional
6 Plexiglas/Lucite (PMM)
7 Silicone/Neoprene

* Trademark of DuPont

Source: Barcelona et al., 1983

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.

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1.0 PURPOSE

The purpose of this guideline is to furnish a guide to planning and implementing a program of borehole geophysics applicable to hazardous waste site investigations.

2.0 SCOPE

This guideline provides a description of the principles of operation, instruments, applicability, and implementability of borehole geophysics to determine subsurface stratigraphy and groundwater conditions at hazardous waste sites. The document is intended to allow personnel to develop an understanding of the available tools for proper work plan development, scheduling, and resource planning. This guidance is not intended to provide a detailed description of operations. The highly specialized nature of borehole geophysics requires inclusion of site-specific information prior to development of detailed operating plans.

3.0 GLOSSARY

Dead Time. Measurement errors in nuclear logging occurring from the inability to record all of the pulse energy within the resolving time.

Density. Mass per unit volume (g/cm^3). Bulk rock densities vary mainly because of porosity and typically range from 1.9 to 2.8 g/cm^3.

Electric Logs. The generic term for a well log that displays electrical measurements of induced current flow between electrodes. The electric log generic term usually relates to a resistivity log and spontaneous potential (SP) log. Electric logs discussed in this subsection include single-point resistivity, SP, electromagnetic (EM) induction, and normal resistivity.

Nuclear Logs. The generic term for a well log that either measures natural or induces and measures radioactive isotopes in the borehole environment. Discussion in this text is limited to natural gamma, gamma-gamma, and neutron.

Porosity. The ratio of void volume to total volume. Usually expressed as a percent.

Sonde. The elongate cylindrical tool assembly used in a borehole to acquire well log information.

4.0 RESPONSIBILITIES

Project Manager. Responsible for scoping of borehole geophysical surveys during development of the Work Plan, with input from the FOL, site geologist, and site geophysicist.

Field Operations Leader. Responsible for overall management and coordination of the field work.

Site Geophysicist. As a specialist in this field, the site geophysicist plays a central role in determining the appropriateness of this technique for providing necessary data. Field work for these surveys is supervised by the site geophysicist, with support from geophysical technical specialists and other personnel as needed. Data reduction and interpretation are performed by the site geophysicist or technical specialist.

5.0 PROCEDURES

5.1 Applicability

Discussion in this subsection will introduce a variety of borehole geophysical methods. The general logging categories discussed are electrical, nuclear, sonic, and mechanical. Although other borehole techniques are available, such as three-dimensional vertical seismic profiling, borehole televiewing, and a variety of crossbore techniques, these are not discussed in detail because of their limited application and the very specialized nature of their use. A combination of surface and borehole techniques offers a three-dimensional understanding of subsurface conditions, but that approach is also beyond the introductory detail in this compendium.

5.2 Preliminary Considerations

Equipment discussed in this guideline is capable of performing electric, nuclear, and mechanical logging. This equipment is available from a variety of vendors and can usually be rented for short periods of time or leased on a long-term basis. In any case, the application of these techniques is quite complex, and the site geophysicist should provide input for planning and implementing borehole programs.

The following general types of information can be expected from borehole measurements:

- Vertical changes in porosity
- Relative vertical changes in permeability and transmissivity
- Lithology and structure
- Lithologic conditions
- Vertical distribution of leachate plumes
- Groundwater gradients, flow direction, and rate
- Water quality parameters

To determine a logging program that will enhance evaluation of the site, the site geophysicist (SG) must thoroughly evaluate two key items. First, the SG must identify the regional bedrock geology (e.g., igneous, sedimentary, metamorphic) and typical surficial units. Then the SG must gather as much local information as possible regarding geologic units (e.g., boring logs of monitoring wells, domestic water supply depths, and well yields) and any hydrogeologic reports or information.

Second, the SG must identify which logs are applicable in the site's geologic setting and which logs will provide the required information for meeting project objectives. Table 1 is a general guide to data collection objectives that will aid in the selection process. However, each function under consideration must be researched in more detail using publications listed as information sources in this manual and consult with borehole geophysical logging specialists.

There are, of course, limiting factors for each of the logging techniques. Table 2 identifies some limiting factors for the logs.

Once the geologic environment has been evaluated and the logging functions narrowed, the SG must select the appropriate equipment. Portable units that can be carried on a backpack enable access to most well locations; however, they are limited to logging functions requiring low power operation (e.g., battery packs).

TABLE 1

GENERAL GUIDE TO DATA COLLECTION ACTIVITIES

Data Collection	Available Techniques
Lithology and stratigraphic correlation	Electric(1)*, caliper*, nuclear, sonic(1), induction
Total porosity or bulk density	Gamma-gamma, neutron, and sonic(1)
Effective porosity or true resistivity	Long-normal resistivity(1)* (records the resistivity beyond the invaded zone)
Clay or shale content	Natural gamma, induction, spectral gamma
Secondary permeability (fractures, solution openings)	Caliper*, electric(1)*, sonic(1), video inspection, vertical flow meter(1)*
Specific yields of unconfined aquifer	Neutron
Water level and saturated zones	Electric(1)*, neutron, gamma-gamma, temperature(1)*, fluid conductivity*, and induction
Moisture content	Neutron, induction
Dispersion, dilution, and movement of waste	Fluid conductivity(1)* and temperature(1)*
Groundwater movement through a borehole	Flow meter (vertical)(1)*
Cementing	Caliper*, temperature(1)*, gamma-gamma, and sonic(1) (cement bond log)
Casing corrosion	Caliper*, video inspection
Vertical delineation of conductive contaminant plume	Induction
Measuring radionuclide contamination	Spectral gamma
Groundwater flow	Vertical flow meter(1)*

1 Requires fluid filled borehole for valid data collection.
* Generally not applicable in a cased well.

TABLE 2

LOGGING FUNCTION BOREHOLE LIMITATIONS

Logging Function	Limiting Factors		Fluid Filled Borehole Required	Able to Log Saltwater or Brackish Ground Waters/Muds
	Uncased Open Boreholes (Only)	Minimum Diameter (Inches)		
Spontaneous potential	X	2	X	
Single-point resistance	X	2	X	
Natural gamma		2		X
Gamma-gamma		2		X
Neutron		2		X
Caliper		2		X
Temperature	X	2	X	X
Fluid conductivity	X	2	X	
Fluid movement	X	2	X	X
Sonic		2	X	X
EM induction		2		
Normal resistivity	X	2	X	
Cement bond		2	X	X
Spectral gamma		3		X
Video inspection		2		X

X = Required condition.

Functions that require 110 volt AC usually operate from a larger unit that is typically mounted in a vehicle. These units cost considerably more, and access to well locations can present problems in swampy areas. However, these units are able to run the majority of log functions available today.

A basic description of available logs, the parameters that affect response, and the sensing devices are presented in the following sections to aid in evaluating the applicability of logging functions.

5.2.1 Electrical

Electrical logging includes spontaneous potential, single point resistance, resistivity, and EM induction techniques.

5.2.1.1 Spontaneous Potential (SP)

The response is the result of small differences in voltage caused by chemical and physical contacts between the borehole fluid and the surrounding formation. These voltage differences appear at lithology changes or bed boundaries, and their response is used quantitatively to determine bed thickness or formation water resistivity. Qualitative interpretation of the data can help identify permeable beds.

In a consolidated rock aquifer system where groundwater flow is controlled by secondary permeability (e.g., fractures), SP response may be generated from a streaming potential caused by a zone gaining or losing water.

The SP log is a graphic plot of potentials between the downhole sonde and a surface electrode. The system consists of a moveable lead electrode (located in the sonde) that traverses the borehole and a surface electrode (mud plug) that measures potentials in millivolts. Noise and anomalous potentials are relatively common in SP logs and are discussed in electric log anomalies later in this manual.

5.2.1.2 Single-Point Resistance

This technique is based on the principle of Ohm's Law ($V = Ir$) where V is voltage measured in volts, I is current measured in amperes, and r is resistance measured in ohms. Single-point resistance measures the resistance of in-situ materials (of the rock and the fluid) between an in-hole electrode and a surface electrode. Resistance logging has a small radius of investigation and is very sensitive to the conductivity of the borehole fluid and changes in hole diameter (caving, washouts, and fractures). This condition is advantageous for the operator in that any change in the formation (resistance or fractures) will produce a corresponding change in resistance on the log. These changes in resistance are interpreted to be a result of lithology changes. The single-point log is very desirable for geologic correlation because of its special response to lithology changes.

In crystalline rock (high-resistance formations), single-point resistance logs are useful in locating fractures and often appear as mirror images (opposite deflections) to the caliper log. Hole enlargement, caving, washouts, and fractures appear as excursions to the left (indicating less resistance in normal operation) of the more typical response observed in this log.

The principle of the function is quite simple. The current (I) remains constant while the voltage (V) is measured between the movable lead electrode and the surface electrode. Voltage is then converted internally to resistance using Ohm's Law. SP and single-point resistance logs are designed to be run simultaneously since single-point resistance operates in alternating current (AC) (110 volt) while the SP operates in direct current (DC).

5.2.1.3 Normal Resistivity

Normal resistivity logs measure the apparent resistivity of the subsurface formation in units of ohm-meters. The normal resistivity log measures the voltage between one of two down hole potential electrodes and a surface potential electrode and two current electrodes. One current electrode is placed at ground surface and the second current electrode is located at the bottom of the sonde. The two potential electrodes are generally located 16 inches away and 64 inches away from the downhole current electrode. When data are collected with the potential electrode that is 64 inches away from the downhole current electrode, it is referred to as a "long normal resistivity log" data. When data are collected using the potential electrode spaced 16 inches from the downhole current electrode, it is referred to as the "short normal resistivity log" data.

The radius of investigation with the tool is equal to approximately one-half the distance between the downhole current electrode and the downhole potential electrode. However, the radius of investigation will vary as a result of resistivity and thickness of the zone it is measuring. Therefore, a long normal log has a greater radius of investigation into the formation than the short normal log. If the borehole has been drilled with mud or foam it may be necessary to review both the long and short normal logs. The short normal log is used to measure the resistivity of fluid invasion zone near the borehole wall. The long normal log is used to measure the bulk resistivity of the natural formation, natural pore water, and the fluid invasion zone near the borehole wall. By comparing both responses, an estimate of the true resistivity of the formation can be made.

Normal resistivity logging can only be performed in uncased boreholes that are fluid filled. The presence of borehole fluids acts to electrically couple the electrodes to the borehole wall. Spontaneous potential data are generally collected simultaneously with normal resistivity data.

5.2.1.4 Electromagentic Induction

Electromagnetic (EM) induction tools measure the apparent electrical conductivity of subsurface materials and their pore fluid in units of conductivity [millimhos per meter (mmhos/m) or milliSiemens per meter (mS/m) or in units of resistivity (ohm-meters). Induction tools are equipped with a radio transmitter and receiver and have a radius of investigation that is dependant upon their separation. Generally, the larger the separation of the transmitter from the receiver the greater the radius of investigation at the expense of resolution. The radio transmitter produces a primary EM signal that induces small eddy currents in the subsurface materials. The eddy currents produce a secondary EM field that, together with the primary signal, are sensed by the device's receiver. The relationship of the primary and secondary signals is linearly proportional to the apparent conductivity of the subsurface material.

Induction tools used for environmental and engineering projects are commonly configured such that the instrument's sensitivity increases radially away from the tool to a maximum response at a radius of one foot away from the sonde. This renders the device relatively insensitive to the influence of borehole fluids and well construction materials in small diameter wells (two to eight inch diameter). Induction tools can be operated in polyvinyl chloride (PVC) cased or open boreholes and above or below the water column. Other electrical borehole tools must be used in open or uncased boreholes and require the presence of borehole fluids or muds.

Induction tools are very useful in mapping and delineating stratigraphy based upon contrasts of various soils' or rocks' ability to conduct electricity. Typically clayey soils and rocks have high conductivities relative to sandy soil, limestones, dolostones, and sandstones that generally have low conductivities. The device is also useful in mapping the presence, absence, or vertical extent of a conductive contaminant

plume. A natural gamma tool is often used in conjunction with the induction device when the objectives are to map both a contaminant plume and stratigraphic changes.

5.2.2 Nuclear

Nuclear logging includes natural gamma, gamma-gamma, neutron, and spectral gamma.

5.2.2.1 Natural Gamma

Natural gamma logs measure the presence of naturally occurring gamma radiation in units of counts per second (CPS) through the use of a scintillation crystal (sodium iodide) and a photo-electric tube. The sodium iodide crystal emits light when in the presence of gamma radiation. The light is then converted to an electrical signal by the photo-electric tube. The electrical components measure the number of light flashes per second and digitally record them onto a laptop computer.

The principle sources of natural gamma radiation are from the uranium-thorium decay series and radioactive Potassium (K^{40}). Igneous rocks are generally more radioactive than silica-rich rocks as a result of the presence of potassium feldspar and micas. Clayey soils and rocks containing clay minerals often have large amounts of uranium and thorium as a result of the process of adsorption and ion exchange. Quartz sands and carbonate soil and rock often contain low amounts of radionuclides. This common contrast of naturally occurring radionuclide content of various rock types makes the natural gamma tool an excellent device for mapping stratigraphy based upon the presence or absence of clay materials. This is useful to the hydrogeologist since clay content influences permeability and effective porosity within sedimentary rocks.

The natural gamma log can be used to collect data above or below the water column in PVC-cased, steel-cased, or open boreholes. This device is often used in conjunction with an EM induction log as it is not sensitive to changes in a subsurface material's conductivity. For example, if the induction log and the natural gamma log both show a high response across a zone, it can be interpreted that the zone contains clayey soils or rocks. However, if a high induction log response is observed and a low natural gamma log response is observed across this zone, it can be interpreted that conductive pore fluids may be present, such as contaminated groundwater.

5.2.2.2 Gamma-Gamma

The gamma-gamma log, also known as a density log, is used to measure lithologic changes based upon the bulk density of subsurface materials. The gamma-gamma log typically uses a low energy gamma source such as Cesium (Cs^{137}) and one to two scintillation detectors, The gamma radiation emitted by the source is scattered by the electrons in the rock formation. The gamma radiation that is not scattered is measured by the scintillation detectors attached to the tool. The measured gamma radiation is inversely proportional to electron density of the subsurface material. Density logs are also often used for determination of porosity variations as a function of density, for lithologic correlation, or to characterize subtle variations in depositional environments. The radius of investigation is dependent upon two factors: source strength and source to detector spacing. Typically, 90 percent of the response is from within a radius of 5 to 6 inches from the sonde. Measurements are presented in unit of CPS.

5.2.2.3 Neutron Log

A neutron tool is used to measure the relative amount of hydrogen present (from water or methane) in the subsurface soils or rock. The device operates through the use of a neutron source (radioactive source)

and a neutron detector. Radioactive sources used for neutron logging generally consist of a mixture of beryllium and americium. Neutron detectors generally consist of lithium iodide crystals or helium-3 tubes. The radioactive source emits neutrons that are scattered or lose energy and are captured. Neutrons are scattered by colliding with atoms that are larger than the neutrons. Neutrons that collide with atoms that are their same approximate size lose energy and are captured. A hydrogen atom is approximately the same size as a neutron, thus the more hydrogen present the greater the neutron capture.

The neutron detector measures the presence of non-captured neutrons. The amount of non-captured neutrons are inversely proportional to the amount of hydrogen present. Hydrogen content can be related to the presence of water or methane gas in formation void space. Low neutron detector values (low amount of neutron scatter) are often an indication of high water or gas content. High neutron detector values (large amount of neutron scatter) are an indication of low amounts of water or gas present.

Hydrogeologists often use neutron logs as indicators of the relative porosity of water bearing formations. Clayey soils and rocks with abundant clay minerals often have a high porosity. This can result in a low neutron response if groundwater is present in the pore space. As clay soils often have higher porosity (up to 40%) than sandy soils (generally 10 to 20%), it is not uncommon to have a lower neutron response in an unsaturated clay soil zone than at a saturated sandy soil zone. Neutron logs are also used to measure the presence and vertical extent of perched water tables. Data are presented in units of counts per second (CPS) and can be correlated to formation porosity and moisture content.

5.2.2.4 Spectral Gamma

Spectral gamma logs are used to identify the natural and artificial radionuclides present in soils, fill, groundwater, gases, and rocks. Spectral gamma data are often used for lithologic correlation and to map radioactive groundwater and gas contaminant plumes associated with nuclear waste sites.

The spectral gamma tool is similar to a natural gamma log in that it uses a sodium iodide crystal and a photo-electric tube to measure gamma radiation present in the subsurface. Natural gamma tools measure the full spectrum of gamma radiation present. Spectral gamma tools utilize a multi-channel analyzer to separate the various isotopes based upon their gamma energy [measured in million electron volts (Mev)]. For example, Potassium-40 has a gamma energy of 1.46 Mev whereas the Thorium-232 decay series has a gamma energy of 2.62 Mev (Keys, 1989). Each channel of the multi-channel analyzer is set to record the counts per second within a specific energy range. Data are generally presented as counts per second per each energy range of interest and total counts per second for the entire natural gamma spectrum.

5.2.3 Mechanical

Mechanical logging includes caliper, temperature, fluid conductivity, and fluid movement.

5.2.3.1 Caliper

This log is defined as a continuous record of the average diameter of a drill hole. Caliper sondes can have from one to four arms. The two basic types are bowstring units, which are connected at two hinges, and finger devices, which have single hinges. Three and four arm caliper tools are often preferred for environmental/engineering investigation as they can better define zones of incompetent rock.

Caliper resolution is broken into two categories: horizontal and vertical. The horizontal resolution is the ability of the tool to measure the true size of the hole regardless of its shape (circular or elliptical). Vertical resolution is controlled by the length of the feeler contact on the borehole wall.

Traditionally, caliper logs have been run to correct other logging functions. If this is the primary reason for running caliper, the bowstring or single- hinged unit will both provide adequate data. Calipers using single-hinged feelers provide the best vertical resolution. Interchangeable arms are available for the single-hinged tools and should be selected on the basis of the hole diameter. Single-hinged tools can be used to identify fractures in igneous and metamorphic rocks and solution openings in limestone.

5.2.3.2 Temperature

The temperature log provides continuous records of the borehole fluid environment. This log should always be run first to minimize disturbance of the thermal-stratification of the borehole fluids. Response is caused by temperature changes of the fluid surrounding the sonde, and generally relates to the formation water temperature. The borehole fluid temperature gradient is influenced by fluid movement in the borehole and adjacent rocks. In general, the temperature gradient is greater in low-permeability rocks than high-permeability rocks, which is probably the result of groundwater flow. Therefore, temperature logs can provide the hydrologist with information regarding groundwater movement.

Logging speed should be slow enough (6 ft/min or less) to allow adequate sonde response with depth, because there is a certain amount of lag time. The probe is designed to be run from top to bottom (downward) in the borehole to channel water past the sensor. Because disturbance is inevitable when the sonde moves through the water column, repeat temperature logs should be avoided until the borehole fluid has had time to reach thermal equilibrium. Data are often presented as differential temperature vs. depth.

5.2.3.3 Fluid Conductivity (Fluid Resistivity)

These logs provide a continuous measurement of the conductivity (or resistivity) of the borehole fluid between two electrodes. The contrast in conductivity can be associated with water quality and possibly with recharge zones. Conductivity logs are helpful when interpreting electric logs, because both are affected by fluid conductivity.

The most common sonde measures the AC voltage drop across closely spaced electrodes. These electrodes actually measure the fluid resistivity (which is the reciprocal of conductivity), but are called fluid conductivity logs to avoid confusion with resistivity logs. Simply, conductivity logs actually measure the resistance of the borehole fluid; resistivity logs measure the resistance of the rocks and the fluid they contain. Fluid conductivity logs are often combined with temperature log tools such that the data can be simultaneously collected in the undisturbed borehole fluids.

5.2.3.4 Vertical Flow Meters

Two types of vertical flow meters are commonly used for groundwater investigations: an impeller flow meter or a heat-pulse flow meter.

An impeller flow meter is used to measure vertical flow under high flow rate conditions. The impeller flow meter is a calibrated impeller housed in a protective cage that is either dragged through the water column at a constant rate or kept stationary at a predetermined depth. The revolutions of the impellers can be correlated to a vertical flow rate. When dragged through the water column, zones of anomalous flow rates can be identified by observing changes in the impeller speed. The impeller flow meter is only useful for measuring vertical flow rates of four to five ft/min or greater.

In areas of low vertical flow, a heat-pulse flow meter is used. The heat pulse flow meter consists of a heat source and two thermistors located above and below the heat source. The device is kept stationary at a

pre-determined zone. A heat pulse is then generated at the center of the probe and two thermistors located a known distance above and below the heat source, measure the temperature change verses time. The heat pulse flow meter is used when vertical flow rates are anticipated to be 0.1 to less than 20 ft/minute.

5.2.3.5 Borehole Video Inspection

Video inspection surveys are used in cased water and groundwater monitoring wells to verify well construction details such as screen length. Video inspection surveys are also commonly used in cased wells where casing is suspected to be damaged, or to examine the screen condition (e.g., degree of encrustation or siltation). Video inspection surveys are performed in uncased boreholes and monitoring wells for the purpose of identifying fractures, voids or lithologic changes. A video inspection tool consists of a color or black and white video camera housed in a two-inch diameter water proof stainless steel case. Video inspection systems for environmental logging are generally equipped with 750 feet of PVC coated cable, a digital depth encoder, a PVC camera centralizer, and an 8-mm video recording device.

5.2.3.6 Sonic

This logging technique (also called acoustic logging) uses sound waves to measure porosity and to identify fractures in consolidated rock. Two general types of measurements are internal transit time, (the reciprocal of velocity), and amplitude (the reciprocal of attenuation). The amplitudes of the P- and S-waves are directly related to the degree of consolidation, porosity, and the extent and orientation of fractures.

The instrumentation of acoustic logging is very complex; it includes a downhole sonde with a transmitter and two to four receivers. Sound waves are emitted from the transmitter and their propagation is measured by the receivers.

5.2.3.7 Cement Bond Log

Cement bond logging is performed when it is suspected that the cement or grout between the casing and the borehole wall contains voids. This is an important log to run if it is suspected that contamination is vertically migrating as a result of an improperly grouted or damaged monitoring well. A cement bond log measures the amplitude of an acoustic wave reflecting from the well casing. The amplitude of the reflected acoustic wave is low when a good bond exists between the casing and the borehole wall or areas of increased grout thickness.

5.3 Survey Design

5.3.1 Log Selection

Once the SG has defined the logging program and has identified the general category of logs that will supply the necessary information, the specific logging function(s) can be selected. Table 3 describes the type of log, a basic description, and the primary use of the technique.

TABLE 3

TYPES OF LOGS, DESCRIPTIONS, AND USES

Type of Log	Description	Primary Utilization
Caliper	A caliper produces a record of the average diameter of the drill hole.	Used for correction of other logs, identification of lithology changes, and locations of fractures, voids, and incompetent zones of rock.
Single-Point Resistivity	This log measures the resistance of the earth material between an in-hole electrode and a surface electrode.	Used to determine stratigraphic boundaries, changes in lithology, and the distribution of fractures in resistive rock.
Spontaneous Potential (SP)	SP is a measure of the small differences in voltage that develop between the borehole fluid and the surrounding formation.	Used for geologic correlation, determination of bed thickness, and separation of non-porous from porous rocks in shale-sandstone and shale-carbonate sequences.
Natural Gamma	This log measures natural gamma radiation emitted from Potassium-40, uranium, and thorium decay series elements.	Used for lithology identification and stratigraphy correlation; most advantageous in sediment environments where the fine-grained units often have the highest gamma intensity.
Gamma-gamma	Gamma photons are introduced in the borehole environment, and the absorption and scattering are measured to evaluate the medium they travel through.	Used for identification of lithology, measurement of bulk density, and porosity of rocks.
Neutron	Neutrons are introduced into the borehole and the amount of scattered neutrons are measured to evaluate the presence of hydrogen atoms.	Used to measure the moisture content above the water table and the total porosity below the water table. Also used to map perched water tables.
Temperature	A temperature log is the continuous record of the thermal gradient of the borehole fluid.	Used to determine groundwater flow in a borehole.
Fluid Conductivity	This log provides a measurement of the conductivity of the in-hole fluid between the electrodes.	Used primarily in conjunction with temperature and electric logs to aid in their interpretation; useful for identifying saltwater intrusion into freshwater systems; can be useful in evaluating water quality.

TABLE 3 (Continued)

TYPES OF LOGS, DESCRIPTIONS, AND USES

Type of Log	Description	Primary Utilization
Acoustic (sonic)	A transmitter and a receiver or series of receivers that use acoustic frequencies. These signals are introduced into the borehole, and the elastic waves generated are measured.	Used to measure porosity and identify fractures in igneous and metamorphic rock.
Cement Bond	A transmitter and receiver that uses acoustic frequencies to measure the amplitude of the signal reflected from the casing.	Used to examine the borehole for voids in grout that may permit vertical migration of contaminants.
EM Induction	This tool uses a VLF signal that in turn induces current into the zone of interest.	Most common environmental logging tool. Used to map lithology and conductive contaminant groundwater plumes.
Spectral Gamma	A scintillation crystal and multi-channel analyzer are used to measure the presence of various isotopes based upon their gamma energy.	Used to map lithology and to map radioactive contaminant plumes in groundwater (e.g., uranium) and gas (e.g., radon).
Video Inspection	A waterproof color or black and white camera used to inspect casing, screen, or borehole conditions.	Used to inspect condition and construction of water wells or groundwater monitoring wells. Also used to identify fractures, voids, and lithologic changes in open boreholes.

There are many combinations of logging functions. Generally, several borehole techniques are performed simultaneously or in a series to define the geologic or hydrologic parameters.

5.4 Interferences/Anomalies

5.4.1 Electrical

SP, single point resistance, and normal resistivity logs are susceptible to the same types of interference. Buried cables, pipelines, magnetic storms, and the flow of groundwater can all cause anomalous readings. The most common noise in the SP logs is known as the battery effect and is caused by the polarization of the wetted cable. This condition is most troublesome in highly resistive surface formations. A common interference with the resistivity log is the result of ground currents from power lines and other electrical sources that interfere with the alternating current used in logging. This interference appears as a sine wave superimposed on the resistance curve.

5.4.2 Nuclear

The most common problems with nuclear logs are that they are all affected by borehole diameter changes and changes in borehole media (air, water, mud). These problems are why caliper logs are essential to correlate the results. A natural gamma log is the sum of the radiation emitted from the formation and does not distinguish between elements (e.g., Potassium, Uranium, Thorium). In quantitative applications of nuclear logs, the calibration, standardization, and correction for dead time are essential. However, when the logs are used for qualitative interpretations (e.g., stratigraphic correlation), such corrections may be unnecessary.

5.4.3 Mechanical

Caliper logging is a straightforward mechanical technique and exhibits few anomalies. Instrument malfunctions are more likely to cause anomalous readings than borehole parameters.

Impeller flow anomalies are most often caused by varying the probe position radially in the borehole. Bouncing of the probe from side to side will erroneously indicate flow. Corrective action may include a device that would hold the sonde in the middle of the borehole.

Temperature logs are susceptible to thermal lag time, self-heating, drift from the electronics in the sonde, and borehole conditions. A slow logging speed and additional logging functions (e.g., caliper, fluid conductivity) can aid in temperature log interpretation. Another problem with temperature logs is that after one pass of the sonde, the thermal gradient is disturbed and repeat logs may not be representative. In large-diameter wells, convection can cause a disturbance of the thermal gradient.

Disturbances to the borehole fluid caused by changes in fluid density and thermal convection can cause errors in the fluid conductivity. Because fluid conductivity response is affected by the water chemistry, chemical equilibrium must be reached before measurements are taken. Well water may take months to obtain chemical equilibrium with the surrounding formation after drilling, and water wells with much internal movement may never reach chemical equilibrium. Repeat logs are not usually representative because the sonde disturbs the water column.

Cycle skipping is the most obvious unwanted signal in acoustic logging. It is caused by excessive signal attenuation in the fluid or by equipment malfunction. A problem with interpreting acoustic logs is that the

velocity is dependent on a variety of lithologic factors, and the widely used time-average equation does not account for most of the factors.

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7.0 RECORDS

The following information will be recorded in the field logbook.

- Date
- Type of log/equipment
- Equipment operators
- Name and project number of the site
- Condition of borehole (cased or uncased) and type of fluid present in borehole
- Site conditions that may affect data collection

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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.

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 ($^0/_{00}$) 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 be 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.

- 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.
- 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 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.

   - 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.
- 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 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.
- 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).

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:

- 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

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).
- 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.

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.

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. 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.

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.
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.

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.

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.

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.

- 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 form 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 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 was 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.

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:

- 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).

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
    - Specific conductance ±10%
    - Temperature ±10%
    - Turbidity less than 10 NTUs
    - DO ±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

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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/Length (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.

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:

- NA Not applicable
- AC Alternating current
- 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.

ATTACHMENT B
GROUNDWATER SAMPLE LOG SHEET

Tetra Tech NUS, Inc.

GROUNDWATER SAMPLE LOG SHEET

Page___ of ___

Project Site Name: _____
Project No.: _____

[ ] Domestic Well Data
[x] Monitoring Well Data
[ ] Other Well Type: _____
[ ] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____
Type of Sample:
[X] Low Concentration
[ ] High Concentration

SAMPLING DATA:

	Color	pH	S.C.	Temp.	Turbidity	DO	ORP	Other
Date:	Visual	Standard	mS/cm	Degrees C	NTU	mg/l	mV	NA
Time:								
Method:								

PURGE DATA:

Date:	Volume	pH	S.C.	Temp. (C)	Turbidity	DO	ORP	Other
Method:								
Monitor Reading (ppm):			SEE LOW FLOW PURGE DATA SHEET					
Well Casing Diameter & Material								
Type:								
Total Well Depth (TD):								
Static Water Level (WL):								
One Casing Volume(gal/L):								
Start Purge (hrs):								
End Purge (hrs):								
Total Purge Time (min):								
Total Vol. Purged (gal/L):								

SAMPLE COLLECTION INFORMATION:

Analysis	Preservative	Container Requirements	Collected

OBSERVATIONS / NOTES:

Circle if Applicable:		Signature(s):
MS/MSD	Duplicate ID No.:	

ATTACHMENT C
EQUIPMENT CALIBRATION LOG

Tetra Tech NUS, Inc.

EQUIPMENT CALIBRATION LOG

PROJECT NAME : _____ INSTRUMENT NAME/MODEL: _____

SITE NAME: _____ MANUFACTURER: _____

PROJECT No.: _____ SERIAL NUMBER: _____

Date of Calibration	Instrument I.D. Number	Person Performing Calibration	Instrument Settings		Instrument Readings		Calibration Standard (Lot No.)	Remarks and Comments
			Pre-calibration	Post-calibration	Pre-calibration	Post-calibration		

ATTACHMENT D
LOW FLOW PURGE DATA SHEET

LOW FLOW PURGE DATA SHEET

PROJECT SITE NAME: _____ WELL ID.: _____
PROJECT NUMBER: _____ DATE: _____

Time (Hrs.)	Water Level (Ft. below TOC)	Flow (mL/Min.)	pH (S.U.)	S. Cond. (mS/cm)	Turb. (NTU)	DO (mg/L)	Temp. (Celcius)	ORP mV	Salinity % or ppt	Comments

SIGNATURE(S): _____

PAGE___OF___

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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).

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.
- 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.
- 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 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.

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°C ± 2 °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.

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.
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.

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:

   - 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.

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.

- 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)
- 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:

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 proceeded 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.).
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 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.
- 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 where 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, 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:

- 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 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.

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.
- 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.

  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 <u>In-Pit Sampling</u>

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 trough 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.
- 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 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/O_2 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:

- 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.

ATTACHMENT A
SOIL & SEDIMENT SAMPLE LOG SHEET

Tetra Tech NUS, Inc.

SOIL & SEDIMENT SAMPLE LOG SHEET

Page___ of ___

Project Site Name: _____
Project No.: _____

[] Surface Soil
[] Subsurface Soil
[] Sediment
[] Other: _____
[] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____

Type of Sample:
[] Low Concentration
[] 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:

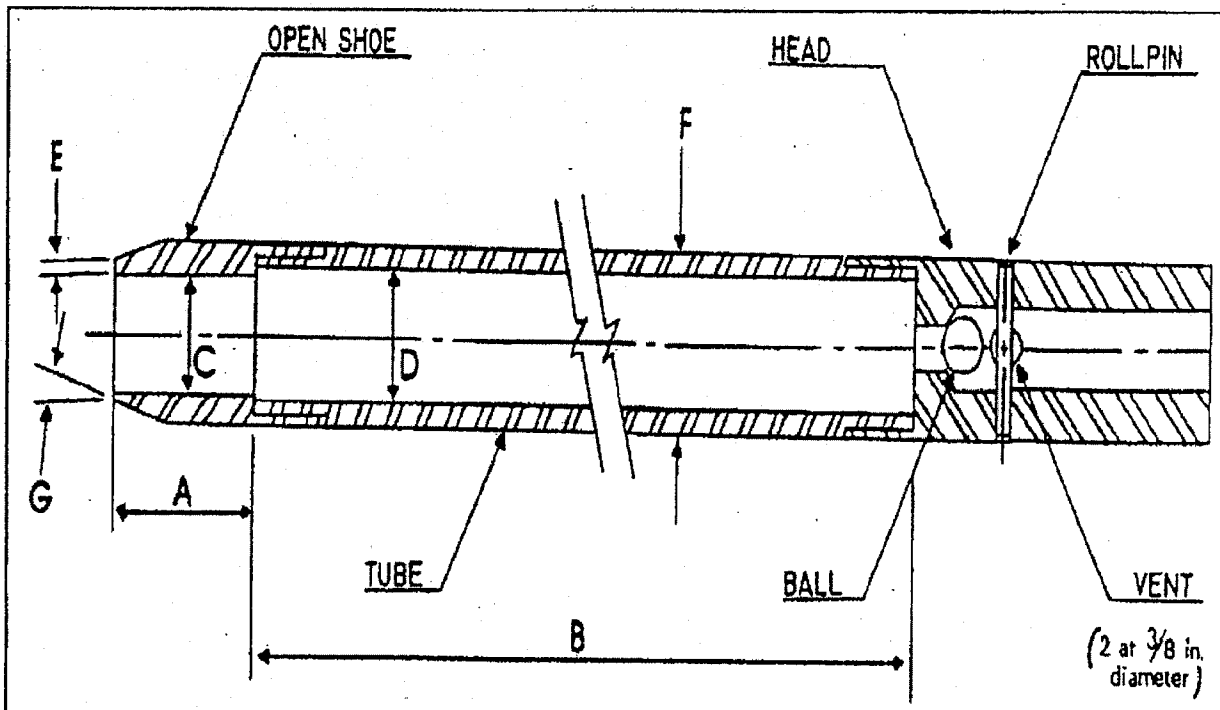
MAP:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

ATTACHMENT B
SPLIT-SPOON SAMPLER



A = 1.0 to 2.0 in. (25 to 50 mm)
B = 18.0 to 30.0 in. (0.457 to 0.762 m)
C = 1.375 ± 0.005 in. (34.93 ± 0.13 mm)
D = 1.50 ± 0.05 – 0.00 in. (38.1 ± 1.3 – 0.0 mm)
E = 0.10 ± 0.02 in. (2.54 ± 0.25 mm)
F = 2.00 ± 0.05 – 0.00 in. (50.8 ± 1.3 – 0.0 mm)
G = 16.0° to 23.0°

The 1½ in. (38 mm) inside diameter split barrel may be used with a 16-gage wall thickness split liner. The penetrating end of the drive shoe may be slightly rounded. Metal or plastic retainers may be used to retain soil samples.

ATTACHMENT C
TEST PIT LOG

Tetra Tech NUS, Inc.

TEST PIT LOG

Page ___ of ___

PROJECT NAME: _____ TEST PIT No.: _____
PROJECT NUMBER: _____ DATE: _____
LOCATION: _____ GEOLOGIST: _____

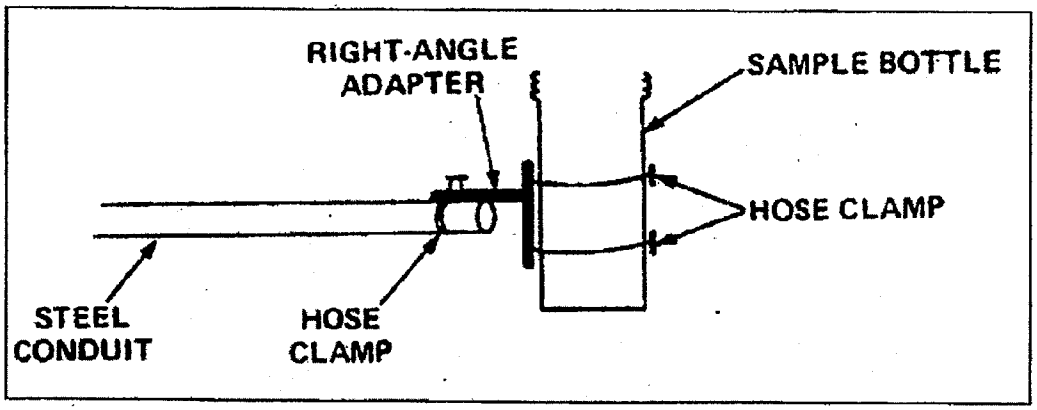
Depth (Ft.)	Lithology Change (Depth/Ft.)	MATERIAL DESCRIPTION Soil/Waste Characteristics (lithology, density, color, etc.)	USCS	Remarks	PID/FID READING Source (ppm)	BZ (ppm)

TEST PIT CROSS SECTION AND / OR PLAN VIEW

REMARKS: _____

PHOTO LOG: _____

ATTACHMENT D
REMOTE SAMPLE HOLDER FOR TEST PIT/TRENCH SAMPLING



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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 (ÿ261.30 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 (ÿ261.20 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 - H_2SO_4
- Nitric Acid - HNO_3
- Sodium Hydroxide - NaOH

Other Preservatives

- Zinc Acetate
- Sodium Thiosulfate - $Na_2S_2O_3$

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

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 HNO_3, 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_2SO_4)	1 part concentrated H_2SO_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:

- 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).

- 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.

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.

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 (Low SVOCs and pesticide/PCBs)	Amber glass	2x2 L or 4x1 L	Cool to 4°C	7 days to extraction; 40 days after extraction
	Extractables (Medium SVOCs and pesticide/PCBs)	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 (Low SVOCs and pesticides/PCBs)	Wide-mouth glass	8 oz.	Cool to 4°C	14 days to extraction; 40 days after extraction
	Extractables (Medium SVOCs and pesticides/PCBs)	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/Inorganic	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.

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_2SO_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_2SO_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_2SO_4 to pH 2	6 months
Total Kjeldahl and Organic Nitrogen	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Nitrate - Nitrogen	P, G	None required	48 hours
Nitrate-Nitrite - Nitrogen	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Nitrite - Nitrogen	P, G	Cool, 4°C	48 hours
Oil & Grease	G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Total Organic Carbon (TOC)	P, G	Cool, 4°C; HCl or H_2SO_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_2SO_4 to pH 2	28 days
Phosphorus, Total	P, G	Cool, 4°C; H_2SO_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

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% $Na_2S_2O_3$[5]	14 days
Purgeable Aromatic Hydrocarbons	G, Teflon-lined septum	Cool, 4°C; 0.008% $Na_2S_2O_3$[5] HCl to pH 2 [9]	14 days
Acrolein and Acrylonitrile	G, Teflon-lined septum	Cool, 4°C; 0.008% $Na_2S_2O_3$[5] adjust pH to 4-5 [10]	14 days
Phenols[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
Benzidines[11],[12]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[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% $Na_2S_2O_3$[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% $Na_2S_2O_3$[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% $Na_2S_2O_3$[5]; store in dark	7 days until extraction; 40 days after extraction
Haloethers[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
Dioxin/Furan (TCDD/TCDF)[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction

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-diphenylthydrazine 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% $Na_2S_2O_3$ 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% $Na_2S_2O_3$.

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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

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.

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.

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.

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 colleted, 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.

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

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

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 jenney 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

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:**	

ATTACHMENT C

TETRA TECH NUS, INC. CHAIN OF CUSTODY | NUMBER 3413 | PAGE ___ OF ___

PROJECT NO:	FACILITY:	PROJECT MANAGER	PHONE NUMBER	LABORATORY NAME AND CONTACT:
SAMPLERS (SIGNATURE)		FIELD OPERATIONS LEADER	PHONE NUMBER	ADDRESS
		CARRIER/WAYBILL NUMBER		CITY, STATE

STANDARD TAT ☐
RUSH TAT ☐
☐ 24 hr. ☐ 48 hr. ☐ 72 hr. ☐ 7 day ☐ 14 day

CONTAINER TYPE PLASTIC (P) or GLASS (G)
PRESERVATIVE USED
TYPE OF ANALYSIS

DATE YEAR	TIME	SAMPLE ID	LOCATION ID	TOP DEPTH (FT)	BOTTOM DEPTH (FT)	MATRIX (GW, SO, SW, SD, QC, ETC.)	COLLECTION METHOD GRAP (G) COMP (C)	No. OF CONTAINERS									COMMENTS

1. RELINQUISHED BY	DATE	TIME	1. RECEIVED BY	DATE	TIME
2. RELINQUISHED BY	DATE	TIME	2. RECEIVED BY	DATE	TIME
3. RELINQUISHED BY	DATE	TIME	3. RECEIVED BY	DATE	TIME
COMMENTS					

DISTRIBUTION: WHITE (ACCOMPANIES SAMPLE) YELLOW (FIELD COPY) PINK (FILE COPY)

4/02R
FORM NO. TtNUS-001

ATTACHMENT D

CHAIN-OF-CUSTODY SEAL

CUSTODY SEAL	**CUSTODY SEAL**
Date	**Date**
Signature	**Signature**

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A INVESTIGATION-DERIVED WASTE LABEL 15

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.

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).
- 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
- 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.

- 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.

  - 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.
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.

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).

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.

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.

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.
   - Where possible, use equipment for moving containers. Where not possible, obtain help to manipulate containers.

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

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.

Attachment A
DW Label

INVESTIGATION DERIVED WASTE

GENERATOR INFORMATION:

SITE _____ JOB NO. _____

LOCATION _____

DATE _____

DRUM# _____

CONTENTS _____

VOLUME _____

CONTACT _____

EMERGENCY PHONE NUMBER _____

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1.0 PURPOSE

To establish procedures for the use, maintenance, and calibration of the Photovac 2020 Photoionization Air Monitor.

2.0 SCOPE

Applies to each usage of the Photovac 2020 Photoionization Air Monitor by TtNUS personnel.

3.0 GLOSSARY

Electron volt (eV) - A unit of energy equal to the energy acquired by an electron when it passes through a potential difference of 1 volt in a vacuum. It is equal to 1.602192∀0.000007 x 10^{-19} volts.

Intrinsically Safe (I.S.) - Based on wiring, configuration, design, operation, gasketing, construction, this instrument may be employed within locations in which flammable gases and/or vapors may exist.

Ionization Potential (I.P.) - The energy required to remove an electron from a molecule yielding a positively charged ion and a negatively charged free electron. The instrument measures this energy level.

Photoionization Detector (PID) - Photoionization detector employed as general reference to air monitors of this type. PIDs detection method employs ultraviolet (UV) radiation as an energy source. As air and contaminant are drawn through the ionization chamber the UV light source causes the contaminant with ionization potentials equal to or less than the UV source to break into positive and negatively charge ions. The created ions are subjected to an electrostatic field. The voltage difference is measured in proportion to the calibration reference and the concentration of the contaminant.

Ultraviolet Radiation (UV) - Ultraviolet radiation is the energy source employed by the instrument to ionize collected sample gas streams. The UV lamp source is required to be equal to or greater than the ionization potential of the substance drawn through the instrument in order to create separate ionized species.

4.0 RESPONSIBILITIES

Office Managers – Office Managers are responsible for ensuring that personnel under their direction who may use this device are first provided with adequate training and information.

Project Managers – Project Managers are responsible for ensuring that appropriate health and safety requirements and resources are addressed for their assigned projects.

Health and Safety Manager (HSM) - The HSM shall ensure that appropriate training is available to users of the Photovac 2020 instrument.

Equipment Manager - The Equipment Manager shall ensure all air monitoring instrumentation slated for field activities has been operationally checked out, fully charged, and calibrated prior to issuing any instrument for field service. Maintenance deficiencies identified by the Equipment Manager will require those instruments to be pulled from service until repairs can be facilitated.

Field Operations Leader (FOL)/Field Team Leader (FTL) - The FOL/FTL shall ensure all field team members employing the monitoring instruments as part of their assigned duties are adequately trained in the operation and limitations of this instrument. The FOL/FTL shall ensure that the air monitoring instruments are employed as directed by site guidance documents (i.e., Work Plan, Health and Safety Plan, etc.). Additionally, the FOL/FTL shall ensure that the appropriate documentation and recordkeeping

requirements are fulfilled including Documentation of Calibration and Direct Reading Instrument Response Data Sheets for air monitoring activities. On projects where a dedicated SSO is not assigned, the FOL/FTL is responsible for assuming the duties of that position.

Health and Safety Officer (HSO) - The HSO is responsible for determining air monitoring requirements for the site activities, and providing direction for air monitoring during specific site activities. This identification of types of air monitoring and direction for use are indicated within the Site-Specific Health and Safety Plan (HASP).

Site Safety Officer (SSO) - The SSO shall ensure the instruments identified are employed in the manner directed by the HSO, and that any action levels specified are observed for the application of engineering controls, personal protective equipment (PPE) use, and administrative controls. Additionally, he/she shall ensure the instruments are properly maintained and calibrated prior to use in the field. The SSO, during specific air monitoring applications including STEL and TWA mode measurements, will be responsible for the operation and application of this specialty air monitoring device. The SSO is also responsible for addressing relevant Hazard Communication requirements (e.g., MSDS, chemical inventories, labeling, training, etc.) on each assigned project.

5.0 PROCEDURES

5.1 Principle of Operation

Direct-reading instruments such as a photoionization detector are typically used to monitor for airborne releases that could present an inhalation threat to personnel, and to screen and bias environmental samples. Proper use of these instruments by trained, qualified personnel is essential to the validity of any acquired results. Also essential is that the devices are properly calibrated according to manufacturers instructions (and the specifications of this SOP), and that users of the instrument properly document results.

The Photovac portable photoionizer detects many organic (and a few inorganic) vapors. The basis for detection of this instrument is the ionization of components of captured gaseous streams. The incoming gas molecules are subjected to ultraviolet (UV) radiation, which is energetic enough to ionize many gaseous compounds. Molecules are transformed into charged-ion pairs, creating a current between two electrodes. Each molecule has a characteristic ionization potential, which is the energy required to remove an electron from the molecule, yielding a positively-charged ion and the free electron. The instrument measures this energy level.

This instrument measures the concentration of airborne photoionizable gases and vapors and automatically displays and records these concentrations. It does not distinguish between individual substances. Readings displayed represent the total concentration of all photoionizable chemicals present in the sample. This instrument is factory set to display concentration in units of ppm or mg/m^3.

The 2020 instrument is easy to operate. The meter display updates itself once per second. Concentrations are directly displayed on the readout.

The 2020 instrument also performs short-term exposure limit (STEL), time-weighted average (TWA) and PEAK calculations. Any of these results can be viewed, but only one mode may be viewed at a time.

2020 has 6 keys for alphanumeric entry and for accessing multiple functions. The keys are used to set up and calibrate the 2020 instrument. They allow for the manipulation of the data in various ways.

All information entered with the keys and stored in the instrument's memory. This is retained when the instrument is switched off. The clock and calendar continue to operate and do not need to be set each time the instrument is turned on.

5.1.1 Displays

The 2020 instrument has a meter display for reporting detected concentration, and a display used to indicate status information and guide the user through configuration options. All functions of the instrument will be controlled or reported using one of these displays.

5.1.1.1 Meter Display

The meter has a 4-digit display. It will always be used for reporting detected concentrations. When the detector and pump are off, the meter display will be blank.

In order to accommodate the entire range of concentrations the 2020 can detect, the instrument has 2 resolution settings. The 0.1 resolution setting should be used for concentrations below 100 ppm, and the 1 resolution setting should be used for concentrations above 100 ppm.

5.1.1.2 Status Display

The status display is a 2 line by 16 character display. The top line is used to display status information and prompts the user for information. The bottom line is used for soft key names. Up to 3 names can be displayed for the 3 soft keys. If a name does not appear for a soft key, then the soft key has no associated function.

5.1.2 Keys

5.1.2.1 Fixed Keys

The three round keys below the soft keys each have a fixed function. The first key is the ON/OFF key, the middle key is the EXIT key, and the last key is the ENTER key.

The ON/OFF key is used both to turn power on to the 2020 as well as to turn the power off. To turn on 2020, press the ON/OFF key. To turn the power off, press the ON/OFF key and hold it down for 2 seconds, and then release it. This is done to prevent an accidental power off.

The EXIT key provides a way of returning to the default display. In the functional map, the soft keys allow the user to advance and the EXIT key provides a way to go back. At the initial entry of the menu, EXIT will return the user to the default display.

The ENTER key has a context sensitive function. When operating or navigating through the function map, the ENTER key is used to exit the functions and return to the default display. When entering data such as a name, number, date, or time, ENTER is used to confirm the entry.

5.1.2.2 Soft Keys

The three soft keys on the 2020 are located directly below the status display. Each key has varying functions for configuring the 2020, editing the data logger, and controlling the display. Since only three soft keys are available, each function is broken down into a path.

5.1.2.3 Entering Text With the Soft Keys

For all information that must be entered, the left, center, and right soft keys correspond to the up, down, and right arrow.

The up and down arrows are used to change the character highlighted by the cursor. The right arrow is used to advance the cursor to the next character to the right. When the cursor is advanced past the right most character, it wraps around to the first character again. To accept the changes, press the ENTER key. To ignore the change, press the EXIT key.

Formatting characters, such as the colon (:) in the time, the decimal (.) in a concentration, and the slash (/) in the date are skipped when advancing the cursor.

All inputs 8 characters long, which is displayed on the right side of the status display line. The prompt, describing the input, occupies the left side of the top line. The soft keys are defined on the bottom line of the status display.

5.2 Default Display

The meter display shows the detected concentration. The resolution of the display automatically changes with the magnitude of the reading. A reading of 0 to 99.9 will be displayed with a resolution of 0.1 ppm or mg/m^3. A reading greater than 99.9 will be shown with a resolution of 1 ppm or mg/m^3. The meter will display concentrations up to 2000 ppm or 2(XX) mg/m^3.

The status display is used to indicate the instrument status, date, time, units, and active soft keys.

The default display provides the following information: instrument status, current detected concentration, time, date, and measurement units. The status display toggles between showing time and units and then the date.

When the display mode is MAX, the date and time correspond to the date and time the MAX concentration was recorded. In TWA mode, the time represents the number of hours and minutes during which the TWA has been accumulating. For PEAK and STEL monitoring, the date and time correspond to the current date and time.

5.3 Monitoring

5.3.1 Use and Documentation of Results

As with any direct-reading instrument, understanding not only how – but when to use this instrument is essential to gathering relevant and valid data. This device will only respond to volatile substances in sampled air that have ionization potentials below the UV lamp strength. Inappropriate instrument selection, or use/interpretation of instrument results by an unqualified user not only can yield inaccurate results, but could place personnel at risk of exposure to hazardous agents. Only personnel who are properly trained and authorized to use this device will be permitted to operate it.

It is essential that instrument operators understand and comply with the requirements to document results. This includes the need to document calibration results as well as operational readings. Calibration results must be recorded using Figure 5-1. Operational results can be recorded in several ways, including:

- Direct-Reading Instrument Response Data (Figure 5-2) – preferred method
- Boring Log Forms (Figure 5-3)
- Test Pit Log Forms (Figure 5-4)

- Log book entries

When using direct-reading instruments, it is important to monitor the air near the source of potential releases (e.g., drilling boreholes, tank entrances, drum openings, etc.) and at worker breathing zone areas. All readings should be recorded, including readings noted where background levels were not exceeded.

5.3.2 Instrument Status

The instrument status is shown on the left side of the first line of the status display and on the Table and Graph outputs. Each status has an assigned priority assigned to it. If more than one status is in effect, then the status with the highest priority is displayed until the condition is corrected or until the option is turned off.

5.3.3 Alarms

While operating the instrument, any one of three audible alarm conditions can occur. To accurately identify the source of the alarm, each type of alarm has been given a unique status.

The 2020 also has an audible alarm and a visual alarm LED. To conserve power, the 2020 alternates between these two alarm indicators, rather than operating both concurrently. Different alarms are identified by the frequency at which the 2020 alternates as follows: PEAK alarm-5 times per second; STEL alarm-2.5 times per second; and TWA alarm-1.25 times per second.

The left soft key is used for acknowledging alarm conditions, and is named "Ack." If no alarm conditions exist , then the "Ack" key is not shown. To clear an alarm, press the "Ack" key. Once acknowledged, the alarm indicators are cleared. The alarm status will remain until the alarm condition clears.

The 2020 updates the peak concentration once every second. Following every update, the peak concentration is compared to the peak alarm level, and if exceeded, an alarm is triggered.

If the 15 minute average exceeds the selected STEL, a STEL alarm is generated.

The TWA alarm is generated when the current average of concentration, since the TWA was last cleared, has exceeded the TWA exposure limit.

FIGURE 5-1

DOCUMENTATION OF FIELD CALIBRATION

SITE NAME: _____ PROJECT NO.: _____

Date of Calibration	Instrument Name and Model	Tetra Tech Instrument I.D. Number	Person Performing Calibration	Instrument Settings/Readings		Calibration Standard (Lot Number)	Remarks/ Comments
				Pre-Calibration Setting	Post-Calibration Reading		

FIGURE 5-2

DIRECT-READING INSTRUMENT RESPONSE DATA

SITE NAME: _____

PROJECT NO.:_____

PAGE OF

Date and Time(s)	Instrument Operator Name	Instrument Used	Instrument Reading and Duration	Location (e.g., borehole vs breathing zone)	Operation(s) Being Performed	Personnel in Vicinity at Time of Readings and Level of PPE Worn
Example 12-12-91/14:30	Joe Field	HNU 101	15 ppm/15 min.	breathing zone	sample location no. 12	Jim Technician/Level B George Grunt/Level B

FIGURE 5-2 (Continued)

SITE NAME: _____

PROJECT NO.: _____

PAGE _____ OF ____

Date and Time(s)	Instrument Operator Name	Instrument Used	Instrument Reading and Duration	Location (e.g., borehole vs breathing zone)	Operation(s) Being Performed	Personnel in Vicinity at Time of Readings and Level of PPE Worn

FIGURE 5-3

BORING LOG

Page ___ of ___

PROJECT NAME: _____ BORING NUMBER: _____
PROJECT NUMBER: _____ DATE: _____
DRILLING COMPANY: _____ GEOLOGIST: _____
DRILLING RIG: _____ DRILLER: _____

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: Soil Density/ Consistency or Rock Hardness	Color	Material Classification	USCS *	Remarks	PID/FID Reading (ppm): Sample	Sampler BZ	Borehole**	Driller BZ**

* When rock coring, enter rock brokeness.

** Include monitor reading in 6 foot intervals @ borehole. Increase reading frequency if elevated response read.

Remarks: _____

Drilling Area Background (ppm): [ ]

Converted to Well: Yes _____ No _____ Well I.D. #: _____

FIGURE 5-4

Page ___ of ___

TEST PIT LOG

PROJECT NAME: _____ TEST PIT NUMBER: _____
PROJECT NUMBER: _____ DATE: _____
LOCATION: _____ GEOLOGIST: _____

Depth (Ft.)	Lithology Change (Depth/Ft.)	MATERIAL DESCRIPTION Soil/Waste Characteristics (lithology, density, color, etc.)	USCS	Remarks	PID/FID READING Source (ppm)	BZ (ppm)

TEST PIT CROSS SECTION AND / OR PLAN VIEW

REMARKS: _____

PHOTO LOG: _____

During calibration, all alarms are disabled. Once the calibration is complete the alarms are re-enabled.

5.4 STEL, TWA, MAX, and PEAK Operation

The 2020's display can be configured to show one of four values: STEL, TWA, PEAK, and MAX.

5.4.1 Short-term Exposure Limit (STEL) Mode

The Short-term Exposure Limit (STEL) mode displays the concentration as a 15 minute moving average. The 2020 maintains 15 samples, each representing a one-minute averaging interval.

Once every minute, the oldest of the 15 samples is replaced with a new one minute average. This moving average provides a 15-minute average of the last 15 minutes with a one-minute update rate. Since the average is calculated using 15 one-minute averages, the meter display will only update once every minute.

STEL is set to zero each time the instrument is turned on. Since STEL is a 15-minute moving average, there is no need to clear or reset the STEL.

STEL calculations are always being performed by the 2020. The results of the calculations can be displayed by selecting STEL as the Display mode.

5.4.2 Time-weighted Average (TWA) Mode

The TWA accumulator sums concentrations every second until 8 hours of data have been combined. If this value exceeds the TWA alarm setting, a TWA alarm is generated. The TWA is not calculated using a moving average. Once 8 hours of data have been summed, the accumulation stops. In order to reset the TWA accumulator, press the "Clr" key.

This sum will only be complete after 8 hours, so the meter displays the current sum divided by 8 hours. While in the TWA mode, the time on the status display will show the number of minutes and hours of data that TWA data has been accumulated. When the sample time reaches 8 hours, the 2020 stops accumulating data and the TWA is complete.

TWA calculations are always being performed by the 2020. The results of the calculations can be displayed by selecting TWA as the Display mode. When the sampling period is less than 8 hours, record the TWA readout along with the sampling duration displayed on the meter.

5.4.3 MAX Mode

The MAX mode displays the maximum signal, with the date and time that it was recorded. 2020 continues to log data according to the selected averaging interval, but only the maximum detected concentration is shown on the meter display.

The right soft key is used to clear the meter when displaying MAX. The "Clr" key only affects the reading that the meter is displaying. For example, if you display the MAX reading, and you press "Clr," only the MAX value is cleared. The TWA is still accumulating in the background.

5.4.4 PEAK Mode

The PEAK mode displays the current detected concentration. The reading is updated every second. In the background, the 2020 data logger is sampling the concentration and measuring minimum, maximum,

and average concentrations for the selected averaging interval. At the end of every interval, one entry is placed in the data logger until the data logger is full. Typically, the instrument is operated in the PEAK mode. Operation within the other specialized modes are the responsibility of the SSO.

5.5 Set Functions

Pre-set functions are used to setup the 2020. There are three functions which can be used: Calibration, Pump and Clock.

5.5.1 Pump

The Pump function is used to control the pump. After selecting Set Pump, the 2020 responds by displaying the new pump status.

The detector is also turned off when turning the pump off. This prevents the detector from being damaged when there is no sample flowing through the detector.

When the pump and the detector are off, the meter display will be blank. Turn the pump and detector off when concentration measurements are not necessary, and the 2020 will only be used for reviewing data or generating reports. Operating the instrument with the pump and detector turned off will conserve the lives of the battery and ultraviolet (UV) lamp.

1. Press the ENTER key. The top line of the status display changes to "Select?". The bottom line displays 3 soft key names: "Set," "Log," and "Disp."

2. Press the soft key below "Set."

3. The names of the soft keys change to reflect the Set options. The display now shows 3 devices which can be set: "Clock," "Pump," and "Cal." Press the "Pump" key.

4. If the pump is off, pressing the "Pump" key will turn the pump on.

5. A message will be displayed showing the status of the pump. The display reverts back to the previous menu after a few seconds.

6. To return to the default display, press the ENTER key.

5.5.2 Clock

The Clock function is used to set both the current date and time.

1. Press the ENTER key.

2. Press the "Set" key.

3. When the names of the soft keys change, press the "Clock" key.

   The up and down arrows are used to change the character underlined by the cursor. The right arrow is used to advance the cursor to the next character to the right. When the cursor is advanced past the right-most character, it wraps around to the first character again.

   Formatting characters, such as the colon (:) in the time and the slash (/) in the date are skipped when advancing the cursor.

4. Use the "arrow keys" to enter the correct time. The time is formatted as Hour:Minute:Second.

5. Press the ENTER key to confirm the time and move to the date option.

6. When setting the date, the 2020 prompts the user to input the current date formatted as Year/Month/Day. Use the "arrow keys" to enter the correct date.

7. Press the ENTER key to confirm the date and return to the Set options.

5.5.3 Calibration (Cal)

The Cal function allows the user to setup and calibrate the 2020. Three options are available under the Cal function: "Zero," Span," and "Mem."

A calibration memory consists of a name, a response factor, and PEAK, TWA, and STEL alarm levels.

The "Zero" and "Span" keys are covered in detail in the manufacturer's operations manual for the instrument.

To edit the calibration memory, select "Mem" and then "Chng." The 2020 prompts the user with two new soft keys: "User" and "Lib."

5.5.4 Library (Lib)

Library selections simplify Cal Memory programming, and provide standard response factors for approximately 70 applications. "Lib" allows you to select an entry from a pre-programmed library. The name, response factor, and three alarm levels are all set from the library. To select a library entry to program the selected Cal Memory:

1. Select "Set," "Cal," "Mem," "Chng," and "Lib."

2. Use the "Next" and "Prev" keys to scroll through the list. See the manufacturer's manual for a list of the library entries.

5.6 Preparing for Field Operation of the Photovac 2020

Turning 2020 On

1. Turn the 2020 on by pressing the ON/OFF key.

2. The instrument will display the software version number. Wait for the 2020 to proceed to the default display.

3. Allow 10 minutes for the instrument to warm up and stabilize.

4. Press the "Enter" Key. The default display will provide 3 soft key selections: "Set," "Log," and "Display."

5. Press "Set." From this option 3 other soft key selections will be offered: "Pump," "Clock," and "Cal."

6. Press "Cal." This will begin the calibration sequence. The first selection is to Zero the instrument.

7. Press "Enter," zeroing will begin. (Note: When employing zero gas attach and activate zero gas supply at this time.)

8. The next selection is for Span determination. Press "Enter" and the concentration will be requested. The isobutylene calibration gas employed under general service will be marked on the side of the container. Use the soft keys to toggle into position and to log the concentration. Once the concentration is logged press "Enter." The direction or status display will indicate spanning. At this time hook up the span gas with a regulator to the Photovac 2020, and open it to supply enough flow to elevate the flow rate indicator to the green indicator line (1/8" from the rest position).

9. Once spanning is complete, the alarms which have been disabled during calibration will activate indicating that calibration is complete.

10. Document this calibration procedure using a Documentation of Calibration form as illustrated in Figure 5-1.

This instrument is ready for use.

Calibration is to be performed daily or prior to each use in accordance with Sections 5.6 and 5.7 of this SOP, and with manufacturer's recommendations.

5.7 Maintenance and Calibration Schedule

Function	Frequency
Routine Calibration	Prior to each use. Complete Figure 5-4 for each calibration.
Factory Inspection and Calibration	Once a year, or when malfunctioning
Wipe Down the Outer Casing of the Unit	After each use
Clean UV Light Source	Every 24 hours of operation
Sample Inlet Filter	Change on a weekly basis or as required by level of use
Battery charging	After each use
Clean ionization chamber	Monthly

5.7.1 Cleaning the UV Light Source Window

1. Turn the FUNCTION switch to the OFF position. Use the instrument's multi-tool and remove lamp housing cover.

2. Tilt the lamp housing with one hand over the opening, slide the lamp out of the housing.

3. The lamp window may now be cleaned with any of the following compounds using lens paper:

   a. 11.7 eV Lamp - Dry Aluminum Oxide Powder (3.0 micron powder)
   b. All other lamps - HPLC Grade Methanol

   Observe manufacturer's MSDS requirements when handling these substances.

4. Following cleaning, reassemble by first sliding the lamp back into the lamp housing. Replace o-ring as necessary, reinstall lamp housing cover, tighten using the multi-tool. (Do not over tighten).

5. Recalibrate as per Section 5.6.

5.7.2 Cleaning the Ionization Chamber

1. Turn the FUNCTION switch to the OFF position and remove the lamp housing cover and lamp as per Section 5.7.1.

2. Using a gentle jet of compressed air, gently blow out any dust or debris.

3. Following cleaning, reassemble by first sliding the lamp back into the lamp housing. Replace o-ring as necessary, reinstall lamp housing cover, tighten using the multi-tool. (Do not over tighten).

4. Recalibrate as per Sections 5.6 and 5.7.

5.8 Instrument Advantages

The Photovac 2020 is easy to use in comparison to many other types of monitoring instrumentation. Its detection limit is in the low parts-per-million range. Response time quickly reaches 90 percent scale of the indicated concentration (less than 3 seconds for benzene). This instrument's automated performance covers multiple monitoring functions simultaneously, and incorporates data logging capabilities.

5.9 Limitations of the Photovac 2020 Photoionization Monitor

- Since the 2020 is a nonspecific total gas/vapor detector, it cannot be used to identify unknown chemicals; it can only quantitate them in relationship to a calibration standard (relative response ratio).

- For appropriate application of the 2020, ionization potentials of suspected contaminants must be known.

- Because the types of compounds that the 2020 can potentially detect are only a fraction of the chemicals possibly present at a hazardous waste site, a background or zero reading on this instrument does not necessarily signify the absence of air contaminants.

- The 2020 instrument can monitor only certain vapors and gases in air. Many nonvolatile liquids, toxic solids, particulates, and other toxic gases and vapors cannot be detected.

- PID's are generally not compound-specific. Their response to different compounds is relative to the calibration gas used. This is generally referred to as the relative response ratio. Instrument readings may be higher or lower than the true concentration. This can be especially serious when monitoring for total contaminant concentrations if several different compounds are being detected at once.

- The 2020 is a small, portable instrument which cannot be expected to yield results as accurately as laboratory instruments.

5.9.1 Variables Affecting Monitoring Data

Monitoring hazardous waste site environments can pose a significant challenge in assessing airborne concentrations and the potential threats to site personnel. Several variables may influence both dispersion and the instrument's ability to detect actual concentrations. Some of the variables which may impact these conditions are as follows:

- Temperature Pressure - changes in temperature and/or pressure will influence volatization, and effect airborne concentrations. Additionally, an increase or decrease in temperature ranges may have an adverse effect on the instrument's ability to detect airborne concentrations. Significant changes in temperature or pressure from the time of calibration to the time of sample measurement may result in erroneous results.

- Humidity - excessive levels of humidity may interfere with the accuracy of monitoring results.

- Rainfall - through increased barometric pressure and water may influence dispersion pathways effecting airborne emissions.

- Electromagnetic interference - high voltage sources, generators, other electrical equipment may interfere with the operation and accuracy of direct-reading monitoring instruments.

6.0 TROUBLESHOOTING

6.1 Fault Messages

When the "Fault" status is displayed, the 2020's operation is comprised.

Fault 1: Signal from zero gas is too high.

Cause: If another fault occurred while the 2020 was setting its zero point, then this fault is displayed.

Action: Ensure no faults are occurring and calibrate the 2020 again.

Cause: Contamination of sample line, sample probe or fittings before the detector.

Action: Clean or replace the sample line, sample probe or the inlet filter.

Cause: Span gas and zero air are mixed up.

Action: Ensure clean air is used to zero the 2020. If you are using Tedlar bags, mark the calibration and zero gas Tedlar bags clearly.

Cause: Ambient air is contaminated.

Action: If the quality of ambient air is unknown, use a supply of commercial zero grade air.

Fault 2: Signal from span gas is too small.

Cause: Operator may have incorrectly used the span gas for the zero air source.

Action: Ensure clean air is used to zero the 2020. If you are using Tedlar bags, mark the calibration and zero gas Tedlar bags clearly.

Action: Ensure the span gas is of a reliable concentration.

Cause: UV lamp window is dirty.

Note: Do not remove the detector lamp in a hazardous location.

Action: Clean the UV lamp window.

Cause: UV lamp is failing.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: Install a new UV lamp.

Cause: Incompatible application.

Action: The concentration and sample gas are incompatible for use with the 2020.

Fault 3: UV lamp fault. UV lamp has not started.

Cause: UV lamp has not started immediately.

Action: This fault may be seen momentarily when the 2020 is first turned on. Allow 30 to 60 seconds for the UV lamp to start and the fault to clear.

Cause: UV lamp serial number label is blocking the photocell.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: The UV lamp has a white serial number label, it is possible that the label is blocking the photocell. Rotate the lamp approximately 90 degree and then try to start the 2020 again. If the fault persists, replace the lamp.

Cause: UV lamp not installed.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: Install a UV lamp.

Cause: UV lamp has failed.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: Install a new UV lamp.

Cause: Electronic problem.

Action: If a new UV lamp still generates this fault, then contact the Photovac Service Department.

Fault 4: Pump current too low or too high.

Cause: If the pump sounds labored, then the pump is operating beyond normal operating parameters.

Action: Check for an obstruction in the sample line. Make sure sample line, sample probe or inlet filter are not plugged.

Note: Do not replace the inlet filter in a hazardous location.

Action: Replace the inlet filter.

Action: Ensure the sample outlet, located on the underside of the 2020, is not obstructed.

Cause: UV lamp is too wide, causing flow to be restricted.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: If the UV lamp has a white serial number label, it is possible that the lamp is too wide for the lampholder. Contact the Equipment Manager.

Cause: The 2020 has been exposed to a solvent that can pass through the inlet filter and liquid has been aspirated.

Action: Contact the Equipment Manager.

Cause: The pump has failed.

Action: Contact the Equipment Manager.

6.2 Specific Problems

Problem: Very low or no instrument response detected, yet compounds are suspected to be present.

Cause: The 2020 has not been calibrated properly.

Action: Ensure the calibration gas is of a reliable concentration and then calibrate the instrument as outlined in the User's Manual.

After the instrument has been calibrated, sample the bag of calibration gas. A reading equivalent to the calibration gas should be displayed. If not, contact the Equipment Manager.

Note: Do not remove or recharge the battery pack in a hazardous location.

Action: Disconnect the battery charger before calibrating the 2020.

Cause: Calibration Memories have not been programmed correctly.

Action: Program all the calibration memories required for the intended application. You must use the correct calibration gas and concentration for each Cal Memory.

Cause: Response factor has been set to zero.

Action: Enter the correct response factor. Refer to the list of response factors. If the compound is not listed or you are measuring gas mixtures, then enter a value of 1.0. See User's Manual.

Cause: Not using the correct Cal Memory.

Action: Select the correct Cal Memory for the intended application.

Note: It does not matter which Cal Memory is selected or which response factor is entered. The 2020's response is not specific to any one compound. The reading displayed represents the total concentration of all ionizable compounds in the sample.

Cause: Detector is leaking. A decrease in sensitivity may be due to a leak in the detector.

Note: Do not remove or replace the detection lamp in a hazardous location.

Action: Ensure the UV lamp has been installed correctly.

Action: Ensure the lamp cover has been tightened. Do not overtighten the cover.

Action: Ensure the o-ring seal on the lamp cover is positioned correctly.

Cause: UV lamp is too long, causing flow to be restricted.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: If the UV lamp has a white serial number label, it is possible that the lamp is too long for the lampholder. Replace the lamp and contact the Equipment Manager.

Cause: UV lamp is too wide, causing flow to be restricted.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: If the UV lamp has a white serial number label, it is possible that the lamp is too wide for the lampholder. Contact the Equipment Manager.

Cause: Sampling environment is extremely humid.

Action: Water vapor is not ionized by the PID, but it does scatter and absorb the light and results in a lower reading.

The 2020 detector has been designed to operate under high humidity conditions. Under extreme humidity conditions, there may be a decreased response.

Cause: UV lamp is failing.

Action: Replace UV lamp.

Note: Do not remove or replace the detector lamp in a hazardous location.

Cause: High concentration of non-ionizable compounds suspected.

Action: Chemical compounds, such as methane, with IPs greater than the 10.6 eV scatter and absorb the UV light. Sensitivity may be decreased significantly.

Application with high backgrounds of such materials, may be incompatible with the 2020. Contact the Photovac Applications Group for more information.

Problem: Erroneously high readings.

Cause: Sampling environment is extremely humid.

Action: Water vapor may contain mineral salts which conduct a charge. The water vapor becomes an electrolytic solution which becomes ionized when it enters the detector.

Atmospheric water in areas around the sea or stagnant water may produce a response in the absence of contaminants. The same effect may be seen when conducting ground water investigations in areas where the water contains a significant concentration of minerals.

Cause: The 2020 has not been calibrated properly.

Action: Ensure the calibration gas is of a reliable concentration and then calibrate the instrument as outlined in Sections 5.6 and 5.7.

After the instrument has been calibrated, sample the bag of calibration gas. A reading equivalent to the calibration gas should be displayed. If not contact the Equipment Manager.

Cause: Cal Memories have not been programmed correctly.

Action: Program all the Cal Memories required for the intended application. The correct calibration gas and concentration must be used for each Cal Memory. See the User's Manual.

Cause: Not using the correct Cal Memory.

Action: Select the correct Cal Memory for the intended application. See the User's Manual.

Note: It does not matter which Cal Memory is selected or which response factor is entered. The 2020's response is not specific to any one compound. The reading displayed represents the total concentration of all ionizable compounds in the sample.

Cause: Detector has been short circuited by foreign matter in the detector cell.

Note: Do not service the 2020 in a hazardous location.

Action: Do not touch the wire grid inside the detector cell. Use a gentle jet of compressed air to remove any dust in the detector cell.

Warning: Do not insert any object, other than the UV lamp, into the lampholder.

Cause: There is an undetermined problem.

Action: Contact the Equipment Manager.

Problem: Date and time settings are not retained.

Cause: The battery pack was removed before the 2020 was turned off.

Note: Do not remove or recharge the battery pack in a hazardous location.

Action: Replace the battery pack and reset the time and date. Ensure that the 2020 has been turned off before removing the battery pack.

Cause: The 2020 has not been used for 3 months or more and the internal battery (not the external battery pack) has discharged.

Note: Do not remove or recharge the battery pack in a hazardous location.

Action: Connect the 2020 to the AC adapter and turn the instrument on. Turn the pump off. While the 2020 is running the internal battery is charging. Leave the instrument running for approximately 24 hours.

Problem: Instrument status shows "Over."

Cause: High concentrations of gases and vapors will cause a rapid change in signal level. The detector and associated electronics may become temporarily saturated.

Action: Wait a few seconds for the status to return to normal. PIDs are designed to detect relatively low concentrations of gases and vapors. Exposure to very high concentrations may result in a very high or maximum response.

Cause: The detector has become saturated.

Action: Move the 2020 to a location where it can sample clean air. Sample clean air until the reading stabilizes around 0.

Cause: Detector has been short circuited by foreign matter in the detector cell.

Note: Do not service the 2020 in a hazardous location.

Action: Do not touch the wire grid inside the detector cell. Use a gentle jet of compressed air to remove any dust or dirt in the detector cell.

Warning: Do not insert any object, other than the UV lamp, into the lampholder.

Cause: There is an undetermined problem.

Action: Contact the Equipment Manager.

Problem: Display is blank.

Cause: Battery pack is critically low.

Note: Do not remove or recharge the battery pack in a hazardous location.

Action: Replace the battery pack or connect the 2020 to the AC adapter.

Cause: The battery pack is not connected to the instrument correctly.

Action: Ensure the battery pack connector is securely attached to the connector on the 2020.

Cause: There is an undetermined problem.

Action: Reset the 2020. Leave the instrument on while disconnecting the battery pack. This will reset the instrument. Reconnect the battery pack and close the battery hatch. Turn on the 2020, set the time and date and program all the calibration memories.

Action: Contact the Equipment Manager.

Problem: Sample flow rate is less than 300 ml/min.

Cause: Inlet filter is plugged.

Note: Do not replace the inlet filter in a hazardous location.

Action: Replace inlet filter.

Cause: Inlet filter has not been installed properly.

Action: Ensure that the inlet filter has been installed correctly.

Cause: UV lamp is too long, causing flow to be restricted.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: If the UV lamp has a white serial number label, it is possible that the lamp is too long for the lampholder. Replace the lamp and contact the Equipment Manager.

Cause: UV lamp is too wide, causing flow to be restricted.

Action: If the UV lamp has a white serial number label, it is possible that the lamp is too wide for the lampholder. Contact the Equipment Manager.

Cause: The 2020 has been exposed to a solvent that can pass through the inlet filter and liquid has been aspirated.

Action: Contact the Equipment Manager.

Cause: Sample outlet is obstructed.

Action: Ensure the sample outlet is not obstructed in any way.

Cause: Pump has been damaged.

Action: Contact the Equipment Manager.

Problem: Liquid has been aspirated.

Cause: The 2020 has been exposed to a solvent that can pass through the inlet filter.

Action: Contact the Equipment Manager.

Problem: Corrosive gases and vapors have been sampled.

Cause: The 2020 has been exposed to corrosive gases and vapors.

Action: Corrosive gases and vapors can affect the electrodes within the detector as well as the lamp window. Prolonged exposure to corrosive materials may result in permanent fogging or etching of the window. If the 2020 is exposed to corrosive material, contact the Equipment Manager.

7.0 SHIPPING

- The Photovac may be shipped as cargo or carried on as luggage provided that there is no calibration gas cylinder accompanying the kit. When shipping or transporting the calibration gas, a Hazardous

Materials (Dangerous Good) Airbill, including the information as stipulated in Figure 5-5 will be prepared. **Only personnel who have been properly trained are permitted to offer a hazardous material for shipment.** The "Shipping Hazardous Materials" course offered by Tetra Tech NUS is considered acceptable training for this purpose. Specific instructions on packaging, labeling, and otherwise preparing a hazardous material shipment are presented in the Student Manual that accompanies the course.

8.0 REFERENCES

Photovac 2020 Photoionization Monitor User's Manual, 1995.

Student Manual from "Shipping Hazardous Materials" course, Tetra Tech NUS, 1999.

FIGURE 5-5

FedEx *Dangerous Goods Airbill*

Sender's Copy
RETAIN FOR 1 YEAR
RETAIN THIS COPY FOR YOUR RECORDS

The World On Time

1 From (please print and press hard)

Date _____ Sender's FedEx Account Number _____

Sender's Name _____ Phone ( ) _____

Company _____

Address _____ Dept/Floor/Suite/Room

City _____ State _____ ZIP _____

2 Your Internal Billing Reference Information (Optional) (First 24 characters will appear on invoice)

3 To (please print and press hard)

Recipient's Name Tom Patton Phone (412) 262-4583

Company Tetra Tech NUS

Address Spring Run Road Extension, Suite 140 (We Cannot Deliver to P.O. Boxes or P.O. ZIP Codes) Dept/Floor/Suite/Room

☐ Check here if residence (Extra charge applies for FedEx Express Saver)

City Coraopolis State PA ZIP 15108

For HOLD at FedEx Location check here: ☐ Hold Weekday ☐ Hold Saturday (Not available at all locations) (Available for FedEx Priority Overnight and FedEx 2Day only)

For WEEKEND Delivery check here (Extra Charge. Not available to all locations): ☐ Saturday Delivery (Available for FedEx Priority Overnight and FedEx 2Day only) ☐ NEW Sunday Delivery (Available for FedEx Priority Overnight only)

4a Express Package Service *Packages under 150 lbs.* Delivery commitment may be later in some areas.

☐ FedEx Priority Overnight (Next business morning) ☐ FedEx Standard Overnight (Next business afternoon)

☐ FedEx 2Day (Second business day) ☐ FedEx Express Saver (Third business day)

4b Express Freight Service *Packages over 150 lbs.* Delivery commitment may be later in some areas

☐ FedEx Overnight Freight (Next business day) ☐ FedEx 2Day Freight (Second business day) ☐ FedEx Express Saver Freight (Up to 3 business days)

(Call for delivery schedule. Some restrictions apply. See back for detailed descriptions of freight services.)

Service Conditions, Declared Value, and Limit of Liability – By using this Airbill you agree to the service conditions in our current Service Guide or U.S. Government Service Guide. Both are available on request. SEE BACK OF SENDER'S COPY OF THIS AIRBILL FOR INFORMATION AND ADDITIONAL TERMS. We will not be responsible for any claim in excess of $100 per package whether due to loss of box, damage, delay, non-delivery, misdelivery, or misinformation, unless you declare a higher value, pay an additional charge, and document your actual loss in a timely manner. Your right to recover from us for any loss includes intrinsic value of the package, loss of sales, interest, profit, attorney's fees, costs, and other forms of damage, whether direct, incidental, consequential, or special, and is limited to the greater of $100 or the declared value but cannot exceed actual documented loss. The maximum declared value for any FedEx Letter and FedEx Pak is $500. Federal Express may, upon your request, and with some limitations, refund all transportation charges paid.

See the FedEx Service Guide for further details

Questions? Call 1•800•Go•FedEx® (800)463-3339

5 Packaging

☒ Other Packaging

Dangerous Goods cannot be shipped in FedEx packaging.

6 Special Handling

☒ Dangerous Goods as per attached Shipper's Declaration ☐ Cargo Aircraft Only

7 Payment

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FedEx Account No. _____

Credit Card No. _____ Exp. Date _____

Total Packages _____ Total Weight _____ Total Declared Value* $ .00 Total Charges $

* When declaring a value higher than $100 per shipment, you pay an additional charge. See SERVICE CONDITIONS, DECLARED VALUE, AND LIMIT OF LIABILITY section for further information.

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Page 1 of 1 Pages

Two completed and signed copies of this Declaration must be handed to the operator.

WARNING

Failure to comply in all respects with the applicable Dangerous Goods Regulations may be in breach of the applicable law, subject to legal penalties. This Declaration must not, in any circumstances, be completed and/or signed by a consolidator, a forwarder or an IATA cargo agent.

TRANSPORT DETAILS

This shipment is within the limitations prescribed for: (delete non-applicable)

PASSENGER AND CARGO AIRCRAFT ~~XXXX CARGO AIRCRAFT XXXX~~

Airport of Departure:

Airport of Destination:

Shipment type: *(delete non-applicable)* NON-RADIOACTIVE ~~RADIOACTIVE XX~~

NATURE AND QUANTITY OF DANGEROUS GOODS

Dangerous Goods Identification							
Proper Shipping Name	Class or Division	UN or I.D. No.	Packing Group	Subsidiary Risk	Quantity and Type of Packaging	Packing Inst.	Authorization
Compressed Gas N.O.S. (mixture Nitrogen and Oxygen)	2.2	UN 1956			1 Plastic box x 0.56 Kg	200	

FOR SHIPMENT TO USA AIR AND HI ONLY

Additional Handling Information

Prepared for AIR TRANSPORT according to: (Customer MUST check one) ☐ 49 CFR ☒ ICAO / IATA

I hereby declare that the contents of this consignment are fully and accurately described above by the proper shipping name and are classified, packaged, marked, and labelled/placarded, and are in all respects in proper condition for transport according to applicable international and national governmental regulations.

Name/Title of Signatory

Place and Date

Signature *(see warning above)*

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IF ACCEPTABLE FOR PASSENGER AIRCRAFT, THIS SHIPMENT CONTAINS RADIOACTIVE MATERIAL INTENDED FOR USE IN, OR INCIDENT TO, RESEARCH, MEDICAL DIAGNOSIS, OR TREATMENT.

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1.0 PURPOSE

To establish procedures for the use, calibration, maintenance, troubleshooting, and shipment of the Photovac MicroFID handheld flame ionization detector.

2.0 SCOPE

Applies to all Brown & Root Environmental personnel who operate the MicroFID instrument during the performance of their work.

3.0 GLOSSARY

None.

4.0 RESPONSIBILITIES

Office Managers - Office Managers are responsible for ensuring that personnel under their direction who may use this device are first provided with adequate training and information.

Project Managers - Project Managers are responsible for ensuring that appropriate health and safety requirements and resources are addressed for their assigned projects.

Health and Safety Manager (HSM) - The HSM shall ensure that appropriate training is available to users of the Photovac MicroFID instrument.

Equipment Manager - The Equipment Manager shall ensure that all air monitoring instrumentation slated for field activities has been operationally checked out, fully charged, and calibrated prior to issuance for field service. Maintenance deficiencies identified by the Equipment Manager will require those instruments to be pulled from service until repairs can be facilitated.

Field Operations Leader (FOL)/Field Team Leader (FTL) - The FOL/FTL shall ensure all field team members using monitoring instruments as part of their assigned duties are adequately trained in their proper operation and limitations. The FOL/FTL shall ensure that the air monitoring instruments are employed as directed by site guidance documents (i.e., Work Plan, Health and Safety Plan, etc.). Additionally, the FOL/FTL shall ensure that the appropriate documentation and recordkeeping requirements are fulfilled including Documentation of Calibration and Direct Reading Instrument Response Data Sheets for air monitoring activities. On projects where a dedicated SSO is not assigned, the FOL/FTL is responsible for assuming the duties of that position.

Health and Safety Officer (HSO) - The HSO is responsible for determining air monitoring requirements for the site activities, and providing direction for air monitoring during specific site activities. This identification of types of air monitoring and direction for use are indicated within the Site-Specific Health and Safety Plan (HASP).

Site Safety Officer (SSO) - The SSO shall ensure the instruments identified are employed in the manner directed by the HSO and action levels employed as contingencies marks for the application of engineering controls, personal protective equipment (PPE) use, and administrative controls are employed as directed. Additionally, he/she shall ensure the instruments are properly maintained and calibrated prior to use in the field. The SSO during specific air monitoring applications including STEL and TWA mode measurements will be responsible for operation and application of this specialty air monitoring employment duty. The SSO is also responsible for addressing relevant Hazard Communication requirements (e.g., MSDS, chemical inventories, labeling, training, etc.) on each assigned project.

5.0 PROCEDURES

5.1 General

Direct-reading instruments such as a flame ionization detector are typically used to monitor for airborne releases that could present an inhalation threat to personnel, and to screen and bias environmental samples. Proper use of these instruments by trained, qualified personnel is essential to the validity of any acquired results. Also essential is that the devices are properly calibrated according to manufacturers instructions (and the specifications of this SOP), and that users of the instrument properly document results.

5.2 Use and Documentation of Results

As with any direct-reading instrument, understanding not only how - but when to use this instrument is essential to gathering relevant and valid data. This device will only respond to volatile organics in air that are combustible. Inappropriate instrument selection, use, or interpretation of instrument results by an unqualified user not only can yield inaccurate results, but could place personnel at risk of exposure to hazardous agents. Only personnel who are properly trained and authorized to use this device will be permitted to operate it.

It is essential that instrument operators understand and comply with the requirements to document results. This includes the need to document calibration results as well as operational readings. Calibration results must be recorded using Figure 5-1. Operational results can be recorded in several ways, including:

- Direct-Reading Instrument Response Data (Figure 5-2) - preferred method
- Boring Log Forms (Figure 5-3)
- Test Pit Log Forms (Figure 5-4)
- Log book entries

When using direct-reading instruments, it is important to monitor the air near the source of potential releases (e.g., drilling boreholes, tank entrances, drum openings, etc.) and at worker breathing zone areas. All readings should be recorded, including readings noted where background levels were not exceeded.

5.3 Principles of Operation

The MicroFID is a flame ionization detector used for the measurement of combustible organic compounds in air at parts per million levels. Permanent air gases (argon, carbon dioxide, nitrogen, oxygen, water vapor, etc.) are not ionized by the flame.

When the MicroFID is turned on, the display prompts you to turn on the hydrogen. The internal pump draws sample air in through MicroFID's inlet. This sample air provides the oxygen necessary for combustion in the hydrogen-fueled flame. When the proper ratio of hydrogen to air is present in the combustion chamber, the flame is automatically started with a glow plug. A thermocouple is used to monitor the status of the flame. When the sample passes through the flame the combustible organic compounds in the sample will be ionized. After the compounds have been ionized, they are subjected to

FIGURE 5-1

DOCUMENTATION OF FIELD CALIBRATION

SITE NAME: _____ PROJECT NO.: _____

Date of Calibration	Instrument Name and Model	Tetra Tech Instrument I.D. Number	Person Performing Calibration	Instrument Settings/Readings		Calibration Standard (Lot Number)	Remarks/ Comments
				Pre-Calibration Setting	Post-Calibration Reading		

FIGURE 5-2

DIRECT-READING INSTRUMENT RESPONSE DATA

SITE NAME: _____

PROJECT NO.: _____

PAGE OF

Date and Time(s)	Instrument Operator Name	Instrument Used	Instrument Reading and Duration	Location (e.g., borehole vs breathing zone)	Operation(s) Being Performed	Personnel in Vicinity at Time of Readings and Level of PPE Worn
Example 12-12-91/14:30	Joe Field	HNU 101	15 ppm/15 min.	breathing zone	sample location no. 12	Jim Technician/Level B George Grunt/Level B

FIGURE 5-2 (Continued)

SITE NAME: _____

PROJECT NO.: _____

PAGE _____ OF _____

Date and Time(s)	Instrument Operator Name	Instrument Used	Instrument Reading and Duration	Location (e.g., borehole vs breathing zone)	Operation(s) Being Performed	Personnel in Vicinity at Time of Readings and Level of PPE Worn

FIGURE 5-3

BORING LOG

Page ___ of ___

PROJECT NAME: _____ BORING NUMBER: _____
PROJECT NUMBER: _____ DATE: _____
DRILLING COMPANY: _____ GEOLOGIST: _____
DRILLING RIG: _____ DRILLER: _____

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: Soil Density/ Consistency or Rock Hardness	MATERIAL DESCRIPTION: Color	MATERIAL DESCRIPTION: Material Classification	USCS *	Remarks	PID/FID Reading (ppm): Sample	PID/FID Reading (ppm): Sampler BZ	PID/FID Reading (ppm): Borehole**	PID/FID Reading (ppm): Driller BZ**

* When rock coring, enter rock brokeness.

** Include monitor reading in 6 foot intervals @ borehole. Increase reading frequency if elevated response read.

Remarks: _____

Drilling Area Background (ppm): [ ]

Converted to Well: Yes _____ No _____ Well I.D. #: _____

FIGURE 5-4

Page ___ of ___

TEST PIT LOG

PROJECT NAME: _____ TEST PIT NUMBER: _____
PROJECT NUMBER: _____ DATE: _____
LOCATION: _____ GEOLOGIST: _____

Depth (Ft.)	Lithology Change (Depth/Ft.)	MATERIAL DESCRIPTION Soil/Waste Characteristics (lithology, density, color, etc.)	USCS	Remarks	PID/FID READING Source (ppm)	BZ (ppm)

TEST PIT CROSS SECTION AND / OR PLAN VIEW

REMARKS: _____

PHOTO LOG: _____

a continuous electric field between the repeller electrode at the jet and the collector electrode. The ions in the electric field generate a current which is proportional to the concentration of the ionized molecules in the ionization chamber. An electrometer circuit converts the current to a voltage that is then fed to the microprocessor which interprets the current in units of ppm. After the sample passes through the flame and has become ionized, it is vented from the detector through a flame arrestor. The flame arrestor prevents the flame from igniting any flammable gases present in the working atmosphere.

MicroFID is strictly an organic compound detector. It does not respond to inorganic compounds. MicroFID's sensitivity is highly dependent on chemical structure and bonding characteristics. The combustion efficiency of a compound determines its sensitivity. Simple saturated hydrocarbons (methane, ethane, etc.) posses high combustion efficiencies and are among the compounds that produce the highest MicroFID response. Organic fuels (acetylene, refined petroleum products), burn easily and are also extremely well detected.

The presence of substituted functional groups (amino, hydroxyl, halogens) on a simple hydrocarbon reduces its combustion efficiency and the MicroFID's sensitivity to the compound. For example, methanol and chloromethane are detectable with MicroFID, but not at the same sensitivity as methane. The number of carbon atoms can also affect the instrument's sensitivity due to substitution. For example, MicroFID is more sensitive to n-butanol than it is to methanol. For additional information regarding response factors of the MicroFID, consult the manufacturer's of the User's Manual.

Beginning Operation

The MicroFID can be operated without the activation of the flame to print or review logged data. In this way, the hydrogen fuel is conserved.

MicroFID will attempt to ignite the flame once the flow of hydrogen gas has been started. If the MicroFID has not been used for a while, it is possible that the gas supply lines are filled with air. If the flame cannot be started, MicroFID will begin a 30 second purge cycle. During the purge cycle it will flush the gas supply lines with hydrogen. After the purge cycle, it will attempt to light the flame again. If it fails again, another purge cycle will be performed and MicroFID will try a third time to ignite the flame. The following steps summarize proper start-up procedures.

1. Turn the instrument on by pressing the front of the On/Off switch. When the instrument is powered up, the version number and creation date of the instrument software are displayed. Press ENTER.

2. You will be prompted to start the flame. If you do not want to start the flame, use the ARROW keys to select "No Flame Needed" and press ENTER. To start the flame, use the ARROW keys to select Start Flame and press ENTER.

3. If you selected "Start Flame," MicroFID will prompt you to turn on the hydrogen. Turn the shut-off valve counterclockwise to start the flow of hydrogen and press ENTER.

4. The pump will start and MicroFID will then ignite the flame. You will hear a small pop when the flame has been ignited. Once the flame has been started the message "Detector flame has been started OK" will be displayed followed by the default display.

The default display provides the following information: instrument status, current detected concentration, event name (if the datalogger is on), time, and date. If an event name is longer than three characters, the bottom line of the display will scroll through the information.

The instrument status appears at the left of the upper line of the display and on the Print and Graph outputs. Each status has a priority assigned to it. If more than one status is in effect, then the status with the highest priority is displayed until the condition is corrected or until the option is turned off.

5.4 Calibration

The calibration (CAL) key is used to calibrate MicroFID. Before beginning calibration, ensure that you have a reliable source of both zero air and calibration gas. To document calibration efforts, field personnel will record information on the Documentation of Field Calibration Form (Figure 5-1), or the same information in the calibration/maintenance log book assigned to an instrument. A brief description of the functions under the CAL key are as follows:

1. When you press the CAL key you will first be prompted to select a Cal Memory. Each Cal Memory stores a unique zero point, sensitivity setting, response factor and alarm level.

2. You will then be prompted to enter a response factor. Refer to the manufacturer's User's Manual for a list of response factors. If the compound is not listed in that reference, or if you are measuring gas mixtures, enter a value of 1.00. The concentration detected by MicroFID will be multiplied by the response factor before it is displayed and logged.

3. Next select Low Range or High Range operation. Use Low Range if you are sampling concentrations between 0.5 and 2000 ppm (methane equivalents). Use High Range if you are sampling concentrations between 10 and 50,000 ppm (methane equivalents).

4. You will now be prompted to connect a supply of zero air. You may use ambient air or, for best results, use a clean Tedlar bag filled with zero grade air. In most cases, ambient air will be used provided calibration is performed in an area in which interfering airborne contaminants are not present. If using ambient air, press <ENTER> to being zeroing.

5. If you are using a charcoal filter to clean ambient air, connect the filter by loading the Teflon ferrules into the nut (the ferrules and the nut are supplied with the filter). Connect the nut to MicroFID's inlet. Do not tighten the nut. Remove the charcoal filter from its plastic bag and insert it into the nut. Finger tighten the nut onto the inlet. If the filter is not secure, ensure you have inserted the tube far enough into the nut. Do not over-tighten the fitting. Press <ENTER> and the MicroFID will set its zero point. NOTE: The charcoal filter does not filter methane or ethane. If these compounds are present, use a gas bag with a supply of commercial zero air.

6. If you are using a Tedlar bag filled with zero air, connect the bag to the inlet. Open the bag and press <ENTER>. MicroFID will set its zero point.

7. After MicroFID has set its zero point, you can then enter the concentration of the calibration gas (span gas), and then connect the Tedlar bag adapter to the inlet. Open the bag and press <ENTER>. MicroFID sets its sensitivity. Note: You must have a supply of calibration gas ready before calibrating MicroFID. When calibrating MicroFID, ensure the instrument is level. If MicroFID is tilted from side to side, gravity will affect the flame height and cause erroneous readings.

8. When MicroFID's display reverts to normal, it is calibrated and ready for use. Remove the Tedlar bag from the inlet.

9. Press the ALARM key and enter the alarm level for the selected CAL memory.

5.5 Routine Maintenance

5.5.1 Battery Charging

A fully charged battery will power the MicroFID for approximately 15 hours. If the instrument is to be used for more than 15 hours, carry a spare battery pack. Battery life is reduced if the instrument is turned off and then on again repeatedly.

When the instrument status displays "LoBat," the battery pack requires changing. When the "LoBat" status is displayed, you have a few minutes of operation left. MicroFID will turn itself off before the battery pack becomes critically low.

To remove the battery pack:

1. Stop the flow of hydrogen gas by turning the hydrogen shut-off valve fully clockwise. Turn the instrument off by pressing the On/Off switch twice.
2. Use the MicroFID multi-tool to loosen the two captive screws in the bottom of the battery pack.
3. A retainer at the rear of the instrument helps secure the battery pack to the instrument. Free the battery pack from the instrument.
4. Connect the charged battery pack to the retainer at the rear of the instrument.
5. Retighten the two captive screws and the bottom of the battery pack.

To charge the battery pack:

1. Ensure the correct plug is installed on the line cord of the battery charger.
2. Plug the charger into the jack located on the front of the battery pack.
3. Plug the charger into an AC outlet. The LED, on the battery pack indicates the charge state. Red indicates the battery is being charged. Green indicates the battery is fully charged and ready for use. It is normal for a fully charged battery to indicate it is charging (red light) when first plugged in. The LED will turn green as the battery charges.
4. When the battery pack is charged remove the charger, first from the wall outlet then from the battery pack.

Charging a fully discharged battery pack will take approximately 8 hours. Leaving the charger connected to a charged battery pack will not harm the battery or the charger in any way. If a battery pack is to be left indefinitely, leave it connected to the charger so that it will be fully charged and ready for operation.

5.5.2 Emptying the Hydrogen Cylinder

When you transport the MicroFID, you should empty the internal hydrogen cylinder and then refill it when you arrive at your destination.

To empty the cylinder:

1. Take the instrument outdoors, or to a well-ventilated area, at least 25 feet from any potential sources of ignition.

2. Turn the MicroFID off and open the hydrogen shut-off valve.

3. Remove the battery pack as described above.

4. Locate the purge outlet. It is located on the underside of the instrument.

5. Use the MicroFID multi-tool to turn the screws counterclockwise. Loosen the screw but do not remove it.

6. Leave the instrument so that the purge outlet is facing up. If the purge outlet is facing down, hydrogen will vent into MicroFID's case.

7. If the cylinder is full, it will take approximately 15 minutes to empty.

8. Watch the Contents gauge. When the cylinder is empty, close the purge outlet. Use the MicroFID multi-tool to turn the screw clockwise.

9. Replace the battery pack as discussed above.

5.5.5 Replacing the Sample Inlet Filter

MicroFID is equipped with a combined dust and water filter to reduce detector contamination. As the filter collects dust, MicroFID's inlet flow rate and sensitivity decrease. The filter will not allow water to pass through, but the filter will not stop gases and vapors.

Replace the filter on a weekly basis, or more frequently if MicroFID is used in a dusty or wet environment. You must replace the filter if MicroFID has been exposed to liquid water. The pump will sound labored when the filter requires replacement.

1. Turn off the instrument and unscrew the filter housing from the detector housing. Be careful not to lose the o-ring seal.

2. Remove the Teflon/Polypropylene filter and install the new filter. Place the filter in the filter housing with the Teflon side facing down into the filter housing and the mesh side facing the MicroFID. Handle the filter disk only by the edges. The mesh may be damaged or contaminated by excessive handling. Use forceps if possible.

3. Replace the filter housing.

4. Calibrate the CAL Memories that you are using before continuing operation.

5.6 Troubleshooting

This section provides guidance for troubleshooting the MicroFID. If problems are not corrected through these troubleshooting methods, contact the Photovac Service Department.

5.6.1 MicroFID Fault Messages

When the "Check" status is displayed, MicroFID's operation is compromised. Press the <TUTOR> key for a two-line description of the fault. One exception is the flame out fault. When a flame out fault occurs, the instrument status changes to "NoFlm."

Fault: Detector flame has gone out.

Cause: The hydrogen gas has run out.

Action: Ensure the shut-off valve is open. Check the hydrogen contents gauge on the side of the instrument and refill the hydrogen cylinder if necessary. Ensure the cylinder purge outlet has been closed.

Cause: Oxygen supply is deficient (Note: This is a Level B PPE condition).

Action: Ensure there is an adequate supply of oxygen. If you are sampling very high concentrations it is possible you are sampling above the flame out concentration. The flame out concentration for methane is approximately 52,000 ppm (5.2 percent methane in air).

A minimum of 17 percent oxygen is required to start the hydrogen flame. The oxygen is supplied from the sample as it is drawn in by the pump. A minimum of 10 percent oxygen is required to maintain the hydrogen flame.

Flame out also may occur when sampling enclosed or confined spaces where vapors and gases cannot escape. Watch for indications of increased flame height such as erratic readings or sudden high concentrations followed by a flame out fault.

If you will be using the MicroFID in a highly contaminated area where it is possible that the oxygen content will fall below 10 percent, watch for indication of reduced flame height such as lowered detection limits or a flame out fault.

Cause: High concentrations of flammable gases (gases within their flammable range) are present. High concentrations of flammable gases can act as an additional fuel source. When this happens, the flame height may increase beyond the confines of the combustion chamber. The hydrogen supply will then be cut-off and the flame will go out. Monitor LEL conditions and observe action levels specified in the Health and Safety Plan.

Action: Move to a location where there is an adequate supply of air and restart the flame. See the information above. Watch for indications of increased flame height such as erratic readings or sudden height concentrations followed by a flame out fault.

Cause: Exhaust port is blocked.

Action: At low temperatures, water vapor, a by-product of the hydrogen flame, may condense at the exhaust port. At sub-zero temperatures the water will freeze and obstruct the exhaust port. If the exhaust port becomes obstructed, pump operation will be inhibited. Flame out may also result. Operate the MicroFID within the operating temperature range 41 to 105 degrees Fahrenheit. In the event that the flame arrestor becomes clogged, contact the Photovac Service Department.

Cause: Sample line is blocked.

Action: Ensure the sample line is not obstructed in any way. If you are using the long sample probe, ensure flow is maintained through the entire length of tubing.

Cause: Inlet filter is plugged.

Action: Replace the inlet filter.

Fault: Signal from zero gas is too high.

Cause: Contamination of sample line or fittings before the detector.

Action: Clean or replace the sample line of the inlet filter.

Cause: Span gas is used instead of zero gas.

Action: Ensure clean gas is used to zero the MicroFID. Mark the calibration and zero gas Tedlar bags clearly.

Cause: Ambient air is contaminated.

Action: If you are unsure about the quality of the ambient air, use a charcoal filter or a supply of commercial zero grade air.

Cause: Hydrogen supply is contaminated.

Action: Hydrogen may react with the carbon element of the steel tank to produce methane. This will only occur if the cylinder is in poor condition and if the hydrogen has a high moisture content. Replace the hydrogen tank. Empty and refill the MicroFID internal cylinder with fresh hydrogen.

Fault: Signal from the calibration gas is too small

Cause: Calibration gas and zero air are switched.

Action: Ensure calibration gas is used to calibrate the MicroFID. Mark the calibration and zero gas Tedlar bags clearly. Ensure the calibration gas is of a reliable concentration.

Fault: Detector field voltage is low.

Cause: Internal fault in electronics.

Action: Contact the Photovac Service Department.

Problem: No instrument response detected, yet compounds are known to be present.

Cause: MicroFID has not been calibrated properly.

Action: Ensure calibration gas is of a reliable concentration and then calibrate the instrument. After the instrument has been calibrated, sample the Tedlar bag of calibration gas. A reading equivalent to the calibration gas should be displayed. If not, contact the Photovac Service Department.

Action: When calibrating the MicroFID, ensure the instrument is level. If the MicroFID is tilted side to side, gravity can affect the flame height and cause erroneous readings. If the sampling location is difficult to reach without tilting the instrument, use the long sample probe.

Cause: Background contamination from the hydrogen.

Action: It is possible that the hydrogen has become contaminated and is contributing a high background signal. If the hydrogen supply tank is more than 6 months old it should be replaced with a new cylinder. When ordering hydrogen, specify ultra-high purity (99.999 percent pure). Empty the MicroFID hydrogen cylinder (as described in Section 5.5.2 of this SOP) and then refill with hydrogen from the new cylinder.

Problem: **Date and time settings are not retained.**

Cause: MicroFID has not been used for 3 months or more and the internal battery (not the external battery pack) has been discharged.

Action: Turn MicroFID on and allow it to run until a "LoBat" status appears. This will take approximately 15 hours. Remove the battery pack and recharge it overnight. Repeat this procedure for 3 or 4 days. While MicroFID is running the internal battery is charging.

Problem: **Cannot fill the internal hydrogen cylinder to 1800 psi.**

Cause: Supply tank has less than 1800 psi of pressure. You can only fill the internal cylinder to a pressure of less than or equal to the tank pressure.

Action: Fill the internal cylinder to the pressure of the tank or replace the tank with a full one.

Cause: The hydrogen purge outlet is open.

Action: Close the outlet and fill the cylinder.

Cause: There is a problem with the refill adapter.

Action: Contact the Photovac Service Department

Problem: **Instrument status shows "Over."**

Cause: Rapid change in signal level. The detector electronics have been momentarily saturated.

Action: Wait a few seconds for the status to return to "Ready."

Cause: The detector has become saturated.

Action: Move the MicroFID to a location where it can sample clean air. Sample zero air until the reading stabilizes around "0." If you were using Low Range, switch to High Range. Calibrate the CAL Memory you were using when the "Over" status appeared.

Problem: **Display contrast bars are on or display is blank.**

Cause: Battery pack is critically low.

Action: Recharge the battery pack or connect the MicroFID to the battery charger.

Cause: The battery pack is not connected to the instrument properly.

Action: Ensure the battery pack has been aligned correctly. Ensure the battery pack is secured by the retainer at the rear of the instrument.

Problem: **Sample flow rate varies from 600 ml/min. +/-10 percent.**

Cause: Inlet filter has not been installed.

Action: Install an inlet filter.

Cause: Inlet filter has not been properly tightened onto the detector cap.

Action: Finger-tighten the filter cap.

Cause: Inlet filter is plugged.

Action: Replace the inlet filter.

Cause: Pump has been damaged.

Action: Contact the Photovac Service Department

Cause: Exhaust port is blocked.

Action: At low temperatures, water vapor (a by-product of the hydrogen flame) may condense at the exhaust port. At sub-zero temperatures the water will freeze and obstruct the exhaust port. If the exhaust port becomes obstructed, pump operation will be inhibited. Flame out may also result. Operate the MicroFID within the operating temperature range 41 to 105 degrees Fahrenheit. In the event that the flame arrestor becomes clogged, contact the Photovac Service Department.

Problem: **Flame will not ignite.**

Cause: The hydrogen gas has run out.

Action: Ensure the shut-off valve is open. Check the hydrogen contents gauge on the side of the instrument and refill the hydrogen cylinder if necessary. Ensure the hydrogen purge outlet is closed.

Cause: Oxygen supply is deficient.

Action: Ensure there is an adequate supply of oxygen. Do not attempt to ignite the flame in a location where there is the suspicion of encountering greater than 10,000 ppm methane or the equivalent concentration of a flammable gas. Move to a location where there are lower concentrations, start the flame and then begin sampling higher concentrations. Monitor for LEL conditions, following action levels specified in the Health and Safety Plan. If the flame goes out while you are sampling very high concentrations, it is possible you are sampling above the flame out concentration. The flame out concentration of methane is approximately 52,000 ppm (5.2 percent methane in air). A minimum of 17 percent oxygen is required to start the hydrogen flame. Oxygen is supplied from the sample as it is drawn in by the pump. A minimum or 10 percent oxygen is required to maintain the hydrogen flame.

Cause: Exhaust port is blocked.

Action: At low temperatures, water vapor (a by-product of the hydrogen flame) may condense at the exhaust port. At sub-zero temperatures the water will freeze and obstruct the exhaust

port. If the exhaust port becomes obstructed, pump operation will be inhibited. Flame out may also result. Operate the MicroFID within the operating temperature range 41 to 105 degrees Fahrenheit. In the event that the flame arrestor becomes clogged, contact the Photovac Service Department.

Cause: Hydrogen supply lines are full of air.

Action: If MicroFID has not been operated for some time, it is possible that the hydrogen supply lines contain air. Fill the hydrogen cylinder and then open the hydrogen shut-off valve. Allow the hydrogen to purge the system for about 5 minutes and then turn MicroFID on and start the flame.

Cause: Hydrogen lines are blocked.

Action: Contact the Photovac Service Department.

Problem: **Liquid has been aspirated.**

Cause: MicroFID has been exposed to a solvent that can pass through the Teflon/Polypropylene filter.

Action: Contact the Photovac Service Department.

5.7 Transporting MicroFID

When you transport MicroFID, you should empty the internal hydrogen cylinder and then refill it when you arrive at your destination (see Section 5.5.2 of this SOP). If you are traveling by passenger aircraft, you **must** empty the hydrogen cylinder. You cannot transport MicroFID by passenger aircraft with hydrogen in the cylinder.

The MicroFID can be shipped to sites. However, if shipment is to be performed while the cylinder still contains hydrogen, a Hazardous Materials Airbill must be filled out and the package must be properly marked and labeled. Examples of various completed forms are provided as Figures 6-1 and 6-2.

6.0 SHIPPING

The Photovac may be shipped as cargo or carried on as luggage providing there is no hydrogen fuel source or calibration gas cylinder accompanying the kit. **Only personnel who have been properly trained are permitted to offer a hazardous material for shipment.** The "Shipping Hazardous Materials" course offered by Tetra Tech NUS is considered acceptable training for this purpose. Specific instructions on packaging, labeling, and otherwise preparing a hazardous material shipment are presented in the Student Manual that accompanies the course. If shipping or transporting the hydrogen fuel source, a Hazardous Materials (or Dangerous Goods) Airbill such as the example in Figure 6-1 must be completed. When shipping or transporting the calibration gas, a separate Airbill (such as the one illustrated in Figure 6-2) must be prepared.

7.0 REFERENCES

MicroFID Handheld Flame Ionization Detector User's Manual, 1995.

Student Manual from "Shipping Hazardous Materials" course, Tetra Tech NUS, 1999.

FIGURE 6-1

EXAMPLE HAZARDOUS AIRBILL FOR HYDROGEN

FedEx. Dangerous Goods Airbill

288/300
7180827

Sender's Copy
RETAIN FOR 1 YEAR
RETAIN THIS COPY FOR YOUR RECORDS

The World On Time.

1 From (please print and press hard)

Date _____ Sender's FedEx Account Number _____

Sender's Name _____ Phone ( )

Company _____

Address _____ Dept./Floor/Suite/Room

City _____ State _____ ZIP _____

2 Your Internal Billing Reference Information (Optional) (First 24 characters will appear on invoice)

3 To (please print and press hard)

Recipient's Name TOM PATTON Phone (412) 262 4583

Company TETRA TECH NUS

Address SPRING RUN RD EXT STE 140 B 1 (To "HOLD" at FedEx location, print FedEx address here) (We Cannot Deliver to P.O. Boxes or P.O. ZIP Codes) Dept./Floor/Suite/Room

☐ Check here if residence (Extra charge applies for FedEx Express Saver)

City CORAOPOLIS State PA ZIP 15108

For HOLD at FedEx Location check here
☐ Hold Weekday ☐ Hold Saturday (Not available at all locations) (Available for FedEx Priority Overnight and FedEx 2Day only)

For WEEKEND Delivery check here (Extra Charge. Not available to all locations)
☐ Saturday Delivery (Available for FedEx Priority Overnight and FedEx 2Day only) ☐ NEW Sunday Delivery (Available for FedEx Priority Overnight only)

4a Express Package Service *Packages under 150 lbs.* Delivery commitment may be later in some areas.
☐ FedEx Priority Overnight (Next business morning) ☐ FedEx Standard Overnight (Next business afternoon)
☐ FedEx 2Day (Second business day) ☐ FedEx Express Saver (Third business day)

4b Express Freight Service *Packages over 150 lbs.* Delivery commitment may be later in some areas.
☐ FedEx Overnight Freight (Next business day) ☐ FedEx 2Day Freight (Second business day) ☐ FedEx Express Saver Freight (Up to 3 business days)
(Call for delivery schedule. Some restrictions apply. See back for detailed descriptions of freight services.)

Service Conditions, Declared Value, and Limit of Liability – By using this Airbill, you agree to the service conditions in our current Service Guide or U.S. Government Service Guide. Both are available on request. SEE BACK OF SENDER'S COPY OF THIS AIRBILL FOR INFORMATION AND ADDITIONAL TERMS. We will not be responsible for any claim in excess of $100 per package whether the result of loss, damage, delay, non-delivery, misdelivery, or misinformation, unless you declare a higher value, pay an additional charge, and document your actual loss in a timely manner. Your right to recover from us for any loss includes intrinsic value of the package, loss of sales, interest, profit, attorney's fees, costs, and either forms of damage, whether direct, incidental, consequential, or special, and is limited to the greater of $100 or the declared value but cannot exceed actual documented loss. The maximum declared value for any FedEx Letter and FedEx Pak is $500. Federal Express may, upon your request, and with some limitations, refund all transportation charges paid.

See the FedEx Service Guide for further details

Questions? Call 1•800•Go•FedEx® (800)463-3339

5 Packaging
☒ Other Packaging
Dangerous Goods cannot be shipped in FedEx packaging.

6 Special Handling
☒ Dangerous Goods as per attached Shipper's Declaration ☒ Cargo Aircraft Only

7 Payment
Bill to: ☐ Sender (Account No. in Section 1 will be billed) ☐ Recipient ☐ Third Party ☐ Credit Card ☐ Cash/Check
(Enter FedEx Account No. or Credit Card No. below)

FedEx Account No. _____

Credit Card No. _____ Exp. Date

Total Packages	Total Weight	Total Declared Value*	Total Charges
		$ .00	$

* When declaring a value higher than $100 per shipment, you pay an additional charge. See SERVICE CONDITIONS, DECLARED VALUE, AND LIMIT OF LIABILITY section for further information.

Signature Release Unavailable

PART #154261 • Rev. Date 4/98 ©1994-98 FedEx • PRINTED IN U.S.A.

FedEx Tracking Number 807286973876 Form I.D. No. 0204 355 98 SRC

Page 1 of 1 Pages

Two completed and signed copies of this Declaration must be handed to the operator.

WARNING
Failure to comply in all respects with the applicable Dangerous Goods Regulations may be in breach of the applicable law, subject to legal penalties. This Declaration must not, in any circumstances, be completed and/or signed by a consolidator, a forwarder or an IATA cargo agent.

TRANSPORT DETAILS
This shipment is within the limitations prescribed for: (delete non-applicable)
X PASSENGER AND CARGO AIRCRAFT | ~~CARGO AIRCRAFT ONLY~~
Airport of Departure:
Airport of Destination:

Shipment type: *(delete non-applicable)*
NON-RADIOACTIVE | ~~RADIOACTIVE~~

NATURE AND QUANTITY OF DANGEROUS GOODS

Dangerous Goods Identification							
Proper Shipping Name	Class or Division	UN or I.D. No.	Packing Group	Subsidiary Risk	Quantity and Type of Packaging	Packing Inst.	Authorization
Hydrogen Compressed	2.1	UN 1049			1 Plastic Box X 1.0 Kg	200	

FOR SHIPMENT TO USA, AK, AND HI ONLY

Additional Handling Information

Prepared for AIR TRANSPORT according to: (Customer MUST check one)
☐ 49 CFR ☒ ICAO / IATA

I hereby declare that the contents of this consignment are fully and accurately described above by the proper shipping name and are classified, packaged, marked, and labelled/placarded, and are in all respects in proper condition for transport according to applicable international and national governmental regulations.

Name/Title of Signatory
Place and Date
Signature (see warning above)

Emergency Telephone Number (Required for US Origin or Destination Shipments) 1-800-535-5053 Infotrac

IF ACCEPTABLE FOR PASSENGER AIRCRAFT, THIS SHIPMENT CONTAINS RADIOACTIVE MATERIAL INTENDED FOR USE IN, OR INCIDENT TO, RESEARCH, MEDICAL DIAGNOSIS, OR TREATMENT.

FIGURE 6-2

EXAMPLE HAZARDOUS AIRBILL FOR METHANE IN AIR

FedEx 6/30d 7181150

Dangerous Goods Airbill

Sender's Copy
RETAIN FOR 1 YEAR
RETAIN THIS COPY FOR YOUR RECORDS

The World On Time

1 From (please print and press hard)

Date _____ Sender's FedEx Account Number _____

Sender's Name _____ Phone ( )

Company _____

Address _____ Dept/Floor/Suite/Room

City _____ State _____ ZIP _____

2 Your Internal Billing Reference Information (Optional) (First 24 characters will appear on invoice)

3 To (please print and press hard)

Recipient's Name Tom Patton Phone (412) 262-4583

Company Tetra Tech NUS

Address Spring Run Road Extension, Suite 140 (To "HOLD" at FedEx location, print FedEx address here) (We Cannot Deliver to P.O. Boxes or P.O. ZIP Codes) Dept/Floor/Suite/Room

☐ Check here if residence (Extra charge applies for FedEx Express Saver)

City Coraopolis State PA ZIP 15108

For HOLD at FedEx Location check here
☐ Hold Weekday ☐ Hold Saturday (Not available at all locations) (Available for FedEx Priority Overnight and FedEx 2Day only)

For WEEKEND Delivery check here (Extra Charge. Not available to all locations)
☐ Saturday Delivery (Available for FedEx Priority Overnight and FedEx 2Day only) ☐ NEW Sunday Delivery (Available for FedEx Priority Overnight only)

4a Express Package Service *Packages under 150 lbs.* Delivery commitment may be later in some areas.
☐ FedEx Priority Overnight (Next business morning) ☐ FedEx Standard Overnight (Next business afternoon)
☐ FedEx 2Day (Second business day) ☐ FedEx Express Saver (Third business day)

4b Express Freight Service *Packages over 150 lbs.* Delivery commitment may be later in some areas.
☐ FedEx Overnight Freight (Next business day) ☐ FedEx 2Day Freight (Second business day) ☐ FedEx Express Saver Freight (Up to 3 business days)
(Call for delivery schedule. Some restrictions apply. See back for detailed descriptions of freight services.)

Service Conditions, Declared Value, and Limit of Liability – By using this Airbill you agree to the service conditions in our current Service Guide or U.S. Government Service Guide. Both are available on request. SEE BACK OF SENDER'S COPY OF THIS AIRBILL FOR INFORMATION AND ADDITIONAL TERMS. We will not be responsible for any claim in excess of $100 per package whether due to loss, damage, delay, non-delivery, misdelivery, or misinformation, unless you declare a higher value, pay an additional charge, and document your actual loss in a timely manner. Your right to recover from us for any loss includes actual value of the package, loss of sales, interest, profit, attorney's fees, costs, and other forms of damage, whether direct, incidental, consequential, or special, and is limited to the greater of $100 or the declared value but cannot exceed actual documented loss. The maximum declared value for any FedEx Letter and FedEx Pak is $500. Federal Express may, upon your request, and with some limitations, refund oil transportation charges paid.
See the FedEx Service Guide for further details

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5 Packaging
☒ Other Packaging
Dangerous Goods cannot be shipped in FedEx packaging.

6 Special Handling
☒ Dangerous Goods as per attached Shipper's Declaration ☐ Cargo Aircraft Only

7 Payment
Bill to: ☐ Sender (Account No. in Section 1 will be billed) ☐ Recipient ☐ Third Party ☐ Credit Card ☐ Cash/Check
(Enter FedEx Account No. or Credit Card No. below)

FedEx Account No. _____

Credit Card No. _____ Exp. Date

Total Packages	Total Weight	Total Declared Value*	Total Charges
		$ .00	

* When declaring a value higher than $100 per shipment, you pay an additional charge. See SERVICE CONDITIONS, DECLARED VALUE, AND LIMIT OF LIABILITY section for further information.

Signature Release Unavailable

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FedEx Tracking Number 807286974806 Form I.D. No. 0204 355 98 SRC

Page 1 of 1 Pages

Two completed and signed copies of this Declaration must be handed to the operator.

WARNING
Failure to comply in all respects with the applicable Dangerous Goods Regulations may be in breach of the applicable law, subject to legal penalties. This Declaration must not, in any circumstances, be completed and/or signed by a consolidator, a forwarder or an IATA cargo agent.

TRANSPORT DETAILS

This shipment is within the limitations prescribed for: (delete non-applicable)
PASSENGER AND CARGO AIRCRAFT | ~~CARGO AIRCRAFT ONLY~~

Airport of Departure:

Airport of Destination:

Shipment type: *(delete non-applicable)*
NON-RADIOACTIVE | ~~RADIOACTIVE~~

NATURE AND QUANTITY OF DANGEROUS GOODS

Dangerous Goods Identification

Proper Shipping Name	Class or Division	UN or I.D. No.	Packing Group	Subsidiary Risk	Quantity and Type of Packaging	Packing Inst.	Authorization
Compressed Gas N.O.S. (mixture Nitrogen and Oxygen)	2.2	UN 1956			1 Plastic box x 0.56 Kg	200	

Additional Handling Information

Prepared for AIR TRANSPORT according to: (Customer MUST check one)
☐ 49 CFR ☒ ICAO / IATA

I hereby declare that the contents of this consignment are fully and accurately described above by the proper shipping name and are classified, packaged, marked, and labelled/placarded, and are in all respects in proper condition for transport according to applicable international and national governmental regulations.

Name/Title of Signatory
Place and Date
Signature (see warning above)

Emergency Telephone Number (Required for US Origin or Destination Shipments) 1-800-535-5053 InfoTRAC

IF ACCEPTABLE FOR PASSENGER AIRCRAFT, THIS SHIPMENT CONTAINS RADIOACTIVE MATERIAL INTENDED FOR USE IN, OR INCIDENT TO, RESEARCH, MEDICAL DIAGNOSIS, OR TREATMENT.

U.S. ENVIRONMENTAL PROTECTION AGENCY
REGION I

LOW STRESS (low flow) PURGING AND SAMPLING PROCEDURE FOR THE COLLECTION OF GROUND WATER SAMPLES FROM MONITORING WELLS

July 30, 1996
Revision 2

U.S. ENVIRONMENTAL PROTECTION AGENCY
REGION I

LOW STRESS (low flow) PURGING AND SAMPLING PROCEDURE FOR THE COLLECTION OF GROUND WATER SAMPLES FROM MONITORING WELLS

I. SCOPE & APPLICATION

This standard operating procedure (SOP) provides a general framework for collecting ground water samples that are indicative of mobile organic and inorganic loads at ambient flow conditions (both the dissolved fraction and the fraction associated with mobile particulates). The SOP emphasizes the need to minimize stress by low water-level drawdowns, and low pumping rates (usually less than 1 liter/min) in order to collect samples with minimal alterations to water chemistry. This SOP is aimed primarily at sampling monitoring wells that can accept a submersible pump and have a screen, or open interval length of 10 feet or less (this is the most common situation). However, this procedure is flexible and can be used in a variety of well construction and ground-water yield situations. Samples thus obtained are suitable for analyses of ground water contaminants (volatile and semi-volatile organic analytes, pesticides, PCBs, metals and other inorganics), or other naturally occurring analytes.

This procedure does not address the collection of samples from wells containing light or dense non-aqueous phase liquids (LNAPLs and DNAPLs). For this the reader may wish to check: Cohen, R.M. and J.W. Mercer, 1993, DNAPL Site Evaluation; C.K. Smoley (CRC Press), Boca Raton, Florida and U.S. Environmental Protection Agency, 1992, RCRA Ground-Water Monitoring: Draft Technical Guidance; Washington, DC (EPA/530-R-93-001).

The screen, or open interval of the monitoring well should be optimally located (both laterally and vertically) to intercept existing contaminant plume(s) or along flowpaths of potential contaminant releases. It is presumed that the analytes of interest move (or potentially move) primarily through the more permeable zones within the screen, or open interval.

Use of trademark names does not imply endorsement by U.S.EPA but is intended only to assist in identification of a specific type of device.

Proper well construction and development cannot be overemphasized, since the use of installation techniques that are appropriate to the hydrogeologic setting often prevents "problem well" situations from occurring. It is also recommended that as part of development or redevelopment the well should be tested to determine the appropriate pumping rate to obtain stabilization of field indicator parameters with minimal drawdown in shortest amount of time. With this information field crews can then conduct purging and sampling in a more expeditious manner.

The mid-point of the saturated screen length (which should not exceed 10 feet) is used by convention as the location of the pump intake. However, significant chemical or permeability contrast(s) within the screen may require additional field work to determine the optimum vertical location(s) for the intake, and appropriate pumping rate(s) for purging and sampling more localized target zone(s). Primary flow zones (high(er) permealability and/or high(er) chemical concentrations) should be identified in wells with screen lengths longer than 10 feet, or in wells with open boreholes in bedrock. Targeting these zones for water sampling will help insure that the low stress procedure will not underestimate contaminant concentrations. The Sampling and Analysis Plan must provide clear instructions on how the pump intake depth(s) will be selected, and reason(s) for the depth(s) selected.

Stabilization of indicator field parameters is used to indicate that conditions are suitable for sampling to begin. Achievement of turbidity levels of less than 5 NTU and stable drawdowns of less than 0.3 feet, while desirable, are not mandatory. Sample collection may still take place provided the remaining criteria in this procedure are met. If after 4 hours of purging indicator field parameters have not stabilized, one of 3 optional courses of action may be taken: a) continue purging until stabilization is achieved, b) discontinue purging, do not collect any samples, and record in log book that stabilization could not be achieved (documentation must describe attempts to achieve stabilization) c) discontinue purging, collect samples and provide full explanation of attempts to achieve stabilization (note: there is a risk that the analytical data obtained, especially metals and strongly hydrophobic organic analytes, may not meet the sampling objectives).

Changes to this SOP should be proposed and discussed when the site Sampling and Analysis Plan is submitted for approval. Subsequent requests for modifications of an approved plan must include adequate technical justification for proposed changes. All changes and modifications must be approved before implementation in field.

II. EQUIPMENT

A. Extraction device

Adjustable rate, submersible pumps are preferred (for example, centrifugal or bladder pump constructed of stainless steel or

Teflon).

Adjustable rate, peristaltic pumps (suction) may be used with caution. Note that EPA guidance states: "Suction pumps are not recommended because they may cause degassing, pH modification, and loss of volatile compounds" (EPA/540/P-87/001, 1987, page 8.5-11).

The use of inertial pumps is discouraged. These devices frequently cause greater disturbance during purging and sampling and are less easily controlled than the pumps listed above. This can lead to sampling results that are adversely affected by purging and sampling operations, and a higher degree of data variability.

B. Tubing

Teflon or Teflon lined polyethylene tubing are preferred when sampling is to include VOCs, SVOCs, pesticides, PCBs and inorganics.

PVC, polypropylene or polyethylene tubing may be used when collecting samples for inorganics analyses. However, these materials should be used with caution when sampling for organics. If these materials are used, the equipment blank (which includes the tubing) data must show that these materials do not add contaminants to the sample.

Stainless steel tubing may be used when sampling for VOCs, SVOCs, pesticides, and PCBs. However, it should be used with caution when sampling for metals.

The use of 1/4 inch or 3/8 inch (inner diameter) tubing is preferred. This will help ensure the tubing remains liquid filled when operating at very low pumping rates.

Pharmaceutical grade (Pharmed) tubing should be used for the section around the rotor head of a peristaltic pump, to minimize gaseous diffusion.

C. Water level measuring device(s), capable of measuring to 0.01 foot accuracy (electronic "tape", pressure transducer). Recording pressure transducers, mounted above the pump, are especially helpful in tracking water levels during pumping operations, but their use must include check measurements with a water level "tape" at the start and end of each record.

D. Flow measurement supplies (e.g., graduated cylinder and stop watch).

E. Interface probe, if needed.

F. Power source (generator, nitrogen tank, etc.). If a gasoline generator is used, it must be located downwind and at least 30 feet from the well so that the exhaust fumes do not contaminate the samples.

G. Indicator field parameter monitoring instruments - pH, Eh, dissolved oxygen (DO), turbidity, specific conductance, and temperature. Use of a flow-through-cell is required when measuring all listed parameters, except turbidity. Standards to perform field calibration of instruments. Analytical methods are listed in 40 CFR 136, 40 CFR 141, and SW-846. For Eh measurements, follow manufacturer's instructions.

H. Decontamination supplies (for example, non-phosphate detergent, distilled/deionized water, isopropyl alcohol, etc.).

I. Logbook(s), and other forms (for example, well purging forms).

J. Sample Bottles.

K. Sample preservation supplies (as required by the analytical methods).

L. Sample tags or labels.

M. Well construction data, location map, field data from last sampling event.

N. Well keys.

O. Site specific Sample and Analysis Plan/Quality Assurance Project Plan.

P. PID or FID instrument (if appropriate) to detect VOCs for health and safety purposes, and provide qualitative field evaluations.

III. PRELIMINARY SITE ACTIVITIES

Check well for security damage or evidence of tampering, record pertinent observations.

Lay out sheet of clean polyethylene for monitoring and sampling equipment.

Remove well cap and immediately measure VOCs at the rim of the well with a PID or FID instrument and record the reading in the field logbook.

If the well casing does not have a reference point (usually a V-cut or indelible mark in the well casing), make one. Describe its location and record the date of the mark in the logbook.

A synoptic water level measurement round should be performed (in the shortest possible time) before any purging and sampling activities begin. It is recommended that water level depth (to 0.01 ft.) and

total well depth (to 0.1 ft.) be measured the day before, in order to allow for re-settlement of any particulates in the water column. If measurement of total well depth is not made the day before, it should not be measured until after sampling of the well is complete. All measurements must be taken from the established referenced point. Care should be taken to minimize water column disturbance.

Check newly constructed wells for the presence of LNAPLs or DNAPLs before the initial sampling round. If none are encountered, subsequent check measurements with an interface probe are usually not needed unless analytical data or field head space information signal a worsening situation. Note: procedures for collection of LNAPL and DNAPL samples are not addressed in this SOP.

IV. PURGING AND SAMPLING PROCEDURE

Sampling wells in order of increasing chemical concentrations (known or anticipated) is preferred.

1. Install Pump

Lower pump, safety cable, tubing and electrical lines slowly (to minimize disturbance) into the well to the midpoint of the zone to be sampled. The Sampling and Analysis Plan should specify the sampling depth, or provide criteria for selection of intake depth for each well (see Section I). If possible keep the pump intake at least two feet above the bottom of the well, to minimize mobilization of particulates present in the bottom of the well. Collection of turbid free water samples may be especially difficult if there is two feet or less of standing water in the well.

2. Measure Water Level

Before starting pump, measure water level. If recording pressure transducer is used-initialize starting condition.

3. Purge Well

3a. Initial Low Stress Sampling Event

Start the pump at its lowest speed setting and slowly increase the speed until discharge occurs. Check water level. Adjust pump speed until there is little or no water level drawdown (less than 0.3 feet). If the minimal drawdown that can be achieved exceeds 0.3 feet but remains stable, continue purging until indicator field parameters stabilize.

Monitor and record water level and pumping rate every three to five minutes (or as appropriate) during purging. Record any pumping rate adjustments (both time and flow rate). Pumping rates should, as needed, be reduced to the minimum capabilities of the pump (for example, 0.1 - 0.4 l/min) to ensure stabilization of indicator

parameters. Adjustments are best made in the first fifteen minutes of pumping in order to help minimize purging time. During pump start-up, drawdown may exceed the 0.3 feet target and then "recover" as pump flow adjustments are made. Purge volume calculations should utilize stabilized drawdown value, not the initial drawdown. Do not allow the water level to fall to the intake level (if the static water level is above the well screen, avoid lowering the water level into the screen). The final purge volume must be greater than the stabilized drawdown volume plus the extraction tubing volume.

Wells with low recharge rates may require the use of special pumps capable of attaining very low pumping rates (bladder, peristaltic), and/or the use of dedicated equipment. If the recharge rate of the well is lower than extraction rate capabilities of currently manufactured pumps and the well is essentially dewatered during purging, then the well should be sampled as soon as the water level has recovered sufficiently to collect the appropriate volume needed for all anticipated samples (ideally the intake should not be moved during this recovery period). Samples may then be collected even though the indicator field parameters have not stabilized.

3b. Subsequent Low Stress Sampling Events

After synoptic water level measurement round, check intake depth and drawdown information from previous sampling event(s) for each well. Duplicate, to the extent practicable, the intake depth and extraction rate (use final pump dial setting information) from previous event(s). Perform purging operations as above.

4. Monitor Indicator Field Parameters

During well purging, monitor indicator field parameters (turbidity, temperature, specific conductance, pH, Eh, DO) every three to five minutes (or less frequently, if appropriate). Note: during the early phase of purging emphasis should be put on minimizing and stabilizing pumping stress, and recording those adjustments. Purging is considered complete and sampling may begin when all the above indicator field parameters have stabilized. Stabilization is considered to be achieved when three consecutive readings, taken at three (3) to five (5) minute intervals, are within the following limits:

turbidity (10% for values greater than 1 NTU),
DO (10%),
specific conductance (3%),
temperature (3%),
pH (± 0.1 unit),
ORP/Eh (± 10 millivolts).

All measurements, except turbidity, must be obtained using a flow-through-cell. Transparent flow-through-cells are preferred, because they allow field personnel to watch for particulate build-up within the cell. This build-up may affect indicator field parameter values

measured within the cell and may also cause an underestimation of turbidity values measured after the cell. If the cell needs to be cleaned during purging operations, continue pumping and disconnect cell for cleaning, then reconnect after cleaning and continue monitoring activities.

The flow-through-cell must be designed in a way that prevents air bubble entrapment in the cell. When the pump is turned off or cycling on/off (when using a bladder pump), water in the cell must not drain out. Monitoring probes must be submerged in water at all times. If two flow-through-cells are used in series, the one containing the dissolved oxygen probe should come first (this parameter is most susceptible to error if air leaks into the system).

5. Collect Water Samples

Water samples for laboratory analyses must be collected before water has passed through the flow-through-cell (use a by-pass assembly or disconnect cell to obtain sample).

VOC samples should be collected first and 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.

During purging and sampling, the tubing should remain filled with water so as to minimize possible changes in water chemistry upon contact with the atmosphere. It is recommended that 1/4 inch or 3/8 inch (inside diameter) tubing be used to help insure that the sample tubing remains water filled. If the pump tubing is not completely filled to the sampling point, use one of the following procedures to collect samples: (1) add clamp, connector (Teflon or stainless steel) or valve to constrict sampling end of tubing; (2) insert small diameter Teflon tubing into water filled portion of pump tubing allowing the end to protrude beyond the end of the pump tubing, collect sample from small diameter tubing; (3) collect non-VOC samples first, then increase flow rate slightly until the water completely fills the tubing, collect sample and record new drawdown, flow rate and new indicator field parameter values.

Add preservative, as required by analytical methods, to samples immediately after they are collected if the sample containers are not pre-preserved. Check analytical methods (e.g. EPA SW-846, water supply, etc.) for additional information on preservation. Check pH for all samples requiring pH adjustment to assure proper pH value. For VOC samples, this will require that a test sample be collected during purging to determine the amount of preservative that needs to be added to the sample containers prior to sampling.

If determination of filtered metal concentrations is a sampling objective, collect filtered water samples using the same low flow procedures. The use of an in-line filter is required, and the filter

size (0.45 um is commonly used) should be based on the sampling objective. Pre-rinse the filter with approximately 25 - 50 ml of ground water prior to sample collection. Preserve filtered water sample immediately. Note: filtered water samples are not an acceptable substitute for unfiltered samples when the monitoring objective is to obtain chemical concentrations of total mobile contaminants in ground water for human health risk calculations.

Label each sample as collected. Samples requiring cooling (volatile organics, cyanide, etc.) will be placed into a cooler with ice or refrigerant for delivery to the laboratory. Metal samples after acidification to a pH less than 2 do not need to be cooled.

[illegible] Sampling Activities

If recording pressure transducer is used, remeasure water level with [illegible].

After collection of the samples, the pump tubing may either be [illegible] to the well for resampling (by hanging the tubing inside the well), decontaminated, or properly discarded.

Before securing the well, measure and record the well depth (to 0.1 [illegible]), if not measured the day before purging began. Note: Measurement of total well depth is optional after the initial low flow sampling event. However, it is recommended if the well has a "silting" problem or if confirmation of well identity is needed.

Secure the well.

[illegible] DECONTAMINATION

Decontaminate sampling equipment prior to use in the first well and following sampling of each subsequent well. Pumps will not be removed between purging and sampling operations. The pump and tubing (including support cable and electrical wires which are in contact with the well) will be decontaminated by one of the procedures listed below.

Procedure 1

The decontaminating solutions can be pumped from either buckets or short PVC casing sections through the pump or the pump can be disassembled and flushed with the decontaminating solutions. It is recommended that detergent and isopropyl alcohol be used sparingly in the decontamination process and water flushing steps be extended to ensure that any sediment trapped in the pump is removed. The pump exterior and electrical wires must be rinsed with the decontaminating solutions, as well. The procedure is as follows:

Flush the equipment/pump with potable water.

Flush with non-phosphate detergent solution. If the solution is recycled, the solution must be changed periodically.

Flush with potable or distilled/deionized water to remove all of the detergent solution. If the water is recycled, the water must be changed periodically.

Flush with isopropyl alcohol (pesticide grade). If equipment blank data from the previous sampling event show that the level of contaminants is insignificant, then this step may be skipped.

Flush with distilled/deionized water. The final water rinse must not be recycled.

Procedure 2

Steam clean the outside of the submersible pump.

Pump hot potable water from the steam cleaner through the inside of the pump. This can be accomplished by placing the pump inside a three or four inch diameter PVC pipe with end cap. Hot water from the steam cleaner jet will be directed inside the PVC pipe and the pump exterior will be cleaned. The hot water from the steam cleaner will then be pumped from the PVC pipe through the pump and collected into another container. Note: additives or solutions should not be added to the steam cleaner.

Pump non-phosphate detergent solution through the inside of the pump. If the solution is recycled, the solution must be changed periodically.

Pump potable water through the inside of the pump to remove all of the detergent solution. If the solution is recycled, the solution must be changed periodically.

Pump distilled/deionized water through the pump. The final water rinse must not be recycled.

VI. FIELD QUALITY CONTROL

Quality control samples are required to verify that the sample collection and handling process has not compromised the quality of the ground water samples. All field quality control samples must be prepared the same as regular investigation samples with regard to sample volume, containers, and preservation. The following quality control samples shall be collected for each batch of samples (a batch may not exceed 20 samples). Trip blanks are required for the VOC samples at a frequency of one set per VOC sample cooler.

Field duplicate.

Matrix spike.

Matrix spike duplicate.

Equipment blank.

Trip blank (VOCs).

Temperature blank (one per sample cooler).

Equipment blank shall include the pump and the pump's tubing. If tubing is dedicated to the well, the equipment blank will only include the pump in subsequent sampling rounds.

Collect samples in order from wells with lowest contaminant concentration to highest concentration. Collect equipment blanks after sampling from contaminated wells and not after background wells.

Field duplicates are collected to determine precision of sampling procedure. For this procedure, collect duplicate for each analyte group in consecutive order (VOC original, VOC duplicate, SVOC original, SVOC duplicate, etc.).

If split samples are to be collected, collect split for each analyte group in consecutive order (VOC original, VOC split, etc.). Split sample should be as identical as possible to original sample.

All monitoring instrumentation shall be operated in accordance with EPA analytical methods and manufacturer's operating instructions. EPA analytical methods are listed in 40 CFR 136, 40 CFR 141, and SW-846 with exception of Eh, for which the manufacturer's instructions are to be followed. Instruments shall be calibrated at the beginning of each day. If a measurement falls outside the calibration range, the instrument should be re-calibrated so that all measurements fall within the calibration range. At the end of each day, check calibration to verify that instruments remained in calibration. Temperature measuring equipment, thermometers and thermistors, need not be calibrated to the above frequency. They should be checked for accuracy prior to field use according to EPA Methods and the manufacturer's instructions.

VII. FIELD LOGBOOK

A field log shall be kept to document all ground water field monitoring activities (see attached example matrix), and record all of the following:

Well identification.

Well depth, and measurement technique.

Static water level depth, date, time and measurement technique.

Presence and thickness of immiscible liquid (NAPL) layers and

detection method.

Pumping rate, drawdown, indicator parameters values, and clock time, at the appropriate time intervals; calculated or measured total volume pumped.

Well sampling sequence and time of each sample collection.

Types of sample bottles used and sample identification numbers.

Preservatives used.

Parameters requested for analysis.

Field observations during sampling event.

Name of sample collector(s).

Weather conditions.

QA/QC data for field instruments.

Any problems encountered should be highlighted.

Description of all sampling equipment used, including trade names, model number, diameters, material composition, etc.

VIII. DATA REPORT

Data reports are to include laboratory analytical results, QA/QC information, and whatever field logbook information is needed to allow for a full evaluation of data useability.

EXAMPLE (Minimum Requirements)

Well PURGING-FIELD WATER QUALITY MEASUREMENTS FORM

Page____of____

Location (Site/Facility Name)_____

Well Number_____ Date_____

Field Personnel_____

Sampling Organization_____

Identify MP_____

Depth to _____/_____ of screen
(below MP) top bottom

Pump Intake at (ft. below MP)_____

Purging Device; (pump type)_____

Clock Time 24 HR	Water Depth below MP ft	Pump Dial[1]	Purge Rate ml/min	Cum. Volume Purged liters	Temp. °C	Spec. Cond.[2] µS/cm	pH	ORP/ Eh[3] mv	DO mg/L	Turb-idity NTU	Comments

1. Pump dial setting (for example: hertz, cycles/min, etc).
2. µSiemens per cm(same as µmhos/cm)at 25 °C.
3. Oxidation reduction potential (stand in for Eh).

U.S. ENVIRONMENTAL PROTECTION AGENCY
REGION 1

DRAFT CALIBRATION OF FIELD INSTRUMENTS
(temperature, pH, dissolved oxygen, conductivity/specific conductance, oxidation/reduction potential [ORP], and turbidity)

I. SCOPE & APPLICATION

The purpose of this standard operating procedure (SOP) is to provide a framework for calibrating field instruments used to measure water quality parameters for ground water and surface water. Water quality parameters include temperature, pH, dissolved oxygen, conductivity/specific conductance, oxidation/reduction potential [ORP], and turbidity. This SOP supplements, but does not replace, EPA analytical methods listed in 40 CFR 136 and 40 CFR 141 for temperature, dissolved oxygen, conductivity/specific conductance, pH and turbidity.

This SOP is written for instruments that utilize multiple probes (temperature, pH, dissolved oxygen, conductivity/specific conductance, and/or oxidation/reduction potential [ORP]) and the probe readings for pH, dissolved oxygen, and specific conductance are automatically corrected for temperature. Communications to the instrument (programming and displaying the measurement values) are performed using a display/logger or a computer. Information sent to the instrument is entered through the keypad on the display/logger or computer. It is desirable that the display/logger or computer have data storage capabilities. If the instrument does not have a keypad, follow the manufacturer's instructions for entering information into the instrument.

For ground water monitoring, the instrument must be equipped with a flow-through-cell, and the display/logger or computer display screen needs to be large enough to simultaneously contain the readouts of each probe in the instrument. Turbidity is measured using a separate instrument because turbidity cannot be measured in a flow-through-cell. This procedure is applicable for use with the EPA Region 1 Low Stress (low flow) Purging and Sampling Procedure for the Collection of Ground Water Samples from Monitoring Wells.

II. GENERAL

All monitoring instruments must be calibrated before they are used to measure environmental samples. Part of the calibration is performed prior to the field event. For instrument probes that

rely on the temperature sensor (pH, dissolved oxygen, conductivity/specific conductance, and oxidation/reduction potential [ORP]), each temperature sensor needs to be checked for accuracy against a thermometer that is traceable to the National Institute of Standards and Technology (NIST). Before any instrument is calibrated or used to perform environmental measurements, the instrument must stabilize (warm-up) according to manufacturer's instructions.

Most instruments will require at least two standards to bracket the expected measurement range, that is, one standard less than the expected value and one higher. Calibration must be performed at the beginning of each sampling day prior to sample collection. To determine if the instruments have remained in calibration during transport to each sampling location, use one of the previously used standards as a check standard at the sampling site. If the check measurement does not agree with the initial calibration or to within the specifications of the instrument, then the instrument must be re-calibrated. When an environmental sample measurement falls outside the calibration range, the instrument must be re-calibrated to bracket the new range before continuing measurements.

This SOP requires that the manufacturer's instruction manual (including the instrument specifications) accompany the instrument into the field.

III. CALIBRATION PROCEDURES

Prior to calibration, all instrument probes must be cleaned according to the manufacturer's instructions. Failure to perform this step (proper maintenance) can lead to erratic measurements.

Program the multi-probe instrument so that the following parameters to be measured will be displayed: temperature, pH, percent dissolved oxygen, mg/l dissolved oxygen, conductivity, specific conductance, and ORP.

The volume of the calibration solutions must be sufficient to cover both the probe and temperature sensor (see manufacturer's instructions for additional information).

While calibrating or measuring, make sure there are no air bubbles lodged between the probe and the probe guard.

TEMPERATURE

Most instrument manuals state there is no calibration of the temperature sensor, but the

temperature sensor must be checked to determine its accuracy. This accuracy check is performed at least once per year and the accuracy check date/information is kept with the instrument. If the accuracy check date/information is not included with the instrument or the last check was over a year, the temperature sensor accuracy needs to be checked at the beginning of the sampling event. If the instrument contains multiple temperature sensors, each sensor must be checked.

Verification Procedure

1. Allow a container filled with water to come to room temperature.

2. Place a thermometer that is traceable to the National Institute of Standards and Technology (NIST), and the instrument's temperature sensor into the water and wait for both temperature readings to stabilize.

3. Compare the two measurements. The instrument's temperature sensor must agree with the reference thermometer measurement within the accuracy of the sensor (usually ±0.15°C). If the measurements do not agree, the instrument may not be working properly and the manufacturer needs to be consulted.

pH (electrometric)

The pH of a sample is determined electrometrically using a glass electrode.

Choose the appropriate buffered standards that will bracket the expected values at the sampling locations. For ground water, the pH will usually be close to seven. Three standards are needed for the calibration: one close to seven, one at least two pH units below seven and the other at least two pH units above seven. For those instruments that will not accept three standards, the instrument will need to be re-calibrated if the water sample's pH is outside the initial calibration range described by the two standards.

Calibration Procedure

1. Allow the buffered standards to equilibrate to the ambient temperature.

2. Fill calibration containers with the buffered standards so each standard will cover the pH probe and temperature sensor.

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3. Remove probe from its storage container, rinse with distilled water, blot dry with soft tissue.

4. Select monitoring/run mode. Immerse probe into the initial standard (e.g., pH 7).

5. Stir the standard until the readings stabilize. If the reading does not change within 30 seconds, select calibration mode and then select "pH". Enter the buffered standard value into instrument. Select monitoring/run mode. The readings should remain within manufacturer's specifications; if they change, re-calibrate. If readings continue to change after re-calibration, consult manufacturer.

6. Remove probe from the initial standard, rinse with distilled water, and blot dry.

7. Immerse probe into the second standard (e.g., pH 4). Repeat step 5.

8. Remove probe from the second standard, rinse with distilled water, and blot dry. If instrument only accepts two standards, the calibration is complete. Go to step 11. Otherwise continue.

9. Immerse probe in third buffered standard (e.g., pH 9) and repeat step 5.

10. Remove probe from the third standard, rinse with distilled water, and blot dry.

11. Select monitoring/run mode, if not already selected. To ensure that the initial calibration standard (e.g., pH 7) has not changed, immerse the probe into the initial standard. Wait for the readings to stabilize. The reading should read the initial standard value within the manufacturer's specifications. If not, re-calibrate the instrument. If re-calibration does not help, the calibration range may be too great. Reduce calibration range by using standards that are closer together.

12. The calibration is complete. Place pH probe in its storage container.

DISSOLVED OXYGEN

Dissolved oxygen (DO) content in water is measured using a membrane electrode. The DO probe's membrane and electrolyte solution should be replaced prior to the sampling period. Failure to perform this step may lead to erratic measurements.

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Calibration Procedure

1. Gently dry the temperature sensor according to manufacturer's instructions.

2. Place a wet sponge or a wet paper towel on the bottom of the DO calibration container.

3. Place the DO probe into the container without the probe coming in contact with the wet sponge or paper towel. The probe must fit tightly into the container to prevent the escape of moisture evaporating from the sponge or towel.

4. Allow the confined air to become saturated with water vapor (saturation occurs in approximately 10 to 15 minutes). During this time, turn-on the instrument to allow the DO probe to warm-up. Select monitoring/run mode. Check temperature readings. Readings must stabilize before continuing to the next step.

5. Select calibration mode; then select "DO %".

6. Enter the local barometric pressure (usually in mm of mercury) for the sampling location into the instrument. This measurement must be determined from an on-site barometer. Do not use barometric pressure obtained from the local weather services unless the pressure is corrected for the elevation of the sampling location. [Note: inches of mercury times 25.4 mm/inch equals mm of mercury or consult Oxygen Solubility at Indicated Pressure chart attached to the SOP for conversion at selected pressures].

7. The instrument should indicate that the calibration is in progress. The instrument will take approximately one minute to calibrate. After calibration, the instrument should display percent saturated DO.

8. Select monitoring/run mode. Compare the DO mg/l reading to the Oxygen Solubility at Indicated Pressure chart attached to the SOP. The numbers should agree. If they do not agree, to the accuracy of the instrument (usually ± 0.2 mg/L), repeat calibration. If this does not work, change the membrane and electrolyte solution.

9. Remove the probe from the container and place it into a 0.0 mg/L DO standard (see note). The standard must be filled to the top of its container and the DO probe must fit tightly into the standard's container (no head space). Check temperature readings. They must stabilize before continuing.

10. Wait until the "mg/l DO" readings have stabilized. The instrument should read 0.0 mg/L or to the accuracy of the instrument (usually ± 0.2 mg/L). If the instrument cannot reach these values, it will be necessary to clean the probe, and change the membrane and electrolyte solution. If this does not work, prepare a new 0.0 mg/L DO standard. If these measures do not work, contact manufacturer.

Note: To prepare a zero mg/L DO standard follow the procedure stated in Standard Methods (Method 4500-O G). The method basically states to add excess sodium sulfite (until no more dissolves) and a trace amount of cobalt chloride to water. The standard container must be completely filled (no head space). This solution is prepared prior to the sampling event. If some of the solution is lost during instrument calibration, add more water to the container so that the standard is stored with no head space.

SPECIFIC CONDUCTANCE

Conductivity is used to measure the ability of an aqueous solution to carry an electrical current. Specific conductance is the conductivity value corrected to 25°C.

Most instruments are calibrated against a single standard which is near, but below the specific conductance of the environmental samples. A second standard which is above the environmental sample specific conductance is used to check the linearity of the instrument in the range of measurements.

Calibration Procedure

1. Allow the calibration standard to equilibrate to the ambient temperature.

2. Remove probe from its storage container, rinse the probe with a small amount of the conductivity/specific conductance standard (discard the rinsate), and place the probe into the conductivity/specific conductance standard.

3. Select monitoring/run mode. Wait until the probe temperature has stabilized.

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4. Look up the conductivity value at this temperature from the conductivity versus temperature correction table usually found on the standard bottle or on the standard instruction sheet. You may need to interpolate the conductivity value between temperatures. Select calibration mode, then conductivity. Enter the temperature corrected conductivity value into the instrument.

5. Select monitoring/run mode. The reading should remain within manufacturer's specifications. If it does not, re-calibrate. If readings continue to change after re-calibration, consult manufacturer.

6. Read the specific conductance on the instrument and compare the value to the specific conductance value on the standard. The instrument value should agree with the standard within the manufacturer's specifications. If not, re-calibrate. If the re-calibration does not correct the problem, the probe may need to be cleaned or serviced by the instrument manufacturer.

7. Remove probe from the standard, rinse the probe with a small amount of the second conductivity/specific conductance standard (discard the rinsate), and place the probe into the second conductivity/specific conductance standard. The second standard will serve to verify the linearity of the instrument. Read the specific conductance value from the instrument and compare the value to the specific conductance on the standard. The two values should agree within the specifications of the instrument. If they do not agree, re-calibrate. If readings do not compare, then the second standard may be outside the linear range of the instrument. Use a standard that is closer, but above the first standard and repeat the verification. If values still do not compare, try cleaning the probe or consult the manufacturer.

8. When monitoring ground water or surface water, use the specific conductance readings.

OXIDATION/REDUCTION POTENTIAL (ORP)

The oxidation/reduction potential is the electrometric difference measured in a solution between an inert indicator electrode and a suitable reference electrode. The electrometric difference is measured in millivolts and is temperature dependent.

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Calibration or Verification Procedure

1. Allow the calibration standard (a Zobell solution) to equilibrate to ambient temperature.

2. Remove the probe from its storage container, and place it into the standard.

3. Select monitoring/run mode.

4. While stirring the standard, wait for the probe temperature to stabilize, then read the temperature.

5. Look up the millivolt (mv) value at this temperature from the millivolt versus temperature correction table usually found on the standard bottle or on the standard instruction sheet. You may need to interpolate millivolt value between temperatures. Select "calibration mode", then "ORP". Enter the temperature-corrected ORP value into the instrument.

6. Select monitoring/run mode. The readings should remain unchanged within manufacturer's specifications. If they change, re-calibrate. If readings continue to change after re-calibration, consult manufacturer.

7. If the instrument instruction manual states that the instrument is factory calibrated, then verify the factory calibration against the standard. If they do not agree within the specifications of the instrument, the instrument will need to be re-calibrated by the manufacturer.

TURBIDITY

The turbidity method is based upon a comparison of intensity of light scattered by a sample under defined conditions with the intensity of light scattered by a standard reference suspension. A turbidimeter is a nephelometer with a visible light source for illuminating the sample and one or more photo-electric detectors placed ninety degrees to the path of the light source.

Some instruments will only accept one standard. For these instruments, the standards will serve as check points.

Calibration Procedures

1. Allow the calibration standards to equilibrate at the ambient temperature. The use of commercially available polymer primary standards (AMCO-AEPA-1) is preferred, however, the standards can be prepared using Formazin according to the EPA analytical Method 180.1.

2. If the standard cuvette is not sealed, rinse a cuvette with deionized water. Shake the cuvette to remove as much water as possible. Do not wipe dry the inside of the cuvette because lint from the wipe may remain in the cuvette. Add the standard to the cuvette.

3. Before performing the calibration procedure, make sure the cuvettes are not scratched and the outside surfaces are dry, free from fingerprints and dust. If the cuvette is scratched or dirty, discard or clean the cuvette respectively.

4. Zero the instrument by using either a zero or 0.02 NTU standard. A zero standard (approximately 0 NTU) can be prepared by passing distilled water through a 0.45 micron pore size membrane filter.

5. Using a standard in the range of 5 - 20 NTUs, calibrate according to manufacturer's instructions or verify calibration if instrument will not accept a second standard. If verifying, the instrument should read standard value to within the specifications of the instrument. If the instrument has range of scales, check each range that will be used during the sampling event with a standard that falls within that range.

7. Using a standard between 20 and 100 NTUs, calibrate according to manufacturer's instructions or verify calibration if instrument does not accept a third standard. If verifying, the instrument should read standard value to within the specifications of the instrument. If the instrument has range of scales, check each range that will be used with the proper standard for that scale.

IV. DATA MANAGEMENT AND RECORDS MANAGEMENT

All calibration records must be documented in the project's log book. At a minimum, include the instrument manufacturer, model number, instrument identification number, standards used to calibrate the instruments (including source), calibration date, and the instrument readings.

draft

References

Standard Methods for the Examination of Water and WasteWater, 19th edition, 1995.

Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, Revised March 1983.

Turbidity - Methods for the Determination of Inorganic Substances in Environmental Samples, EPA/600/R-93/100, August 1993.

Oxygen Solubility at Indicated Pressure

Temp.	Pressure (Hg)							
	760	755	750	745	740	735	730	mm
°C	29.92	29.72	29.53	29.33	29.13	28.94	28.74	in
0	14.57	14.47	14.38	14.28	14.18	14.09	13.99	mg/l
1	14.17	14.08	13.98	13.89	13.79	13.70	13.61	
2	13.79	13.70	13.61	13.52	13.42	13.33	13.24	
3	13.43	13.34	13.25	13.16	13.07	12-98	12.90	
4	13.08	12.99	12.91	12.82	12.73	12.65	12.56	
5	12.74	12.66	12.57	12.49	12.40	12.32	12.23	
6	12.42	12.34	12.26	12.17	12.09	12.01	11.93	
7	12.11	12.03	11.95	11.87	11.79	11.71	11.63	
8	11.81	11.73	11.65	11.57	11.50	11.42	11.34	
9	11.53	11.45	11.38	11.30	11.22	11.15	11.07	
10	11.28	11.19	11.11	11.04	10.96	10.89	10.81	
11	10.99	10.92	10.84	10.77	10.70	10.62	10.55	
12	10.74	10.67	10.60	10.53	10.45	10.38	10.31	
13	10.50	10.43	10.36	10.29	10.22	10.15	10.08	
14	10.27	10.20	10.13	10.06	10.00	9.93	9.86	
15	10.05	9.98	9.92	9.85	9.78	9.71	9.65	
16	9.83	9.76	9.70	9.63	9.57	9.50	9.43	
17	9.63	9.57	9.50	9.44	9.37	9.31	9.24	
18	9.43	9.37	9.30	9.24	9.18	9.11	9.05	
19	9.24	9.18	9.12	9.05	8.99	8.93	8.87	
20	9.06	9.00	8.94	8.88	8.82	8.75	8.69	
21	8.88	8.82	8.76	8.70	8.64	8.58	8.52	
22	8.71	8.65	8.59	8.53	8.47	8.42	8.36	
23	8.55	8.49	8.43	8.38	8.32	8.26	8.20	
24	8.39	8.33	8.28	8.22	8.16	8.11	8.05	
25	8.24	8.18	8.13	8.07	8.02	7.96	7.90	
26	8.09	8.03	7.98	7.92	7.87	7.81	7.76	
27	7.95	7.90	7.84	7.79	7.73	7.68	7.62	
28	7.81	7.76	7.70	7.65	7.60	7.54	7.49	
29	7.68	7.63	7.57	7.52	7.47	7.42	7.36	
30	7.55	7.50	7.45	7.39	7.34	7.29	7.24	
31	7.42	7.37	7.32	7.27	7.22	7.16	7.11	
32	7.30	7.25	7.20	7.15	7.10	7.05	7.00	
33	7.08	7.13	7.08	7.03	6.98	6.93	6.88	
34	7.07	7.02	6.97	6.92	6.87	6.82	6.78	
35	6.95	6.90	6.85	6.80	6.76	6.71	6.66	
36	6.84	6.79	6.76	6.70	6.65	6.60	6.55	
37	6.73	6.68	6.64	6.59	6.54	6.49	6.45	
38	6.63	6.58	6.54	6.49	6.44	6.40	6.35	
39	6.52	6.47	6.43	6.38	6.35	6.29	6.24	
40	6.42	6.37	6.33	6.28	6.24	6.19	6.15	
41	6.32	6.27	6.23	6.18	6.14	6.09	6.05	
42	6.22	6.18	6.13	6.09	6.04	6.00	5.95	
43	6.13	6.09	6.04	6.00	5.95	5.91	5.87	
44	6.03	5.99	5.94	5.90	5.86	5.81	5.77	
45	5.94	5.90	5.85	5.81	5.77	5.72	5.68	

(Continued)

Source: Draft EPA Handbook of Methods for Acid Deposition Studies, Field Operations for Surface Water Chemistry, EPA/600/4-89/020, August 1989.

Oxygen Solubility at Indicated Pressure (continued)

Temp.	Pressure (Hg)								
	725	720	715	710	705	700	695	690	mm
°C	28.54	28.35	28.15	27.95	27.76	27.56	27.36	27.17	in
0	13.89	13.80	13.70	13.61	13.51	13.41	13.32	13.22	mg/l
1	13.51	13.42	13.33	13.23	13.14	13.04	12.95	12.86	
2	13.15	13.06	12.07	12.88	12.79	12.69	12.60	12.51	
3	12.81	12.72	12.63	12.54	12.45	12.36	12.27	12.18	
4	12.47	12.39	12.30	12.21	12.13	12.04	11.95	11.87	
5	12.15	12.06	11.98	11.89	11.81	11.73	11.64	11.56	
6	11.84	11.73	11.68	11.60	11.51	11.43	11.35	11.27	
7	11.55	11.47	11.39	11.31	11.22	11.14	11.06	10.98	
8	11.26	11.18	11.10	11.02	10.95	10.87	10.79	10.71	
9	10.99	10.92	10.84	10.76	10.69	10.61	10.53	10.46	
10	10.74	10.66	10.59	10.51	10.44	10.36	10.29	10.21	
11	10.48	10.40	10.33	10.28	10.18	10.11	10.04	9.96	
12	10.24	10.17	10.10	10.02	9.95	9.88	9.81	9.46	
13	10.01	9.94	9.87	9.80	9.73	9.66	9.59	9.52	
14	9.79	9.72	9.65	9.68	9.51	9.45	9.38	9.31	
15	9.58	9.51	9.44	9.58	9.31	9.24	9.18	9.11	
16	9.37	9.30	9.24	9.17	9.11	9.04	8.97	8.91	
17	9.18	9.11	9.05	8.98	8.92	8.85	8.79	8.73	
18	8.99	8.92	8.86	8.80	8.73	8.67	8.61	8.54	
19	8.81	8.74	8.68	8.62	8.56	8.49	8.43	8.37	
20	8.63	8.57	8.51	8.45	8.39	8.33	8.27	8.21	
21	8.46	8.40	8.34	8.28	8.22	8.16	8.10	8.04	
22	8.30	8.24	8.18	8.12	8.06	8.00	7.95	7.89	
23	8.15	8.09	8.03	7.97	7.91	7.86	7.80	7.74	
24	7.99	7.94	7.88	7.82	7.76	7.71	7.65	7.59	
25	7.85	7.79	7.74	7.68	7.60	7.57	7.51	7.46	
26	7.70	7.65	7.59	7.54	7.48	7.43	7.37	7.32	
27	7.57	7.52	7.46	7.41	7.35	7.30	7.25	7.19	
28	7.44	7.38	7.33	7.28	7.22	7.17	7.12	7.06	
29	7.31	7.26	7.21	7.15	7.10	7.05	7.00	6.94	
30	7.19	7.14	7.08	7.03	6.98	6.93	6.88	6.82	
31	7.06	7.01	6.96	6.91	6.86	6.81	6.76	6.70	
32	6.95	6.90	6.85	6.80	6.70	6.70	6.64	6.59	
33	6.83	6.78	6.73	6.68	6.83	6.58	6.53	6.48	
34	6.73	6.68	6.63	6.58	6.53	6.48	6.43	6.38	
35	6.61	6.56	6.51	6.47	6.42	6.37	6.36	6.27	
36	6.51	6.46	6.41	6.36	6.31	6.27	6.22	6.17	
37	6.40	6.35	6.31	6.26	6.21	6.16	6.12	6.07	
38	6.30	6.26	6.21	6.16	6.12	6.07	6.02	5.98	
39	6.26	6.15	6.11	6.06	6.01	5.97	5.92	5.87	
40	6.10	6.06	6.01	5.96	5.92	5.86	5.83	5.78	
41	6.00	5.96	5.91	5.87	5.82	5.78	5.73	5.69	
42	5.91	5.86	5.82	5.77	5.73	5.69	5.64	5.60	
43	5.82	5.78	5.73	5.69	5.65	5.60	5.56	5.51	
44	5.72	5.68	5.64	5.59	5.55	5.51	5.46	5.42	
45	5.64	5.59	5.55	5.51	5.47	5.42	5.38	5.34	

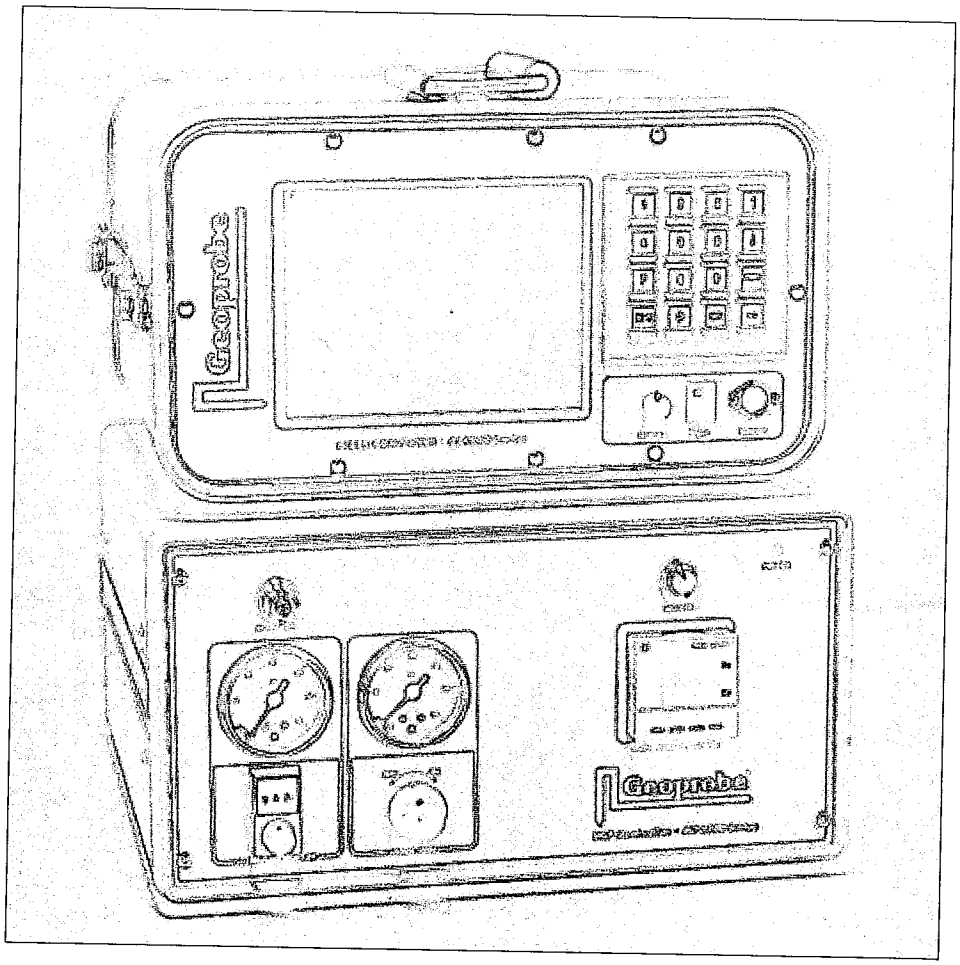
Source: Draft EPA Handbook of Methods for Acid Deposition Studies. Field Operations for Surface Water Chemistry. EPA/600/4-89/020, August 1989.

DIRECT IMAGE®

FC4000 Field Instrument
MP3500 MIP Controller

User's Guide

Geoprobe® Manual No. 20777



FC4000 Field Instrument and MP3500 MIP Controller

Geoprobe® Systems

Geoprobe® Systems

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Section 1.0 General Introduction

Section 1.1 Introduction

The purpose of this manual is to address aspects of Direct Image® safety, hardware, software as well as general system setup. This manual is not intended to be a complete field manual for Direct Image® equipment, but rather a tool to assist the user in general Direct Image® practices.

Section 1.2 Manual Contents

Section 2.0 discusses the FC4000 Field Instrument made by Geoprobe® Systems. Topics covered include: general specifications, connections, component identification, and use as an electrical conductivity (EC) logging tool. Section 3.0 discusses the MP3500 Membrane Interface Probe (MIP) controller made by Geoprobe® Systems. Topics covered include: safety, general specifications, connections, component identification, and use while logging. The Appendix sections include reference information about the MP3500 instrument, MP3500 internal gas plumbing, keystroke navigation diagrams for the Direct Image® EC and MIP FC4000 logging packages, and a consolidated list of all diagrams, tables, and figures in this document.

Section 2.0 FC4000 Series Field Instrument

Section 2.1 Introduction

The FC4000 consists of rugged laptop components together with conductivity instrumentation in a single unit. This combination yields a reliable and easy to use field instrument. All connections to the FC4000 are compatible with existing Geoprobe® brand conductivity equipment. An integrated front mounted sixteen key keypad takes all user inputs for logging. The 7.1-inch sunlight readable "transflective" LCD and a front-mounted contrast control creates an easy viewing environment inside or outside. A rear-mounted, 1.44MB, 3.5-inch floppy drive is used to remove log files. Instrument testing and configuration is handled through built in self test and configuration features.

The Geoprobe® FC4000 Field Instrument is used for Electrical Soil Conductivity logging to define soil lithology and refining sampling plans. The FC4000 is intended to be used with standard Geoprobe conductivity accessories to perform conductivity logs. Using this system, a four-pole Wenner Style probe, approximately 1.5 in. in diameter by 12-in. long, is hydraulically driven into the subsurface. The driving mechanism is a Geoprobe® brand soil probing machine capable of applying a percussion type force to the top of the probe rod at a rate of 30 Hz.

A signal cable is attached to the Wenner probe then run through the inner diameter of the probe rod. From this point an adapter cable is fastened to the probe cord and attached to the Field Instrument. This "rod-string" is then stored in racks on a specially designed rod cart.

Depth measurement is obtained from the stringpot (Linear Position Transducer-80 in. travel) system configured to measure the distance from the driving mechanism to ground surface. When a length of probe rod is driven, a change in string length is indicative of the probe's progression through the soil. The stringpot signal is proportional to the length of the string. A signal cable is fastened to one end of the stringpot and the other to the Field Instrument for monitoring. The stringpot signal is used to determine downhole probe position and the rate at which the probe is moving.

The Geoprobe® FC4000 Field Instrument provides a "real-time" display of conductivity versus depth during probing. Data is stored to an internal "FLASH" memory chip every 0.05 ft. of log depth, in an ASCII tab delimited, spreadsheet format that can be accessed at a later time.

The FC4000 power requirements are 90 VAC to 240 VAC @ 3.0A. Optional: Geoprobe® Systems can supply a 12Vdc inverter that delivers 120 VAC at 200 watts from a 12 V lighter outlet (P/N SC152). The FC4000 is FCC Class B and CISPR Class B certified.

Dimensions (length x width x height): 14.0 in. x 14.0 in. x 9.0 in. (356 mm x 356 mm x 229 mm).

Weight: 18.5 lb. (8.3 kg).

Finish: Gray molded ABS plastic with yellow front and back overlays. Gray molded ABS plastic front and back transport covers with carrying handles.

Mounting hardware: Geoprobe® Systems can supply a mounting fixture for fastening the FC4000 to a Geoprobe® brand probe machine. This hardware is optional and not necessary for system function.

Section 2.2 Front Panel

The front panel consists of an LCD, keypad, contrast control, trigger switch, and optional keyboard connector. See Figure 1 for component identification.

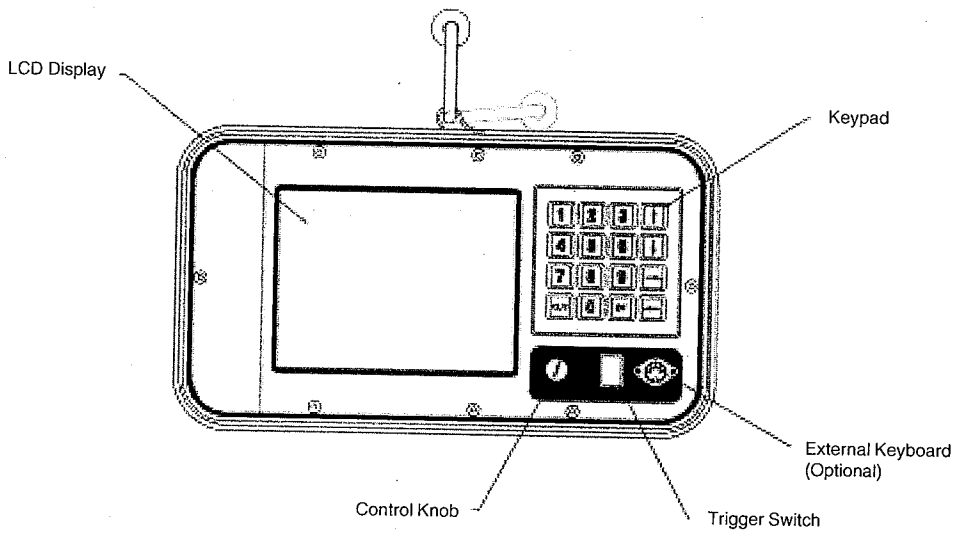


FIGURE I
FC4000 Field Instrument Front Panel

Section 2.3 Rear Panel

The rear panel is where all cables or cords are connected. Note the location of the connectors and floppy drive. The 3.5-in. floppy disk drive is protected with an aluminum spring-loaded door. The power entry module houses the system ON/OFF switch and a fuse for unit protection. The serial port and parallel port are available for future applications. See Figure 2 for component identification.

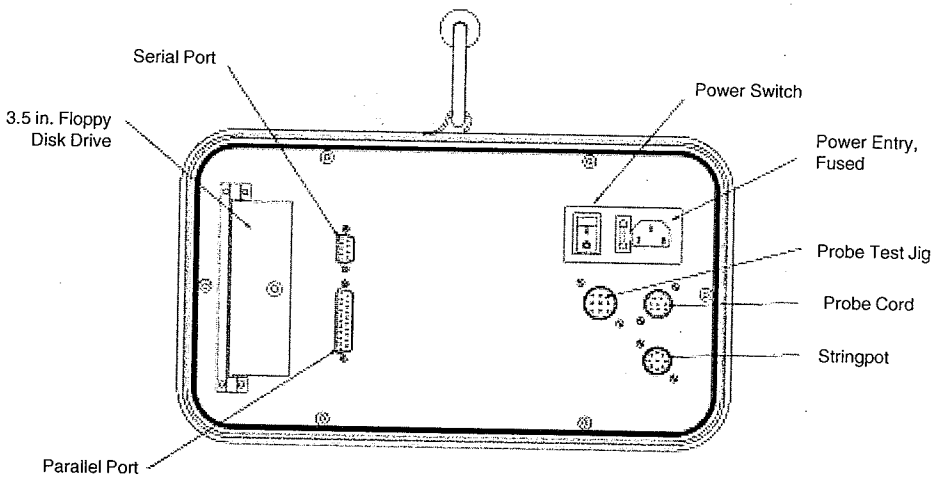


FIGURE 2
FC4000 Field Instrument Rear Panel

Section 2.4 Attaching cords

A few signal cords and a power cord need to be attached to the Field Instrument before operation can begin. First, plug in the power cord. This will deliver 120VAC line power to the instrument. An option here is to use a 200 watt inverter attached to the 12VDC power point on a Geoprobe® direct push machine or vehicle to obtain 120VAC. Second, attach the probe cord and test jig cord to the rear panel of the instrument. The probe connection will be used to obtain electrical conductivity information and the test jig cord will be used when the probe test function is performed. Last, attach the stringpot cord. The instrument will use this connection to track log depth.

Section 2.5 FC4000 Power Up

The power switch is located on the rear panel of the Field Instrument. Turn the switch "on" and the system will start. A contrast knob located on the front panel can be used to adjust for readability. The FC4000 startup screen displays a menu of Direct Image® software options. The discussion of operation for each software package is left for the specific manual section covering that topic.

Section 2.6 Electrical Conductivity Logging Function Setup and Use

Section 2.6.1 Introduction

The purpose of this section is to discuss the use of the Geoprobe® brand FC4000 Field Instrument in Electrical Conductivity (EC) mode.

The EC functionality ships standard with every FC4000, and allows the direct use of standard EC Geoprobe® Systems products.

Section 2.6.2 HARDWARE CONNECTION

EC logging is accomplished by first making the necessary connections to the FC4000. Refer to Figure 3 for basic EC connections. Following is a summary of these connections.

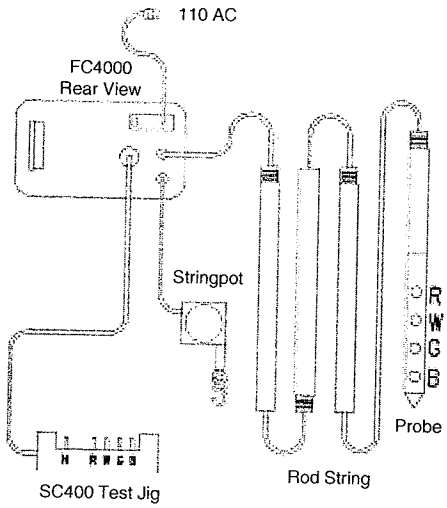


FIGURE 3
Electrical Conductivity System Connections
(SC400 Conductivity Probe Shown)

PROBE CONNECTION: Connect the probe rod cord set to the **PROBE** connector on the back of the field instrument.

STRINGPOT CONNECTION: Connect the stringpot cord to the **STRINGPOT** connector on the back of the field instrument.

TEST JIG CONNECTION: Connect the SC400 test jig (if used) to the **TEST INPUT** connector on the back of the field instrument. The test jig allows for easy testing of the SC400 probe, but it is not required for logging

Section 2.6.3 DATA STORAGE

Data is stored to a "FLASH" memory chip within the FC4000 every 0.05 feet of logging in a tab delimited ASCII spreadsheet format. The FC4000 instrument "FLASH" memory is capable of storing over two hundred 45-ft EC logs without removing any logs. It should be noted that the FC4000 is a field instrument and should not be used as a permanent storage location for logs. A good practice would be to keep no more than one day's worth of logs on the FC4000 and to remove the logs at the end of the day with the 3.5-in. floppy drive located on the back of the instrument. Once removed, the logs can be transferred to another storage device for safe keeping, organizing, and archiving. If the "FLASH" memory is allowed to fill up with logs, the software will not allow a log to begin and will instruct the user to clear the unwanted logs off using the **SAVE/DEL/PRT LOGS** menu option. The FC4000 names the log files automatically with an "EC" prefix and a consecutive numbering scheme starting at 0001. The consecutive numbering scheme may be reset to 1 (one) by the user through the Configuration Screen. Conductivity (EC) data is stored to a **data** file with a **.DAT** extension. The **.DAT** file has five columns of data. Following is a representation of the data columns as they appear in the **.DAT** file:

DEPTH (FT) **EC (mS/M)** **RATE OF PROBING (ft/min)** **DEPTH (M)** **RATE OF PROBING (M/min)**

Example name: EC0050.DAT
Logging information is stored to an **information** file with a **.INF** extension. This file includes the probe type used for the log, start time, end time and the date of the log.

Example name:EC0050.INF

Section 2.6.4 Logging

The FC4000 utilizes an embedded application to acquire, display and store EC data. This section discusses the layout and the use of this function. Before logging, the FC4000 needs basic information about the EC setup. This information is collected through a simple series of menu oriented screens.

Following is a description of each screen with the displayed FC4000 text in **bold** and the narrative comments in *italics.* Refer to Appendix VI for software operation control keys. The narrative comments are not shown on the instrument screen.

The following menu is the first menu displayed when the FC4000 is in EC mode:

Select an Option:

1 **For Conducitivity** ..... *EC LOGGING*
2 **For Save/Del/PRT Logs** ..... *Working with log files*
3 **DS Menu** ..... *Return to DS Main Menu*

Select Option 1 by pressing the '1' key on the FC4000 keypad.

NOTE: A hidden configuration menu exists only while the above screen is displayed. The Configuration Screen is accessable by pressing the **RIGHT ARROW** key, releasing it then pressing the **CE/C** key within two seconds. The configuration menu is shown below:

CURRENT SETTINGS

ENGLISH UNITS

OPTIONS

1 **TO CHANGE TO METRIC UNITS**
2 **TO CLEAR ALL LOGS AND RESET LOG NAME**
3 **TO EXIT**

Option 1 toggles the FC4000 instrument between English and metric units. English and metric data is always stored. this setting only controls the displayed units during logging. Option 2 will permanently erase ALL logs on the FC4000 and reset the log name to '0001'. Option 3 allows the user to exit the configuration screen and return to the normal startup log screen.

Select a Probe Option:

1 **For SC400** ... *SC400 Probe*
2 **For SC310** ... *SC310 Probe*
3 **For SC300** ... *SC300 Probe*

Select Option 1 by pressing the '1' key on the FC4000 keypad.

Select **an Option:**

1 **To test instrument and Probe ..** *Test EC hardware*
2 **To bypass all tests .....** *Bypass the hardware tests*

Select Option 1 by pressing the '1' key on the FC4000 keypad.

NOTE: If Option 2 is selected no tests will be preformed. This may be desirable if the user knows the EC tool string to be in good working order.

When Option 1 is selected three tests are run. First are the Instrument Calibration Tests; second are the Probe Continuity Tests; and third are the probe isolation tests. The following is a description of each. For discussion in this section, probe poles will be referred to as "red" (R); "white" (W), "green" (G), and "black" (B). These colors are assigned for discussion purposes with red being the pole closest to the thread end of the probe descending to black for the pole closest to the point of the probe. The case of the probe will be referred to as "neutral" (N). Refer to Figure 3, Electrical Conductivity System connections for identification of this color scheme.

INSTRUMENT CALIBRATION TESTS:

INSTRUMENT **CALIBRATION 10 OHM TEST**
RESISTANCE **10.23 OHMS**

The above screen is repeated for the 100, 1000, and 2000 OHM instrument calibration tests. Conditions for failure of an associated test are listed below. The purpose of this test is to verify that the field instrument is calibrated.

10 OHM: MORE THAN +- 1 OHM
ALL OTHER TESTS: MORE THAN +-5%

PROBE **CONTINUITY R-R TEST**
RESISTANCE **2.03 OHMS**
OVER 8 **OHMS FAILS**

The above screen is repeated for the W-W, G-G and B-B connections. The purpose of this test is to verify the existance of a good electrical connection between the field instrument and each probe dipole.

PROBE **ISOLATION RED-NEUTRAL TEST**
RESISTANCE **160.23 KOHMS**
UNDER 15 **KOHMS FAILS**

The above screen is repeated for the R-W, R-G, R-B, W-N, W-G, W-B, G-N, G-B, and B-N tests. The purpose of this test is to verify that no dipoles are shorted together. Shorts can occur from damaged cables or connectors.

The next screen shown after the probe tests is:

SELECT THE ROD LENGTH USED

1	**FOR 1M RODS**
2	**FOR 2 ft RODS**
3	**FOR 3 ft RODS**
4	**FOR 4 ft RODS**
5	**FOR 5 ft RODS**

Select the appropriate rod length.

SELECT AN OPTION

1	**FOR WENNER**
2	**FOR TOP DIPOLE**
3	**FOR MIDDLE DIPOLE**
4	**FOR BOTTOM DIPOLE**

NOTE: Depending on the probe type selected and/or the outcome of the probe tests, some probe configurations may not be available.

The EC screen is then shown (see Figure 4), and logging may begin once the trigger is turned on. The trigger switch is located on the front panel of the FC4000 and controls when log data is collected, stored and displayed. No logging will take place unless the trigger switch is in the on position and the "**TRIGGER ON**" message appears on the logging screen.

During logging, the EC log is displayed on a single user screen, the EC screen.

EC Screen: Refer to Figure 4. This screen shows EC logging information and consists of two (2) graphs. The top graph is EC. This graph is an auto-scaled representation of the conducitivity during logging in milliSeimens per meter (mS/M). The bottom graph is probe speed in auto-scaled ft/min units. Both graphs are updated on 0.05ft advancements of the probe and have a default maximum depth of 40 feet. This screen auto-scales the depth once the default 40ft depth is reached in additional 20-ft increments. This allows additional logging while preserving the complete log on the screen for viewing.

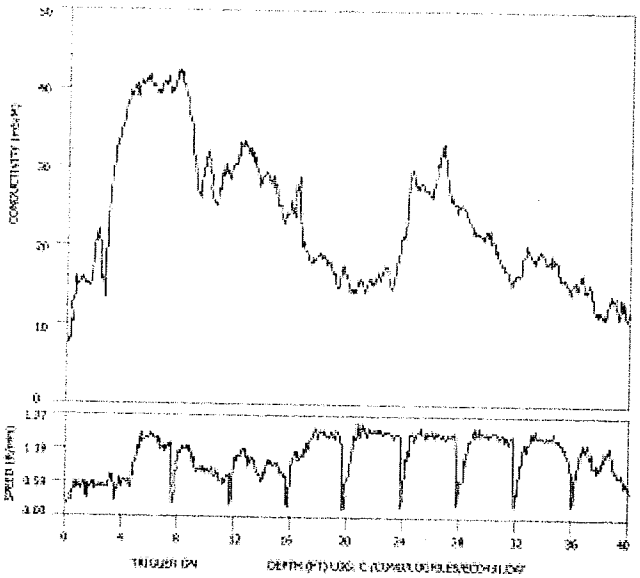


FIGURE 4
Electrical Conductivity (EC) Logging Screen

Section 2.7 Working with Files

Option 2 of the main screen is used to perform file deleting, copying to disk, viewing, and printing. "Files" are defined as the data sets generated while logging that are stored in a spreadsheet ASCII format on the FLASH chip inside the field instrument. These "files" are selected, deleted, copied, viewed, and printed in the following manner:

select any of the files or logs listed by pressing the number on the keypad that corresponds to the appropriate file name

****press ENT to save the file to disk**

****press CE/C to delete the file from the flash disk**

****press 1 to graph the file or log**

press the "PRINT" key to print this log

NOTE: *Printing can only be done if the appropriate EPSON printer, specified by Geoprobe® Systems, is connected.*

****press ENT for more logs (additional listing)****
****press CEC to end listing****

NOTE: To protect data, copy each log to a 3.5 in. floppy disk daily. Always have a backup copy or two of the data files. Use the "**SAVE/DEL/PRT**" logs option to save data files to disk.

Section 3.0 MP3500 Series MIP Controller

Section 3.1 Introduction

The Membrane Interface Probe (MIP) System, manufactured by Geoprobe® Systems, is used for the detection and measurement of volatile organic compounds (VOCs) in the subsurface. Using this system, a heated probe carrying a permeable membrane is advanced to depth in the soil. VOCs in the subsurface, which cross the membrane, enter into a carrier gas stream and are swept to gas phase detectors at ground surface for measurement.

The MP3500 Series MIP Controller is the main operating component for the MIP contaminant detection system. The MP3500 Series Controller regulates gas flow and temperature in the MIP system, and provides digital conversion of analog signals from detectors and other internal devices used with the MIP system.

The MIP Controller consists of the following main components.

- A step-down transformer to convert the designated input voltage to 50 VAC for MIP probe heating.
- A gas flow regulation system to provide a constant carrier gas flow to the probe.
- A temperature measurement and control system to regulate MIP probe temperature.
- An analog to digital conversation system to output digital data via serial cable to an FC4000 Field Instrument.

There are two separate models of the MIP Controller which vary in their input voltages. The two models and their respective input voltages are shown in Table 3.1.

Table 3.1
MIP 3500 Series Controllers
Model Numbers and Input Voltages

Model	Area of Use	Input Voltages
3500	U.S. and Canada	100 VAC
3503	Europe	220, 230, or 240 VAC

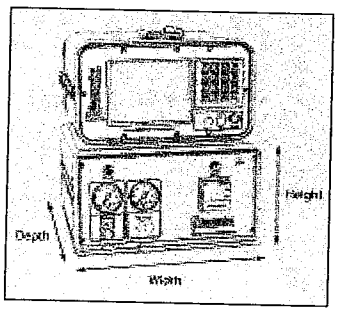
The MIP controller (Figure 5) is an electrical device, which controls the gas flow and the temperature of the MIP probe. The MIP system also measures soil electrical conductivity via the dipole electrode on the probe. The conductivity feature is controlled and monitored by the FC4000 directly. The MIP controller has inputs for analog signal (differential inputs 5VDC or less) from gas phase chemical detectors that are used in conjunction with the MIP system. Trunkline pressure and probe temperature are electronically monitored inside the MP3500 controller. This data is logged and displayed by the FC4000 Field Instrument.

Dimensions and weight of the FC4000 Field Instrument:

Depth: 14.0 in. (356 mm)
Additional 1.5 in. (38 mm) front panel clearance required
Additional 5.0 in. (127 mm) rear panel clearance required
Width: 14.0 in. (356 mm)
Additional 1.0 in. (25 mm) clearance recommended
Height: 9.0 in. (229 mm) clearance recommended
Weight: ..... 18.5 lb. (8.3 kg) (does not include shipping case)

Dimensions and weight of the MIP controller:

Depth: 15 in. (381 mm)
Additional 1.5 in. (38 mm) front panel clearance required
Additional 3.0 in. (127 mm) rear panel clearance required



FC4000 Field Instrument (top)
MP3500 Controller

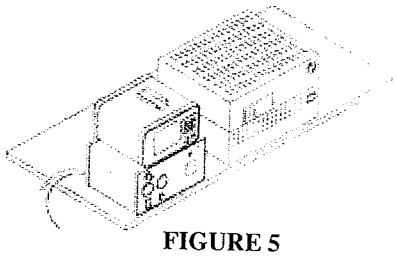


FIGURE 5
MP3500 Series MIP Controller
The MIP can be interfaced to variety of gas chromatography type detectors.

Width: 15 in. (356 mm)
(Additional 1 in. (25 mm) clearance recommended
Height: 7.75 in. (197 mm)
(Additional 4.0 in. (102 mm) clearance recommended
Weight: ..... 31.05 lb. (14.0 kg) (does not include shipping case)

Section 3.2 Safety Precautions

Operator safety is important when adjusting, testing or handling the MP3500 Controller. Although measures have been taken to protect or guard against operator injury, care should be used whenever working with the Membrane Interface Probe Controller. This section describes some of the electrical guarding and protection devices that are used, as well as lists some safety precautions.

CAUTION: NEVER override the **circuit breaker**.

NOTE: This device is intended to trip or "stop current flow" when the circuit is overloaded or a short circuit has occurred on the output device (MIP probe). This device will protect the user and equipment from line-to-line contact hazards.

CAUTION: NEVER override the **GFCI** *(Ground Fault Circuit Interrupter).*

NOTE: The GFCI is designed to provide protection for personnel and equipment when leakage levels have a potentially lethal ground current in excess of six milliamperes. A GFCI is simply a fast-acting circuit breaker that senses small imbalances in the circuit caused by current leakage to ground. In a fraction of a second the GFCI trips and shuts off the electricity. This device protects the operator against the most common shock hazard – the ground fault.

Do not disassemble or modify the unit.

- Use the unit for the purpose for which it is intended.
- For repairs, contact the Technical Service staff at Geoprobe® Systems.
- Never remove the cover of the instrument case with the power ON.
- Be aware of the 50VAC supply voltage on the heater output. Although, by electrical standards this supply is considered to be a low voltage power source, care should be taken when connecting to this output.
- Never have power ON when connecting to the 50VAC output.
- Be aware of the weight of the instrument before lifting.
- Be aware of the weight distribution inside the instrument to avoid mishandling or dropping.
- Be aware of static discharge when troubleshooting or working on the instrument with the cover off. Work on a static mat or use a static discharge wristband whenever possible.

Section 3.3 Front Panel

The front panel of the MIP Controller is show in Figure 6. The front panel is divided into the following sections.

Right section: System power, probe temperature control, and temperature readout.

Left section: Probe gas flow regulation.

System Power: The system power pilot light will indicate when the controller has been connected to the line power and the power switch on the rear panel of the controller has been turned on.

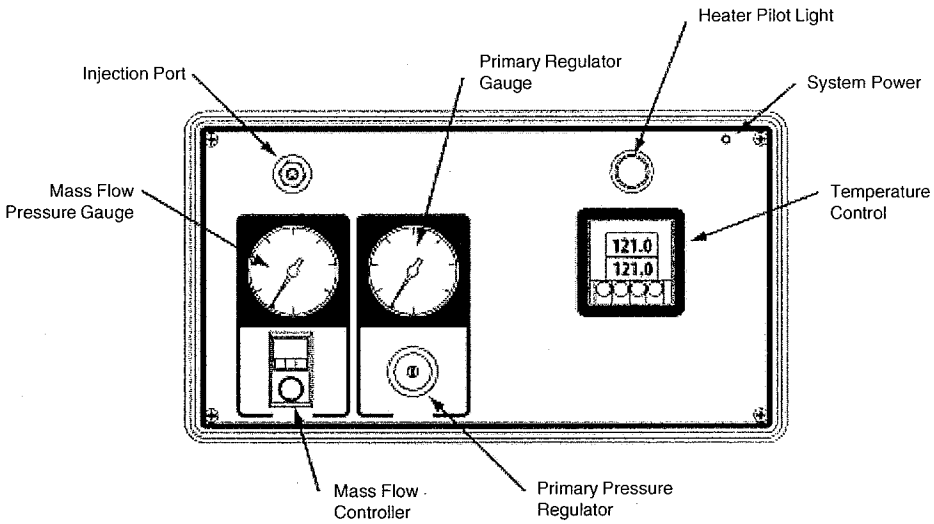


FIGURE 6
MIP MP3500 Series Controller Front Panel

Probe heater and temperature controls: The righthand side of the front panel contains the probe heater controls. Again, refer to Figure 6.

Heater Pilot Light: The heater pilot light is on when the controller closes the probe heater relay in order to supply electrical current to the probe. This pilot does not indicate that the probe is actually connected to and receiving current from the controller. The pilot only indicates that the controller relay has closed and therefore heater power is being supplied to the MP3500 back panel. The set point for the probe temperature is pre-set at the factory to 121 deg C. The controller uses a 'on/off' control scheme. The heater will be on until the probe reaches the set point, the it shuts off. With the heater off, the temperature will momentarily continue to rise and then begin to fall. When the temperature falls to less than the set point, the controller circuit again closes to provide heat to the probe.

Temperature Control: This modular controller displays two temperatures: the top display is the actual temperature of the probe displayed in degrees C, the bottom display is the temperature set point in degrees C. The set point is pre-set at the factory to 121 deg C. This modular controller is a proportional type controller and provides close tolerance regulation of the probe temperature. If the thermocouple goes open, then the temperature controller unit will display a flashing error message (r5t, op5n) and the controller will not supply current to the probe heater. This controller also outputs an analog voltage representative of the probe temperature for logging on the FC4000 Field Instrument.

Gas Flow Controls: These controls are located on the left side of the front panel. A diagram of the internal gas plumbing of the MIP controller is shown in Appendix III.

Gas flow controls and their functions are as follows:

Primary Pressure Regulator: Carrier gas connected to the controller first passes through the primary regulator. The purpose of this regulator is the supply gas at constant pressure to the mass flow controller. Besides being a pressure regulator, this device is also an on/off valve. Carrier gas (nitrogen, helium, hydrogen, argon, or compressed air depending upon the detector used with the system) should be supplied to the rear panel of the controller at 30 to 60 psi (2 to 4 bars). The primary regulator should be adjusted to 20 psi (1.4 bars). Regulation of this valve is performed by first opening the valve by rotating the black knob all the way to the right (clockwise). With the regulator open, the output pressure is adjusted by using a flat screwdriver in the stem embedded in the center of the knob. Output pressure for this regulator is indicated on the pressure gauge located immediately above the regulator.

Be sure to use only light finger pressure to open or close the regulator valve.

It is good practice to shut this valve off at the end of each working day. To do this, rotate the black knob all the way to the left (counterclockwise). By opening the valve all the way to the right the regulator will return to its previously adjusted pressure setting.

Mass Flow Controller: The purpose of this controller is to provide a constant flow of carrier gas through the trunkline. This device works by opening or closing a feed valve in response to changes in downstream friction losses or restrictions. Clockwise rotation of the controller knob will increase carrier flow and visa versa. The numerical counter on this controller is a unitless indicator of controller position.

The pressure gauge immediately above the mass flow controller indicates the gas pressure on the downstream (probe) side of the mass flow controller. The mass flow controller is normally set so that the output pressure will be between 7 and 10 psi (0.47 to 0.68 bar) for a normal, clean, 100 ft (30 m) trunkline and probe. Mass flow controller settings in this range will yield carrier gas flow rates of 25 to 40 mL/min and result in butane (FID detector) return times of 20 to 50 seconds. A graph of typical gas flow rates, mass flow controller pressures, and butane trip times is show in Figure 7.

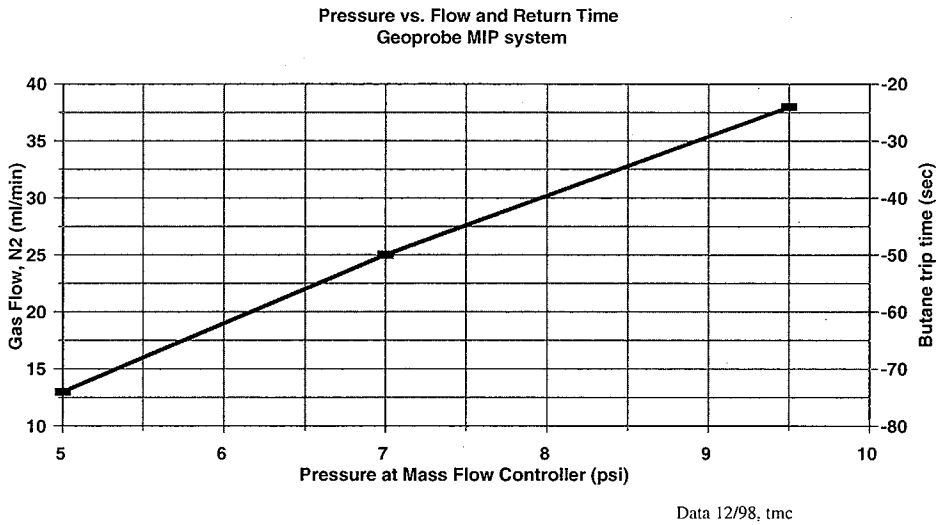


FIGURE 7
Pressure vs Flow and Return Time

The mass flow controller gauge is very important during field operations and should be checked at least once per rod. An increase in this pressure indicates that some downstream blockage has occurred (particulate or water in the line) and the mass flow controller is increasing pressure in order to maintain flow. A decrease in pressure at the mass flow controller indicates that a leak has occurred in the system.

In general, the mass flow controller pressure gauge will not change by more than one psi during MIP logging. If it does change by more than one psi, there is a problem in the system. Logging should be halted, the probe removed, and the system should be examined (see Section 3.10.2: Carrier Gas Flow Circuit Troubleshooting). If the mass flow controller pressure rises to match the primary pressure, then it is safe to assume that the system has a complete blockage and must be examined and corrected.

The MP3500 Controller has an internal electronic pressure sensor that monitors the probe side trunkline pressure. The pressure data is sent to the FC4000 Field Instrument, and displayed as a digital readout on the Real Time detector graph (see Figure 10) while running the MIP software. Pressure data is also saved while logging to the .DAT file on a 0.05-ft increment.

Injection Port: This is a septum-type injection port which is placed in the carrier gas line before it leaves the control box. Gas phase standards can be injected in this port using needle syringes. A standard injected in this port would travel in the carrier gas stream to the MIP probe and back to the detector. It is important that the injection port nut not be tightened too tight, as this will damage the septum and block carrier flow. When replacing the nut simply screw it on finger tight and check the installation for leaks using soap bubbles.

Section 3.4 Rear Panel

The layout of the MP3500 rear panel is shown in Figure 8. The rear panel is used mainly for connection of cables to line power and trunkline inputs. Gas connections are also made at this panel. Each connector or fitting is discussed in this section.

Input Power Label: The line voltage required for the MIP controller is clearly shown on this label. Do not attempt to connect the controller to line voltages other than the voltage indicated on this label. Use of improper input line voltage could result in damage to the equipment.

Unit Power Connection: Connect only to voltage indicated on the input power label. MP3500 Series MIP Controllers vary in their power input requirements depending upon the indicated country of service. Input voltage requirements for the various models within the MP3500 series are shown in Table 3.1. All models are operable on either 50 or 60 Hz power sources.

Table 3.1
MIP 3500 Series Input Voltages

Model	Area of Use	Input Voltage
3500	U.S. and Canada	100 – 120 VAC
3503	Europe	200 – 240 VAC

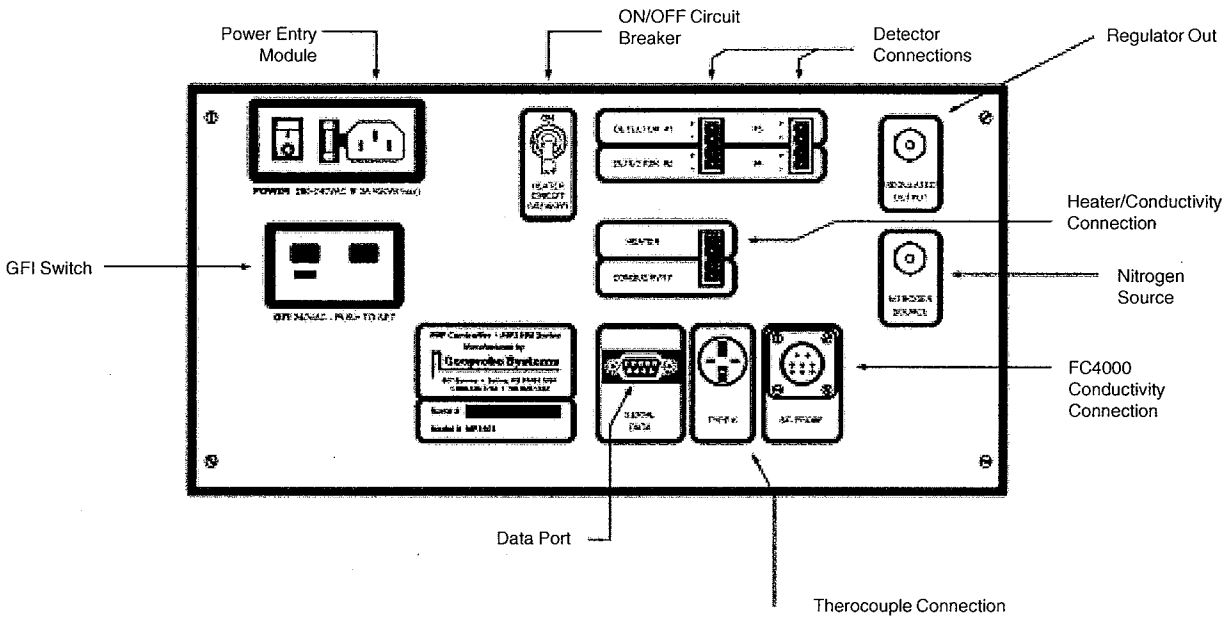


FIGURE 8
MP3500 Series Controller Rear Panel

Fuse Holder: Power to the controller is fused at the input point. Fuse size varies with the designated input voltage as shown in Table 3.2. A spare fuse is provided within the power connection block.

Table 3.2
Input Power Fuse Size

Input Voltage	Fuse Size
100 - 120 VAC	10 Amp
200 - 240 VAC	6.3 Amp

GFI Switch The purpose of this switch is to detect any current leakage to ground that may occur from the probe and automatically disconnect from power if such leakage occurs. To test the operation of the GFI, push the "test" button. This will cut the power to the probe heater circuit only and not the rest of the MIP controller. Pressing the "reset" button will restore power to the heater circuit. A green indicator light on the GFI switch indicates that the power is on.

Data Port: This port accepts a standard DB-9 male connector and is used to transfer digital data from the analog to digital (A/D) converter in the controller to a FC4000 Field Instrument. Data transferred via this port includes: probe temperature, detector response 1, 2, 3, and 4, and trunkline pressure.

On/Off Circuit Breaker: This is both a switch and a safety circuit breaker on the primary side of the probe heater power transformer. This switch may be used to manually disconnect power to the probe (as may be needed when connecting or handling the probe). This switch must be in the "ON" position in order to heat the probe for MIP logging. If primary current to the probe heater power transformer exceeds four amps, this switch will automatically flip to the "OFF" position. This usually indicates that a direct short has occurred in either the probe or trunkline. If this situation occurs, do not attempt to hold this switch in the "ON" position as this will result in damage to the switch.

Detector Inputs: The MP3500 Series MIP Controller is designed to accept analog inputs from up to four gas phase detectors. These inputs accept signals in the range of 0-5VDC. This range is sufficient to work with most all detectors common to the market. Polarity is marked at each input. The MIP controller uses an internal 12 bit A/D to convert the detector signals to digital data sent to the FC4000 Field Instrument.

Heater and Conductivity Connector: The probe heater wires (yellow) from the MIP trunkline are connected to the top two lugs of this connector. The potential between these two lugs is 50 VAC. The dipole soil conductivity wires (one white and one red) from the MIP trunkline are connected to the bottom two lugs of this connector. Note that the red and white wires are interchangeable in these two lugs. There is a 0.75 VAC potential between these lugs.

Thermocouple Connection: This is a standard type K thermocouple connection which accepts the male connector attached to the end of the thermocouple wire in the MIP trunkline. When assembling the male thermocouple connector to the trunkline, note that the red wire goes to the negative pole, yellow to positive.

Regulated Out: This fitting is the outlet for the pressure and flow regulated output from the MIP controller. This fitting is normally equipped with a 1/16-in. Swagelok® brand fitting for attachment to one of the Teflon® tubes in the MIP trunkline. The MIP trunkline has a 1/8 in., brass, tube-type bulkhead fitting for this connection.

Nitrogen Source: This is the input port for carrier gas to the MIP system. This label is somewhat of a misnomer as any one of a number of carrier gasses may be connected to this port. This is a 1/8-in. Swagelok® fitting. Clean carrier gas should be supplied to this port at a regulated pressure of 30 to 60 psi (2 to 4 bars) through a 1/8 in. brass, tube-type bulkhead fitting.

Section 3.5 MIP Controller Connections

Section 3.5.1 Introduction

This chapter explains making connections to a basic field operation of the MP3503 MIP Controller. The first operation task is to make the various gas and electrical connections to the back panel of the controller. With this complete, the controller is turned on, the probe heated, and logging operations started.

Section 3.5.2 Attachment to Gas Supply

Connect a carrier gas supply to the input port labeled "Nitrogen Source" on the rear panel of the controller. This input port is a 1/8-in. Swagelok® fitting. One-eighth inch (3.175 mm) OD tubing of either stainless steel or Teflon® should be used from the carrier gas supply to the input port.

The choice of carrier gas used will depend upon the detector connected to the system. Selection of the proper carrier gas is the responsibility of the user and should be based on the requirements of the detector being connected to the system. The following carrier gases have been used for MIP work:

Nitrogen (most common)
Clean air (used with FID)
Helium
Argon

The carrier gas should be supplied to this port at a regulated pressure of 30 to 60 psi (200 to 400 kPa). Turn the pressure regulator control knob on the front panel of the controller to the off position before applying gas pressure to the controller. Apply gas pressure to the controller and check for leaks at this connection using a soap solution.

Section 3.5.3 Attachment to the Trunkline

The trunkline should be connected to the MP3500 controller in the following manner:

Regulated out gas line.

One of the 1/16-in. (1.6 mm) OD Teflon® tubes from the trunkline should be connected to the "Regulated Out" port on the back of the controller. A Swagelok® type, 1/16-in. fitting is provided for this purpose. The tubing should be inserted into the connection and tightened with a wrench approximately one-quarter turn. When pressure is applied to the trunkline, this fitting should be checked and, if necessary, re-tightened to stop leakage.

Heater and Conductivity Connection

A four-pin plug is provided at the back of the controller for connection of the trunkline heater and conductivity wires. The heater wires (yellow) attach to the top of this plug. These two wires are interchangeable. The conductivity wires (one red and one black) connect to the bottom two pins of this plug. The conductivity wires are also interchangeable.

Thermocouple Connection

A standard type K male thermocouple plug is provided with the controller for attachment of the trunkline thermocouple cable. A small, flat blade screwdriver is required to disassemble the plug and attach the thermocouple wires to their appropriate poles. The red wire should be attached to the negative pole of the plug and the yellow wire to the positive pole of the plug. Once assembled, the trunkline thermocouple cable can be plugged into the male (socket) thermocouple connector on the back of the instrument box.

Trunkline to Detector

The second gas line in the trunkline must be connected to the detector. The manner of attachment of this tube to the detector is dependent upon the detector configuration and is outside the scope of this manual. Usually this connection is made by inserting a "megabore" size (0.53 mm OD) Silcosteel® stainless steel tube into the detector and then inserting this tube in the bore of the gas line of the trunkline. A standard 1/16-in. size Swagelok® connector is then used to compress the gas line into the stainless steel tube. This connection should, of course, be checked for leaks using a soap solution.

NOTE: Do not connect the gas lines to either the MIP probe or the detectors without first purging this line with carrier gas flow from the controller. Failure to do so may introduce particulates into the MIP probe and/or detectors.

Power Supply

A cord is provided with the controller for attachment to the electrical supply. The input voltage for the controller is clearly marked on the back panel of the controller. Do not attempt to connect the controller to any voltage other than the input voltage prescribed on the back panel of the controller. Connection to a line voltage other than described on the back panel can result in damage to the controller and the probe, and possible danger to the operator.

Detector Outputs

Analog outputs in the 0-5 VDC range can be connected to the rear panel of the controller. Attach the positive and negative leads from the detector to the appropriate lugs on the plug on the rear panel. Polarity is marked next to the plug.

Serial Port Connection

Attach the 9-pin serial communications cable (provided) to the Data port on the rear of the controller. The opposite end of this cable should be connected to the FC4000 Field Instrument.

Section 3.6 MP3500 Power up

The following steps should be followed when turning on the MIP controller:

1. Open the valve at the carrier gas supply. Make sure that the pressure is adjusted to between 40 and 60 psi (200 to 400 kPa).
2. Open the primary regulator on the front panel of the controller, making sure that the pressure at this regulator is set to 20 psi (100 kPa).
3. Turn off the heater circuit breaker on the back panel of the controller.
4. Turn on the power switch on the back of the panel.

Under the above conditions, the system power pilot light on the front panel should be lit. If no probe is attached to the trunkline, an error message will be shown on the temperature controller on the front panel of the MP3500 series controller. Once the trunkline is attached to the probe then the probe temperature will be shown on the front panel of the controller. At this time, the heater circuit breaker can be turned on and the MIP will begin heating.

Section 3.7 Using the MIP Logging Feature

The FC4000/MP3500 instrument set is now ready to begin logging.

Section 3.7.1 Introduction

The purpose of this section is to discuss the use of the FC4000 field instrument in MIP logging mode.

Section 3.7.2 Hardware Connection

MIP logging is accomplished by first connecting the FC4000 to the MP3500. This connection has two parts, the Conductivity connection and the MIP serial connection.

Conductivity Connection

This cable is connected to the **Probe** connection on the FC4000 and the **Conductivity** connection on the MP3500. This cable carries the conductivity excitation voltage generated in the FC4000 to the trunkline connection on the back of the MP3500. This cable must be in place in order for conductivity data to be collected while doing MIP logging.

MIP Serial Connection

This cable is a male/female DB9 style six-ft long serial cable. The serial connections are made at the **COM2** connection on the FC4000 and the **Serial Data** connection of the MP3500. MIP signals are sent to the FC4000 in serial format over this cable at a rate of 20Hz. Four channels of Detector data, trunkline gas pressure and probe temperature are the signals currently collected and sent by the MP3500 to the FC4000. Refer to Figure 9 for basic MIP system connections.

Section 3.7.3 Data Storage

Data is stored to a "FLASH" memory chip within the FC4000 every 0.05 foot of logging in a tab delimited ASCII spreadsheet format. The FC4000 instrument "FLASH" memory is capable of storing over fifty 75-ft MIP logs without removing any logs. It should be noted that the FC4000 is a field instrument and should not be used as a permanent storage location for logs. A good practice would be to keep no more than one days worth of logs on the FC4000 and to remove the logs at the end of the day with the 3.5-in. floppy drive located on the back of the instrument. Once removed, the logs can be transferred to another storage device for safe keeping, organizing and archiving. If the "FLASH" memory is allowed to fill up with logs, the software

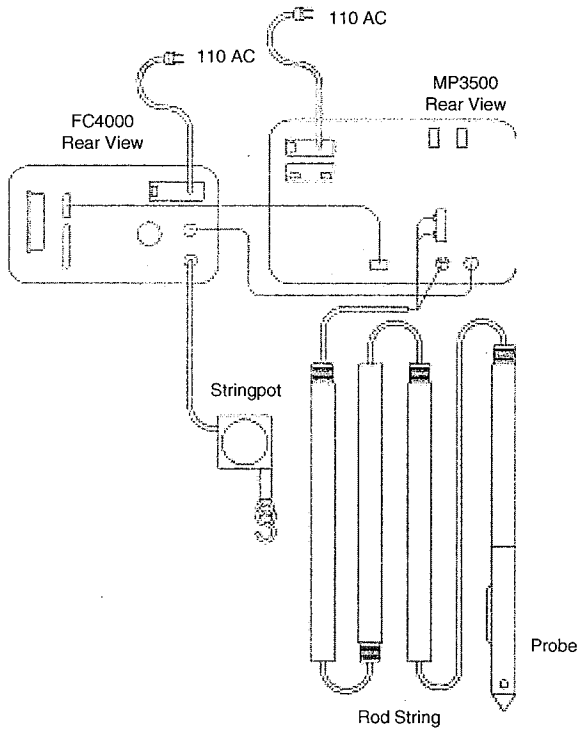


Figure 9
MIP System Connections

will not allow a log to begin and will instruct the user to clear the unwanted logs off using the **"SAVE/DEL PRT"** logs menu option. The FC4000 names the log files automatically with a consecutive numbering scheme starting at 0001.
EC data is stored to a **data** file with a **.DAT** extension. The **.DAT** file has nine columns of data. Following is a description of the data columns:

Depth (ft) **EC (mS/M)** **Rate of probing (ft/m)** **Probe temp min (C)** **Probe temp max (C)** **Trunkline pressure (psig)**
Depth (m) **Rate of Probing (M/m)** **Trunkline pressure (KPA)**

Example name:EC0050.DAT

MIP data is stored to a **gas** file with a **.GAS** extension. The **.GAS** file has eight columns of data. Below is a description of the data columns:

DET1 min(uV) **DET1 max(uV)** **DET2 min(uV)** **DET2 max(uV)** **DET3 min(uV)** **DET3 max(uV)** **DET4 min(uV)** **DET4 max(uV)**

Example name:MIP0050.GAS

Logging information is stored to an **information** file with a **.INF** extension. This file includes the probe type used for the log, the start time, the end time and date of the log.

Example name:EC0050.INF

Section 3.7.4 Logging

The FC4000 utilizes an embedded application to acquire, display and store MIP data. This section discusses the layout and use of this software. Before logging, the FC4000 needs basic information about the MIP setup. This information is collected though a simple series of menu-oriented screens.

Once MIP is selected from the Direct Sense (DS) menu, the FC4000 will display the default options screen.

Following is a description of each screen with the displayed FC4000 text in **bold** and narrative comments in *italics.* Refer to Appendix IV for software operation control keys.

SELECT AN OPTION:

1 **FOR MIP** ..... *MIP LOGGING*
2 **FOR SAVE/DEL/PRT LOGS** ..... *WORKING WITH LOG FILES*
3 **DS MENU**..... *RETURN TO THE DS MAIN MENU*

Select Option 1 by pressing the '1' key on the FC4000 keypad.

NOTE: A hidden configuration menu exists only while the above screen is displayed. The Configuration Screen is accessable by pressing the **RIGHT ARROW** key, releasing it then pressing the **CE/C** key within two seconds. The configuration menu is shown below:

CURRENT SETTINGS

ENGLISH UNITS

OPTIONS

1 **TO CHANGE TO METRIC UNITS**
2 **TO CLEAR ALL LOGS AND RESET LOG NAME**
3 **TO EXIT**

Option 1 toggles the FC4000 instrument between English and metric units. English and metric data is always stored. this setting only controls the displayed units during logging. Option 2 will permanently erase ALL logs on the FC4000 and reset the log name to '0001'. Option 3 allows the user to exit the configuration screen and return to the normal startup log screen.

Enter Gas Trip time:

'ENT' TO ACCEPT 'CE/C' TO CANCEL

Enter the gas trip time in seconds using the numberic keys on the FC4000 keypad. Accept the setting by pressing the "ENT" key on the FC4000 or clear the trip time to the default 1 second value to enter a new trip time by pressing the "CE/C" key.

SELECT AN OPTION

1 TO TEST INSTRUMENT
2 TO BYPASS ALL TESTS

Select Option '1' to test the conductivity portion of the FC4000 instrument.
NOTE: *If Option 2 is selected, no tests will be preformed.*

When Option 1 is selected, one test is run: the instrument calibration test. This test checks the calibration of the field instrument conductivity instrumentation. The following is a description of this test:

INSTRUMENT CALIBRATION TESTS:

INSTRUMENT CALIBRATION 10 OHM TEST
RESISTANCE 10.23 OHMS

The above screen is repeated for the 100, 1000 and 2000 OHM instrument calibration tests. Conditions for failure of an associated test are listed below. The purpose of this test is to verify that the field instrument is calibrated.

10 OHM:..... MORE THAN ± 1 OHM VARIANCE
ALL OTHER TESTS: ..... MORE THAN ± 5% VARIANCE

SELECT THE ROD LENGTH USED:

1 FOR 1 M RODS
2 FOR 2 FT RODS
3 FOR 3 FT RODS
4 FOR 4 FT RODS
5 FOR 5 FT RODS

Select the length of rods used for logging.

The RT screen is then shown, and logging may begin once the trigger is turned on. The trigger switch is located on the front panel of the FC4000 and controls when log data is collected, stored and displayed. No logging will take place unless the trigger switch is in the on position.

Once the log is complete, press **'ENT'** to exit the log. The instrument will return to the startup screen ready for another log.

During logging, MIP log information is displayed on the FC4000 on four user selectable screens. These screens are accessed during logging by pressing the up or down arrows on the FC4000 front keypad. Once in the logging process, each screen is accessible at anytime. Appendix IV shows the log screen navigation scheme for the MIP Logging Software.

Screen 1: Refer to Figure 10: Real time (RT) screen. This screen is the default screen and is shown after log information is entered and the logging process has begun.. This screen shows 120 seconds worth of detector real time history as well as a logging console that displays EC and MIP logging information on digital readouts.

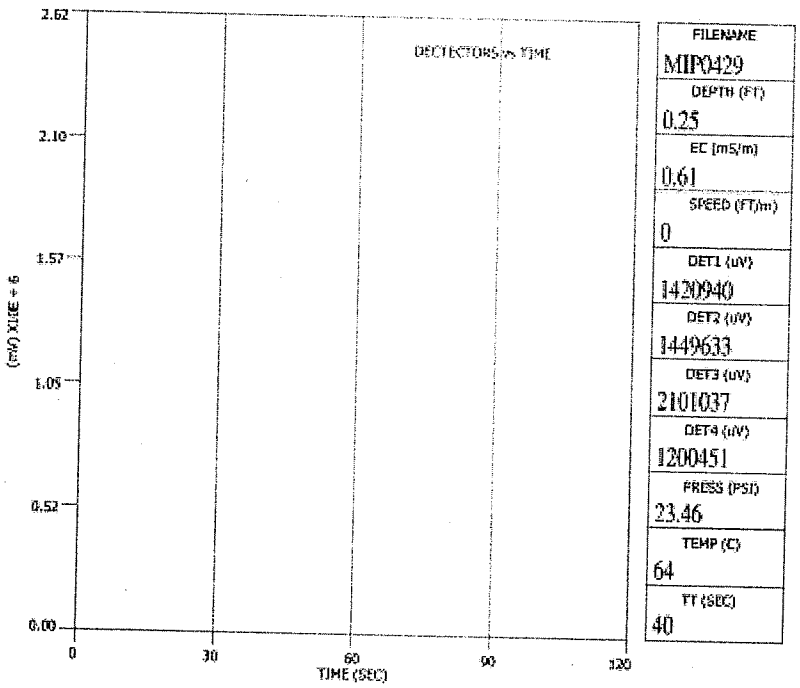


Figure 10
Real Time (RT) Screen

The detector history section is updated once a second and is an averaged response for each detector. This graph is auto-scaling. After the first 120 seconds has passed, the graph is erased and the detector history resumes at the 60 second location. The logging console displays the log name, depth, EC, speed, DET1, DET2, DET3, DET4, pressure, temperature, and trip time. Depth, EC, and speed are updated on a 0.05-ft basis. DET1, DET2, DET3, DET4, pressure and temperature are updated on a one-second time base and reflect what those parameters are doing at that specific time. Log name and trip time are unique to the log and do not change once in the logging process.

Screen 2: Refer to Figure 11: Conductivity Screen. This screen shows EC logging information and consists of three graphs. The top graph is EC. This graph is an auto-scaled representation of the EC during logging in mS/M. The middle graph is probe speed in auto-scaled ft/min or M/min units. The bottom screen is the maximum temperature response in auto-scaled degree C units. Each graph is updated on a 0.05-ft logging increment. This screen auto-scales the depth once the default 40 ft is reached in additional 20-ft increments. This allows additional logging and preserves the complete log on the screen for viewing. The default depth in metric units is 20 meters with additional increments of 10 meters.

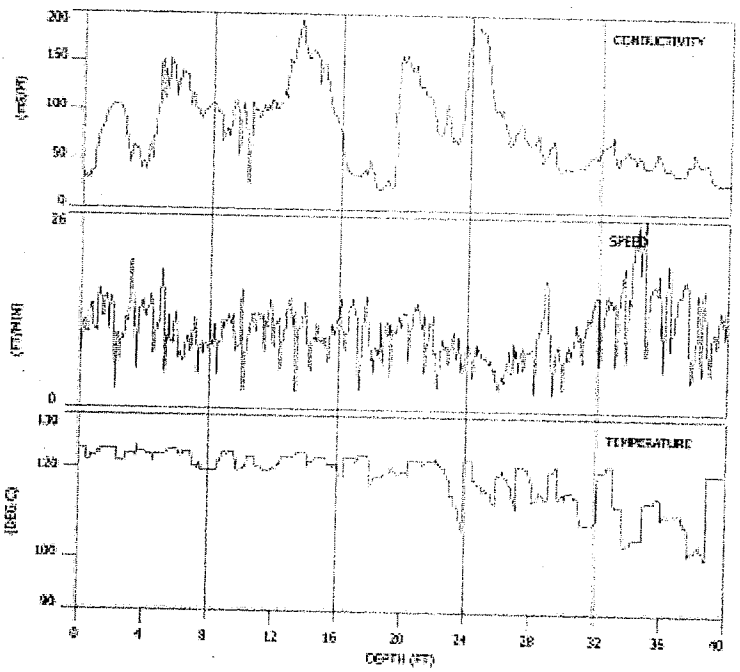


Figure 11
Conductivity Screen

Screen 3: Refer to Figure 12: Conductivity and MIP screen. This screen shows EC information along with DET1 and DET2 information. The top graph is EC and works the same as the EC graph in screen 2. The middle and bottom graphs are DET1 and DET2 responses respectively. These graphs are auto-scaled maximum detector 1 and detector 2 responses in uV units. The DET1 and DET2 graphs are updated on a 0.05-ft logging increment once the set trip time has passed for each MIP probe push. The auto-scale depth of this screen works the same as screen 2.

Screen 4: Refer to Figure 13: MIP Detector response. This screen shows all four auto-scaled detector maximum responses in uV units. Each graph shows one detector response and is updated on a 0.05-ft logging increment once the set trip time has passed for each MIP probe push. The auto-scale depth of this screen works the same as screen 2.

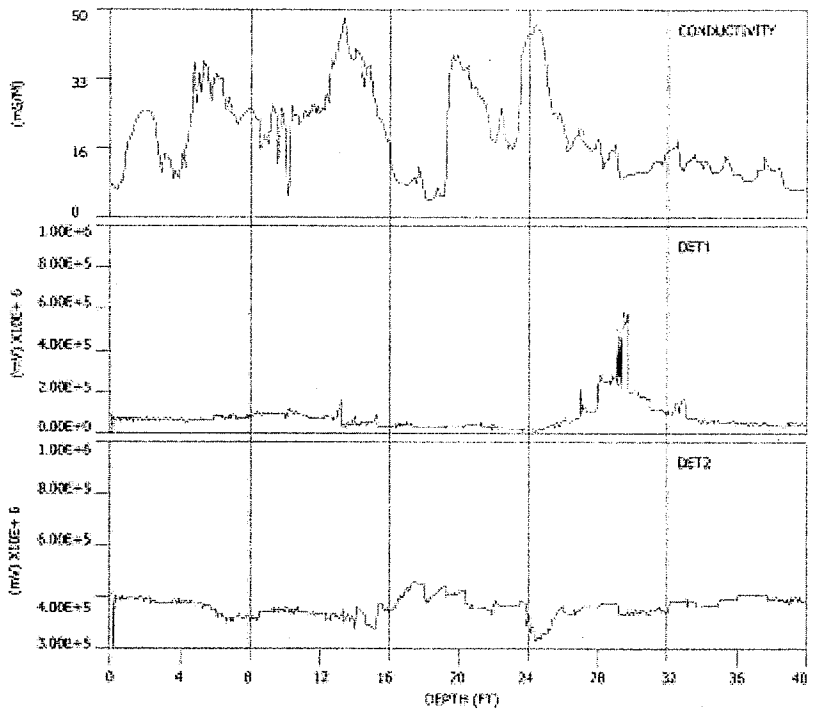


Figure 12
Conductivity and MIP Screen

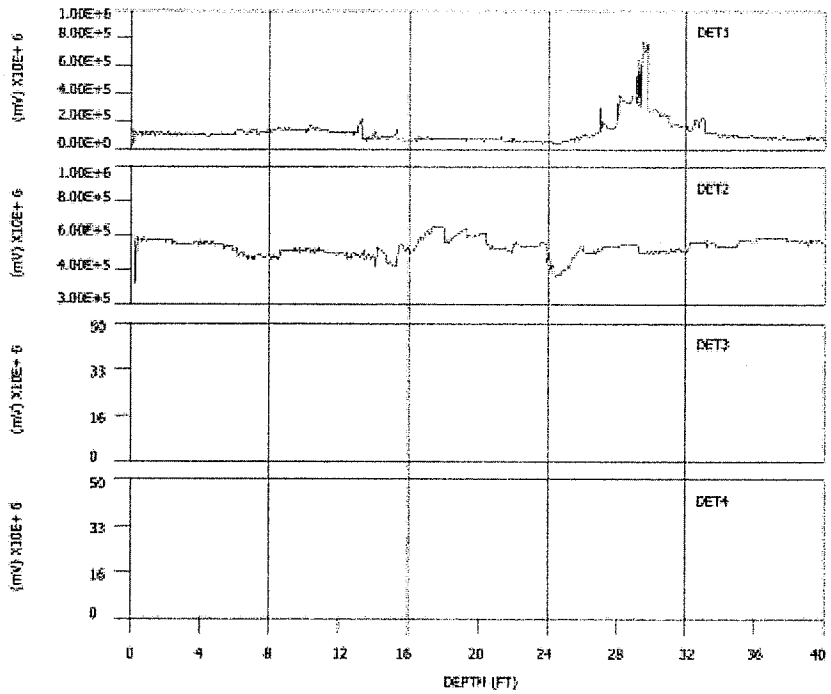


Figure 13
MIP Detector Response

Attenuation: Refer to Figure 14: Detector Attenuation Prompt. This screen allows detector attenuation to be set during the logging process. An attenuation change is made by pressing keys 1, 2, 3, or 4 on the FC4000 keypad during the logging process. Once one of these keys is pressed, the current log graph screen is replaced by the detector attenuation prompt. The attenuation may be entered by pressing the numeric keys on the FC4000. The selected detector attenuation is highlighted by an asterisk '*'. Other detector attenuations may be changed by using the up/down arrows on the FC4000 to move the asterisk to the desired detector number. The minimum and maximum detector attenuations are 1 and 10000 respectively. Press the CE/C key once to return to normal logging. The default attenuation for all detectors is 1 (one) at the beginning of each log.

ATTENUATION
DET1: *1
DET2: 10
DET3: 100
DET4: 1

'CE/C' TO EXIT

Figure 14
Detector Attenuation Prompt

Press the '**ENT**' button on the FC4000 keypad once to exit the log. The user will be prompted with the following screen:

SAVE LOG TO FLOPPY?

1. **YES** ..... *Save Flash stored log directly to the floppy drive*
2. **NO** ..... *Do not save the log to the floppy drive*

Select the desired option and the log will end and the FC4000 will return to the main menu. It should be noted that either selection still maintains the stored file on the FLASH storage module inside the FC4000.

Section 3.8 MIP Display Log

Section 3.8.1 Introduction

The purpose of this section is to discuss the use of the FC4000 field instrument-based and Windows-based MIP Display Log software features.

Section 3.8.2 FC4000 MIP Display Log

The FC4000 MIP Display Log feature allows for field viewing and printing of MIP logs that reside on the FC4000 instrument. This feature is accessed through the **SAVE/DEL/PRT MIP LOGS** option on the beginning MIP menu. Upon selecting this option, the FC4000 displays the available **.DAT** (EC) log files. Select the desired file by pressing the corresponding numerical key on the FC4000 front keypad. If there are more than ten logs on the instrument, use the ENT key to list the next ten available logs or the **CE/C** key to exit back to the beginning MIP menu.

After selecting the file, the user has the option of saving the MIP files (**.DAT, .GAS,** and **.INF**) to the instrument 3.5-inch floppy, permanently deleting the file from the instrument, or graphing the file. Saving and deleting files is considered to be self explanatory. The remainder of this section will deal with displaying MIP logs on the FC4000 instrument. It should be noted that the FC4000 MIP display log capabilities are intended to provide a simple field display of the log. If a presentation quality graph is desired, see Section 3.8.3: MIP Display Log for Windows.

After the EC (**.DAT**) file has been selected, the FC4000 will automatically open and graph the associated MIP (**.GAS**) file to give the user a complete MIP log representation through four user selectable screens. Screen selection is done with the up and down arrow keys on the FC4000 front keypad. The following is a description of each screen.

Screen 1: Refer to Figure 15: Conductivity Screen. This screen is the default screen for the FC4000 MIP Display Log feature. This screen has four graphs that auto scale to the log data. This screen shows Conductivity, Rate of Probing, Probe Temperature, and Trunkline Pressure verses Log Depth data. When in metric mode, Rate of Probing will be in meters per minute; Trunkline Pressure will be in KPA, and Log Depth will be in meters. Figure 15 shows screen 1 in English units.

Screen 2: Refer to Figure 16: Conductivity, Detector 1 and Detector 2 Screen. This screen has three graphs that auto scale to the log data. This screen shows Conductivity, Detector 1, and Detector 2 verses Log Depth data. When in metric mode, log depth is shown in meters.

Screen 3: Refer to Figure 17: Conductivity, Detector 3 and Detector 4 Screen. This screen has three graphs that auto scale to the log data. This screen shows Conductivity, Detector 3, and Detector 4 verses Log Depth data. When in metric mode, log depth is shown in meters.

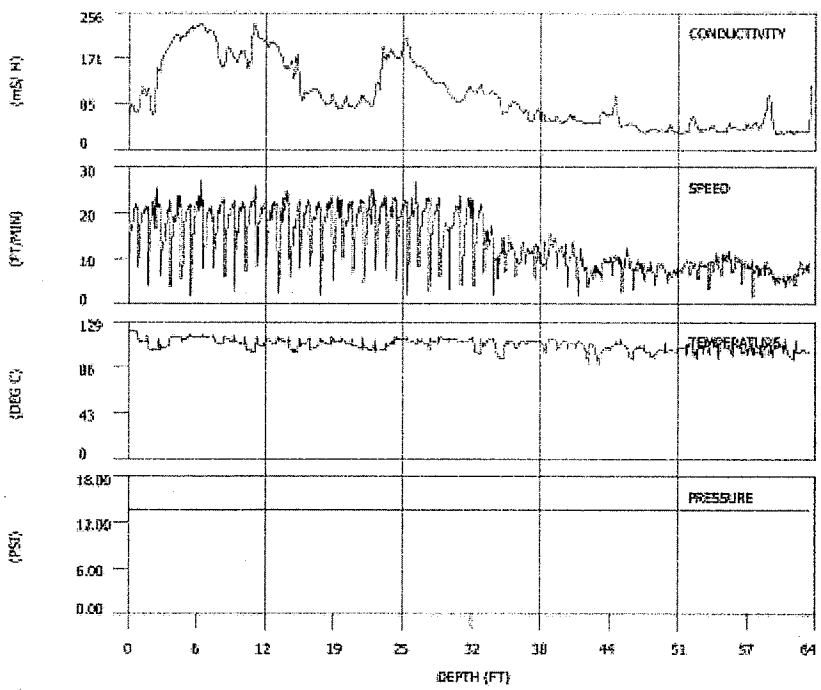


Figure 15
Conductivity Screen

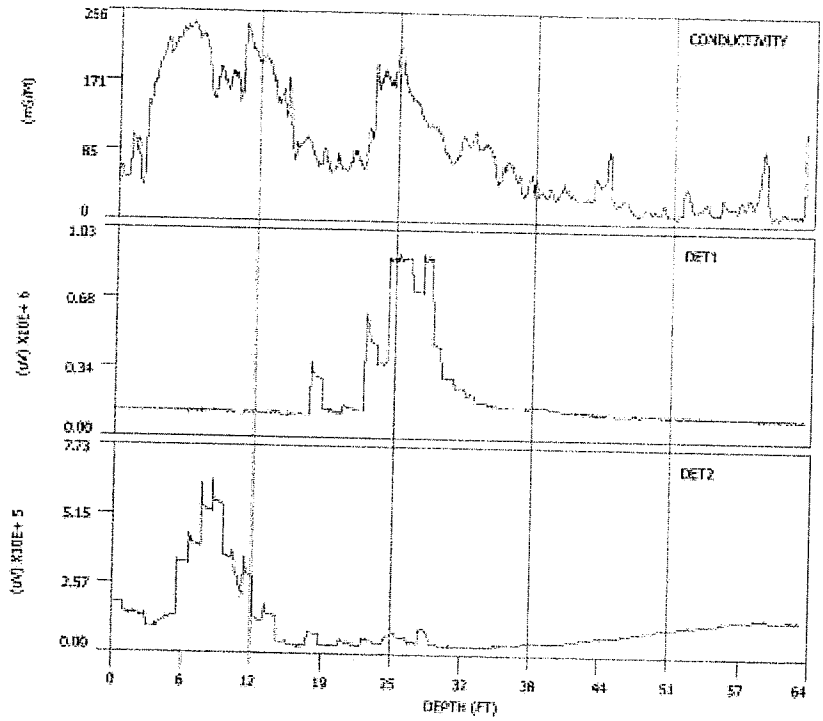


Figure 16
Conductivity, Detector 1, and Detector 2 Screen

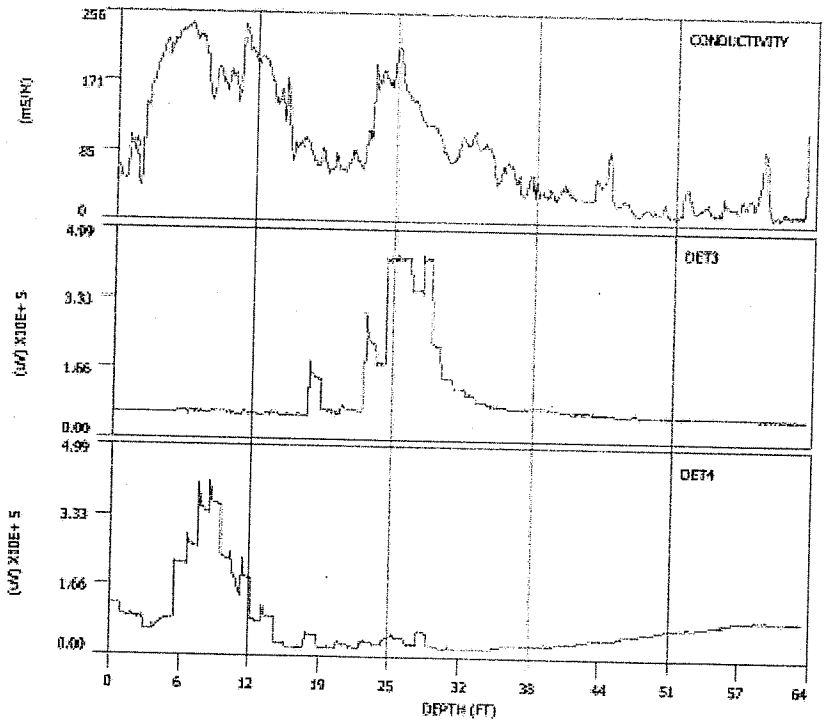


Figure 17
Conductivity, Detector 3, and Detector 4 Screen

Screen 4: Refer to Figure 18: Detector Response Screen. This screen has four graphs that auto scale to the log data. This screen shows all detector responses verses depth. When in metric mode, log depth is shown in meters.

A field hard copy of the selected screen may be printed on an Epson LX300 printer by pressing the **PRINT** key on the FC4000 front keypad.

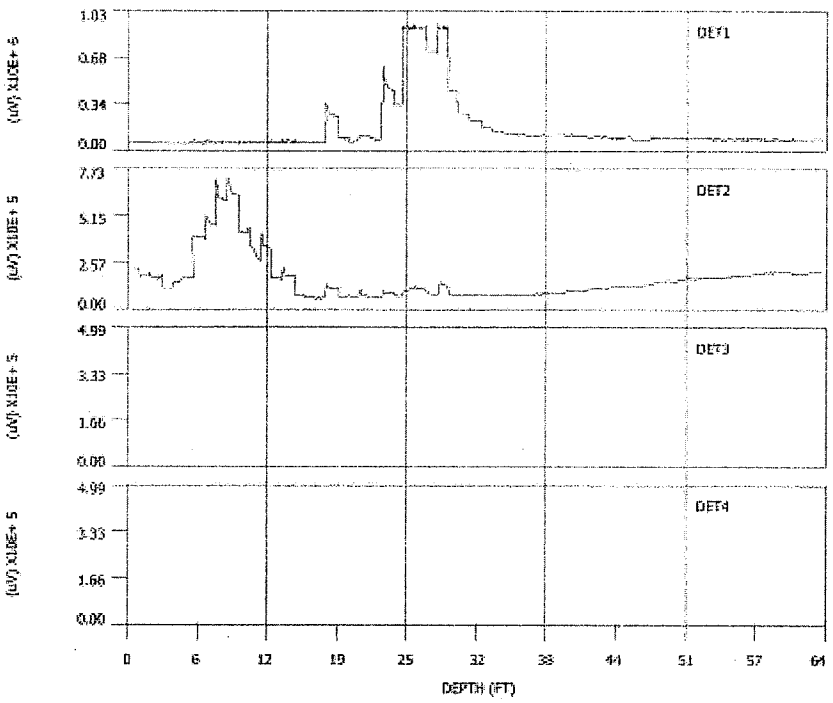


Figure 18
Detector Response Screen

Section 3.8.3 MIP Display Log for Windows

Every FC4000 instrument configured with MIP ships with a Windows MIP Display Log software package that is compatible with Win 9X, Win 2000, Win ME, Win NT 4.0, and Win XP. Installation is straightforward and consists of running the setup program on the CD. The MIP Display Log for Windows provides a quick way for the user to graph, change scaling, and print log information. Figure 19 shows a sample screen from the MIP Display Log for Windows package.

The user may select English or metric units for display. Figure 19 shows English units. When metric units are selected, the log depth is displayed in meters.

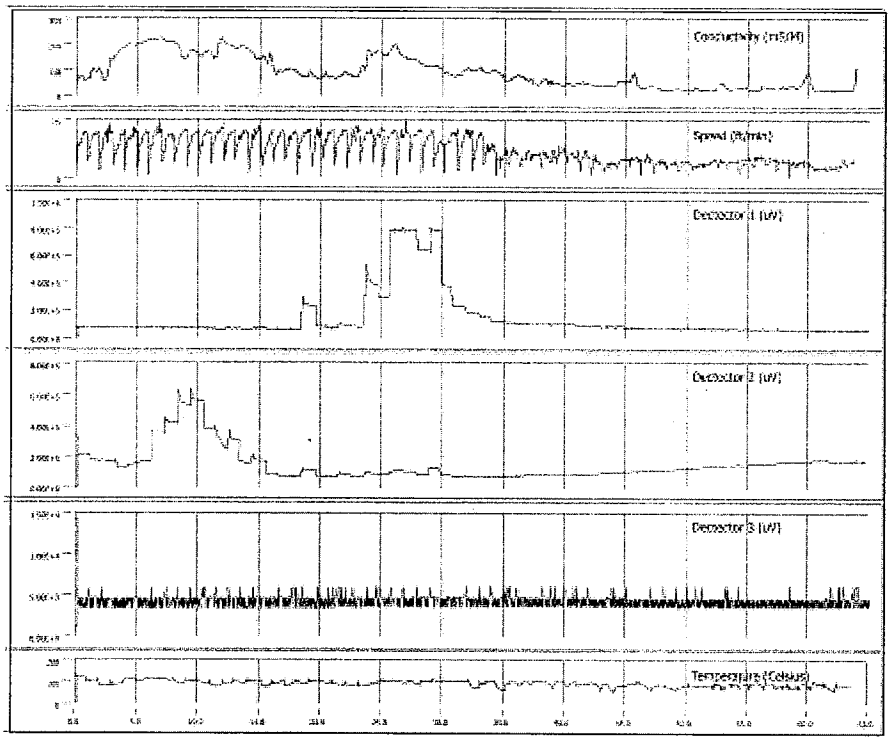


Figure 19
MIP Display Log for Windows

Section 3.9 Using the MIP Time Feature

Section 3.9.1 Introduction

The purpose of this section is to discuss the use of the Geoprobe Systems FC4000 field instrument in (Membrane Interface Probe) MIP Time mode.

Section 3.9.2 Hardware Connections

MIP Time mode only requires the Serial connection between the FC4000 and the MP3500 as only MIP parameters are processed in this mode. See the **Using the MIP Logging Feature** section for a complete explanation of this connection.

Section 3.9.3 Data Storage

Data is stored to a "FLASH" memory chip within the FC4000 once per second of logging in a tab delimited ASCII spreadsheet format. The FC4000 instrument "FLASH" memory is capable of storing over three hundred 5-minute MIP TIME logs without removing any logs. It should be noted that the FC4000 is a field instrument and should not be used as a permanent storage location for logs. A good practice would be to keep no more than one day's worth of logs on the FC4000 and to remove the logs at the end of the day with the 3.5-in. floppy drive located on the back of the instrument. Once removed, the logs can be transferred to another storage device for safe keeping, organizing and archiving. If the "FLASH" memory is allowed to fill up with logs, the software will not allow a log to begin and will instruct the user to clear the unwanted logs off using the EXPORT

TIME FILES menu option. The FC4000 names the log files automatically with a consecutive numbering scheme starting at 0001. MIP time data is stored to a **time** file with a **.TIM** extension. The **.TIM** has seven columns of data. The data in this file is an average response for each parameter. Following is a description of the data columns:

DET1 (uV) DET2 (uV) DET3 (uV) DET4 (uV) TEMP (deg C) PRESSURE (psi) PRESSURE (KPA)

Example name: MPT0050.TIM

Logging information is stored to an **information** file with a **.INF** extension. This file includes the probe type used for the log and the start time, end time, and date of the log.

Example name: MPT0050.INF

Section 3.9.4 Logging

The FC4000 utilizes an embedded application to acquire, display and store MIP time data. This section discusses the layout and use of this software. Before time logging, the FC4000 needs some basic information. This information is collected through a simple series of menu-oriented screens.

Below is a description of each MIP time setup screen with the displayed FC4000 text in **bold** and narrative comments in *Italics*. Refer to Appendix V for software operation control keys.

Once MIP Time is selected from the Direct Sense (DS) menu, the FC4000 will display the default options screen.

SELECT AN OPTION:

1 **BEGIN** ..... *MIP TIME LOGGING*
2 **EXPORT FILES** ..... *WORKING WITH TIME FILES*
3 **DS MENU** ..... *RETURN TO DS MAIN MENU*

Select Option 1 by pressing the '1' (one) key on the FC4000 keypad.

SELECT AN OPTION:

1 **MONITOR ONLY** ..... *MONITOR MIP TIME DATA*
2 **MONITOR AND SAVE** ..... *MONITOR AND SAVE TIME DATA*

Select Option 2 by pressing the '2' (two) key on the FC4000 keypad.

This will save the time data as it is being collected. Selecting **Monitor Only** does not save the log data to file. The Real Time (RT) screen is then shown.

Once logging is complete, press **'ENT'** to exit the log. The instrument will return to the startup screen ready for another log.

During logging, MIP Time log information is displayed on the FC4000 on two user selectable screens. These screens are accessed during logging by pressing the up or down arrow keys on the FC4000 front keypad. Once in the logging process, each screen is accessible at anytime. Appendix V shows the log screen navigation scheme for the MIP Time software.

Screen 1: Refer to Figure 20. Real time (RT) screen. This screen is the default screen and is shown once time logging is started. This screen shows 120 seconds worth of detector real time history as well as a logging console that displays digital information. The detector history is updated once a second and is an averaged response for each detector. This graph is auto-scaling. After the first 120 seconds has passed, the graph is erased and the detector history starts over at the 60 second location. The logging console displays the log name (MONITOR if not saving to a log file), DET1, DET2, DET3, DET4, pressure, temperature, and an estimated available log storage space in minutes. All console parameters are updated on a once per second time base and reflect what those parameters are doing at that specific time. The Log name is unique to the log and does not change once in the logging process.

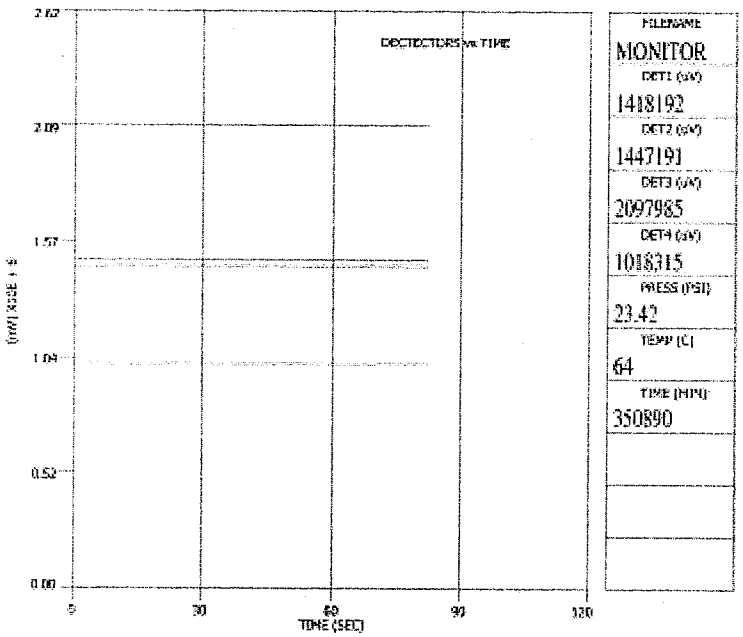


Figure 20
MIP Time Real Time (RT) Screen

Screen 2: Refer to Figure 21: MIP Time Temperature and Pressure Screen. This screen shows the probe temperature and trunkline pressure during logging. These graphs are auto scaling and update on the same time basis as Screen 1.

Attenuation: Refer to Figure 14. Detector attenuation prompt. This screen allows detector attenuation to be set during the logging process. Attenuation works as it does for the Logging software.

Press the '**ENT**' button on the FC4000 keypad once to exit the time log. If the **MONITOR AND SAVE** option was selected earlier, the user will be prompted with the following screen:

SAVE LOG TO FLOPPY?

1. **YES** ..... *SAVE FLASH STORED LOG DIRECTLY TO THE FLOPPY DRIVE*
2. **NO** ... *DO NOT SAVE THE LOG TO THE FLOPPY DRIVE*

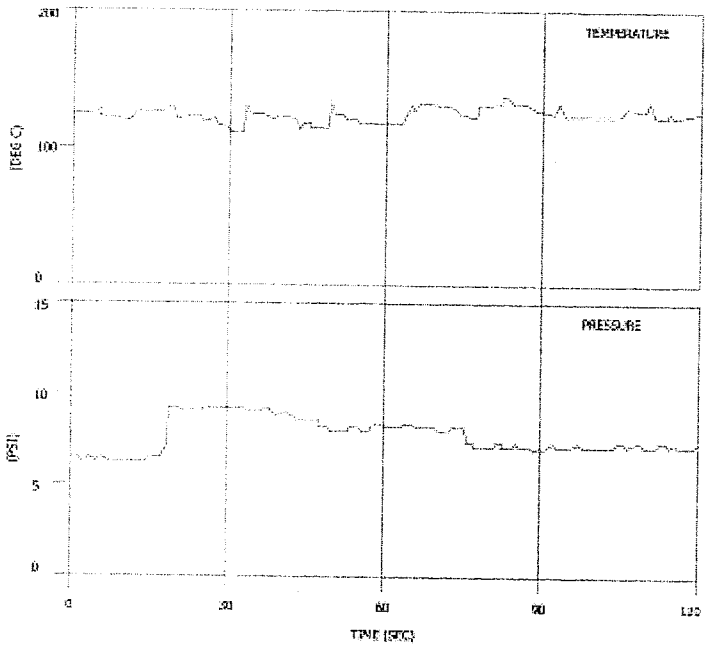


Figure 21
MIP Time Temperature and Pressure Screen

Select the desired option and then the log will end and the FC4000 will return to the main menu. It should be noted that either selection still maintains the stored file on the FLASH storage module inside the FC4000.

Section 3.10 MIP System Servicing and Troubleshooting

The section is intended to assist in identifying where problems may occur and how to fix them. The MIP Controller can be divided into two basic sections to help with diagnostics: Heater and Gas Flow.

Section 3.10.1 Heater Circuit *(for MIP probe heating element)*

Circuit Breaker Trips (line-to-line device)	• Short circuit has occurred in the probe. (This is the cause 95% of the time.) • Short circuit has occured in the trunkline • Short circuit has occured in the MIP controller.
GFCI Trips (Ground Fault device)	• Short to ground in the probe (This is the cause 95% of the time) • Short to ground in the trunkline. • Short to ground in the MIP Controller.
Do not get 50 VAC output (probe does not heat up)	• Power is not ON. • Thermocouple is not connected. • Circuit breaker has tripped. • GFCI has tripped. • Solid State Relay failure.
Temperature controller does not function properly	• Power is not ON. • Poor thermocouple connection. • Temperature controller failure.
How do I replace the temperature controller?	• Remove temperature controller from the front panel. Pull on the module and the entire controller will slide from its mounted housing. • Install new PID temperature controller by inserting into PID housing that is already mounted on the front panel. This procedure is done from the outside ONLY!
How do I replace the Circuit Breaker?	• Turn OFF power and remove "top" lid • Cut protective shrink-tubing and remove leads with a screwdriver. Install new circuit breaker.
How do I replace the Solid State Relay?	• Unfasten control lines from PID temperature controller (bottom two screws) • Loosen large mounting bolts and ground wire; replace with new relay **IMPORTANT: Make sure wires are re-fastened correctly, including the unit ground wire.**

Section 3.10.2 Carrier Gas Flow Circuit

Primary Regulator pressure goes HIGH	• Particles got into the regulator and it cannot function properly. • Restriction in the circuit somewhere after the regulator. • Septa on injection port may be plugging the line.
Primary Regulator pressure unable to set and seems unstable	• Source gas bottle is low. • Particles got into the regulator and it cannot function properly.
Mass Flow Controller not responding to changes in adjustment	• Requires back pressure in order to set and measure easily. • May just appear to be not responding based on pressure gauge. • May have dust particles causing internal malfunction. • Mass Flow Controller malfunction.
Mass Flow pressure goes HIGH	• Line restriction. • Output port may be plugged. • May have dust particles causing internal malfunction.
Mass Flow pressure very LOW (measured with 100 ft. trunkline in place)	• Leak in the system (carrier gas circuit). • May have dust particles causing internal malfunction.

Equipment and System Specifications

MP350X Control Module and A/D

(Designed for function control and data recording of the Membrane Interface Probe)

- GFCI (Ground Fault Circuit Interrupter) protection on the Heater circuit (50VAC)
- Circuit breaker functions as an On/Off switch for Heater circuit control, and for component and user protection.
- Inputs and Outputs:

Power Input	• MP3500 100 – 120 VAC @ 3 amps (input) • MP3503 200 – 240 VAC @ 3 amps (input)
Detector channels	• Four channels 0 – 5VDC analog (input)
Carrier gas input	• Users selected type, 60psi (max)
MIP Probe Heater	• 50VAC @ 6 amps (output)
Thermocouple	• Type K, temperature recording and control (input/output)
Conductivity	• 0.75VAC (low signal input/output)
Data Interface	• DB-9 Serial (output) to FC4000 Field Instrument
Regulated gas output	• Regulated to 20 psi @ controlled flow rate to sensor

APPENDIX I
Components and Specifications

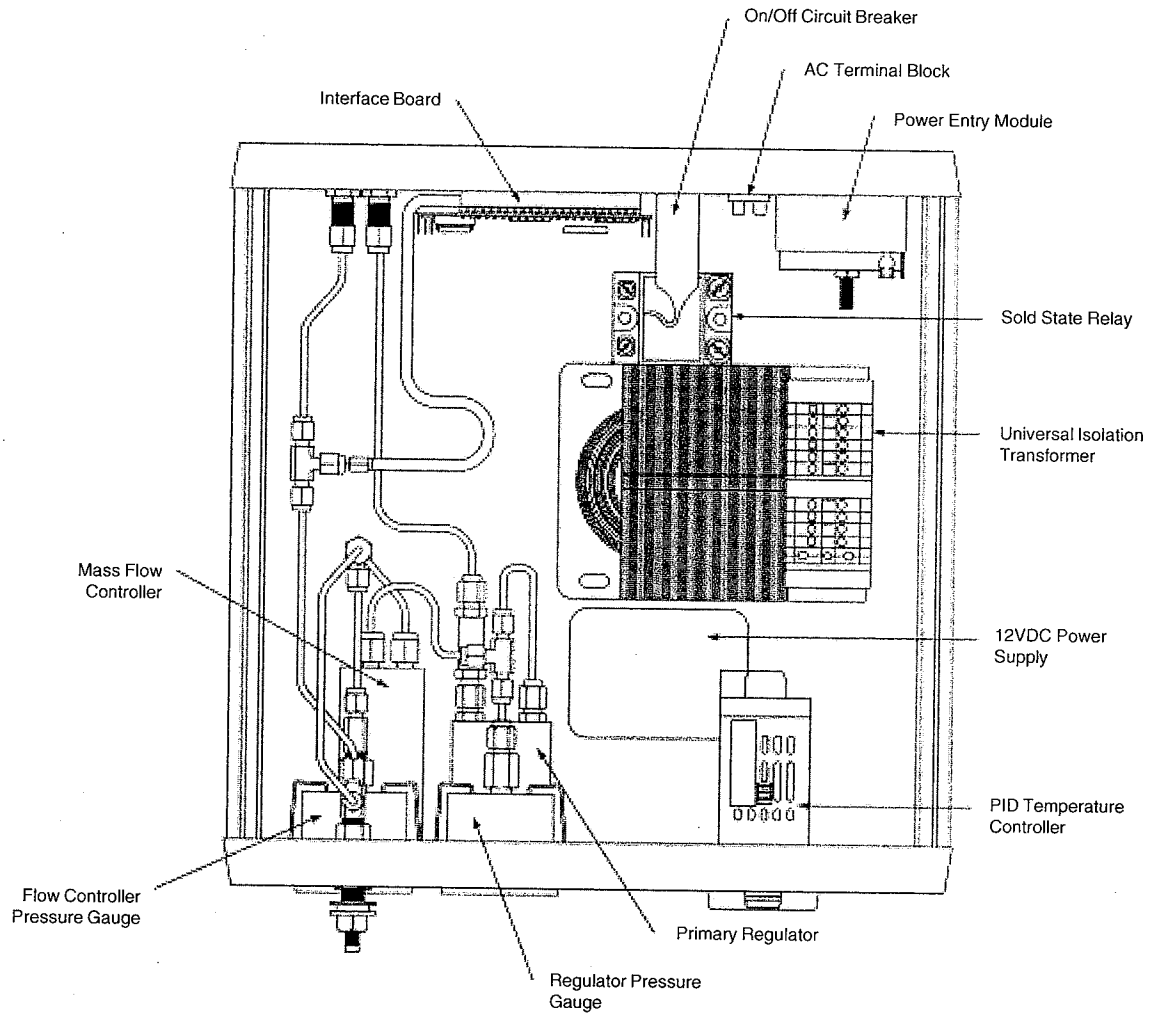


Figure AI-1
MIP Controller, Top View, Lid Removed

Universal Isolation Transformer

- Open style control transformer, with shield between primary and secondary windings.
- Isolation type transformer
- Finger safe terminals
- Primary — 100, 120, 220, 230 and 240VAC
- Secondary — 50VAC (7 amps)
- Dimensions — 4.5 in. x 5.75 in. x 6 in.
- Weight — 13 lbs.
- CE-Mark licensed through TUV—Rheinland

12 VDC Power Supply

• Input	100 – 240 VAC
• Frequency	50 – 60 Hz
• Current rating	0.7 Amps
• Output	12 volt dc @ 2 amps
• TUV and UL recognized	
• CE-Mark	

PID Temperature Controller

• Power	90 to 250VAC, 50 to 400Hz
• Operating Ambient	0 – 55 degree C.
• Dimensions	2 in. x 2 in. x 6 in.
• Input	Type K thermocouple
• Analog output	Isolated, 0 – 5vdc
• Bezel	NEMA-4 square face display
• Settings	Program controlled
• Security	Code set by Geoprobe® Systems
• CE-Mark	

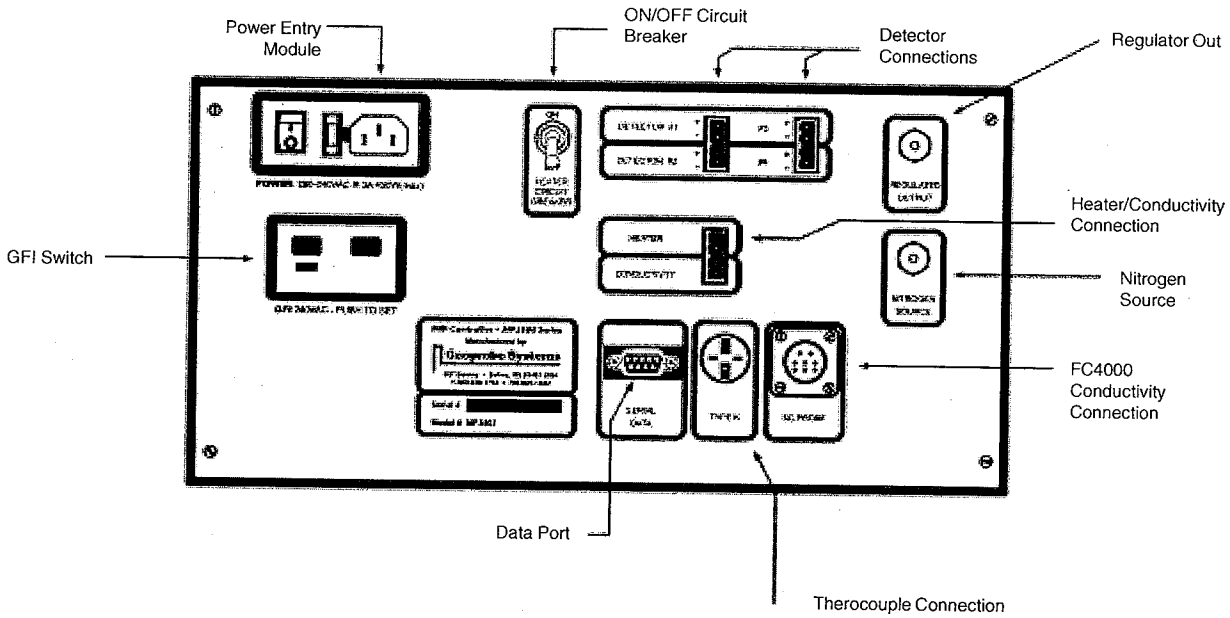


FIGURE AI-2
MP3500 Series Controller Rear Panel

Primary Regulator (0 – 60 psi)

Regulator Pressure Gauge (0 – 60 psi)

Flow Controller Pressure Gauge (0 – 30 psi)

Mass Flow Controller (0 – 100 mL/min.)

Solid State Relay

• Input	5V dc pulse
• Controlled output	50VAC
• Current Rating	25 amp (max)
• Type	SPST - NO

Power Entry Module (fused)

10A 120V Time Delay BUSSMANN # GMC-10A (MP3500)
6.3A 250V Time Delay BUSSMANN # GDC-6.3A (MP3503)

On/Off Circuit Breaker *(Dual Function)*

• Pole	Single pole
• Device type	Thermal
• Switch function	Breaker plus ON/OFF
• Rating	2 or 3 Amp (Two different models used)
• 100% load	No trip
• 135% load	trip in 1 hour
• 200% load	trip in 11 – 15 seconds
• UL recognized	

AC Terminal Block

GFCI *(Ground Fault Circuit Interrupter)*

• Mounting	Panel mount on rear panel
• Phase	Single
• Voltage	120 or 240VAC (Two different models used)
• Current rating	15 Amps
• Trip Level	4-6 mA
• Trip response	< 25 mS
• Frequency	50 / 60 Hz
• Power ON	Indicator lamp
• CSA and UL recognized	

Interface Board

- Analog channel input connections
- 50VAC output for probe heater
- Soil Conductivity I/O for probe
- On board A/D conversion card
- Trunkline pressure sensor
- Probe temperature monitor input

FC4000 Conductivity Connection

- Brings Conductivity signals into the FC4000 Field Instrument

Thermocouple Connection

- Type K thermocouple from the trunkline

Data Port

- Accepts DB-9 Male serial connection to the FC4000 Field Instrument

Heater/Conductivity Connection

- Trunkline heater and conductivity connections

Detector Connections

- GC detector inputs

APPENDIX II
Internal Wiring

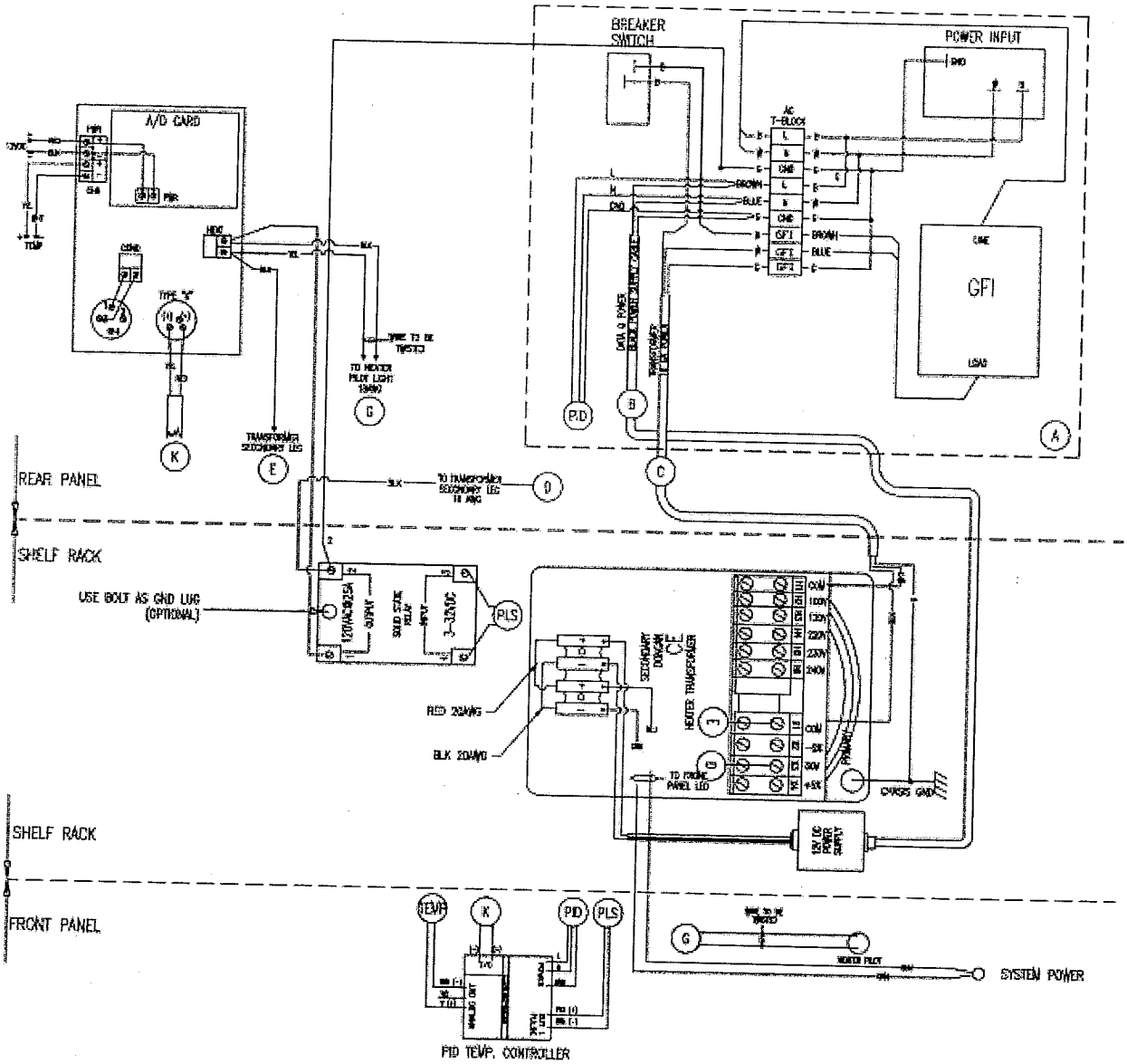


Figure AII-1
Internal Wiring Diagram

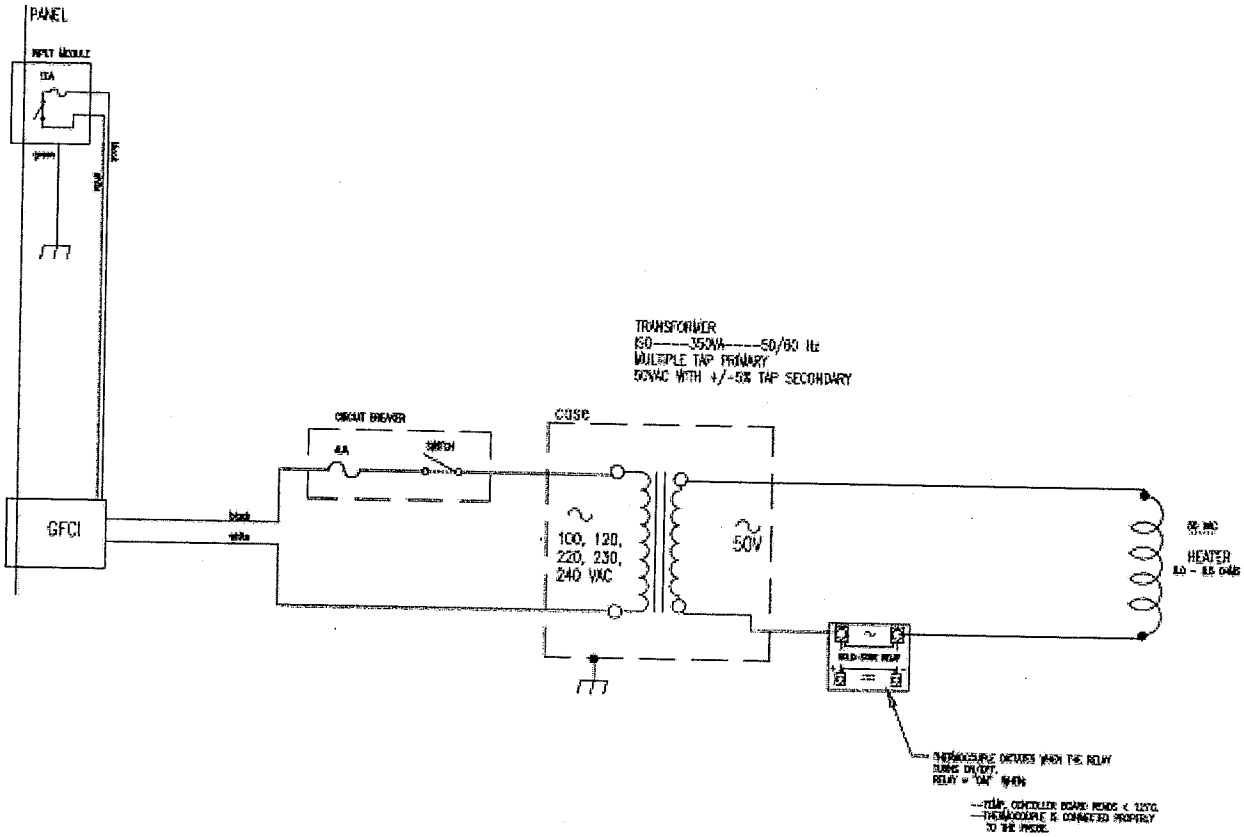


Figure AII-2
Complete Heater Circuit Layout with External Heater

APPENDIX III
Internal Carrier Gas Plumbing

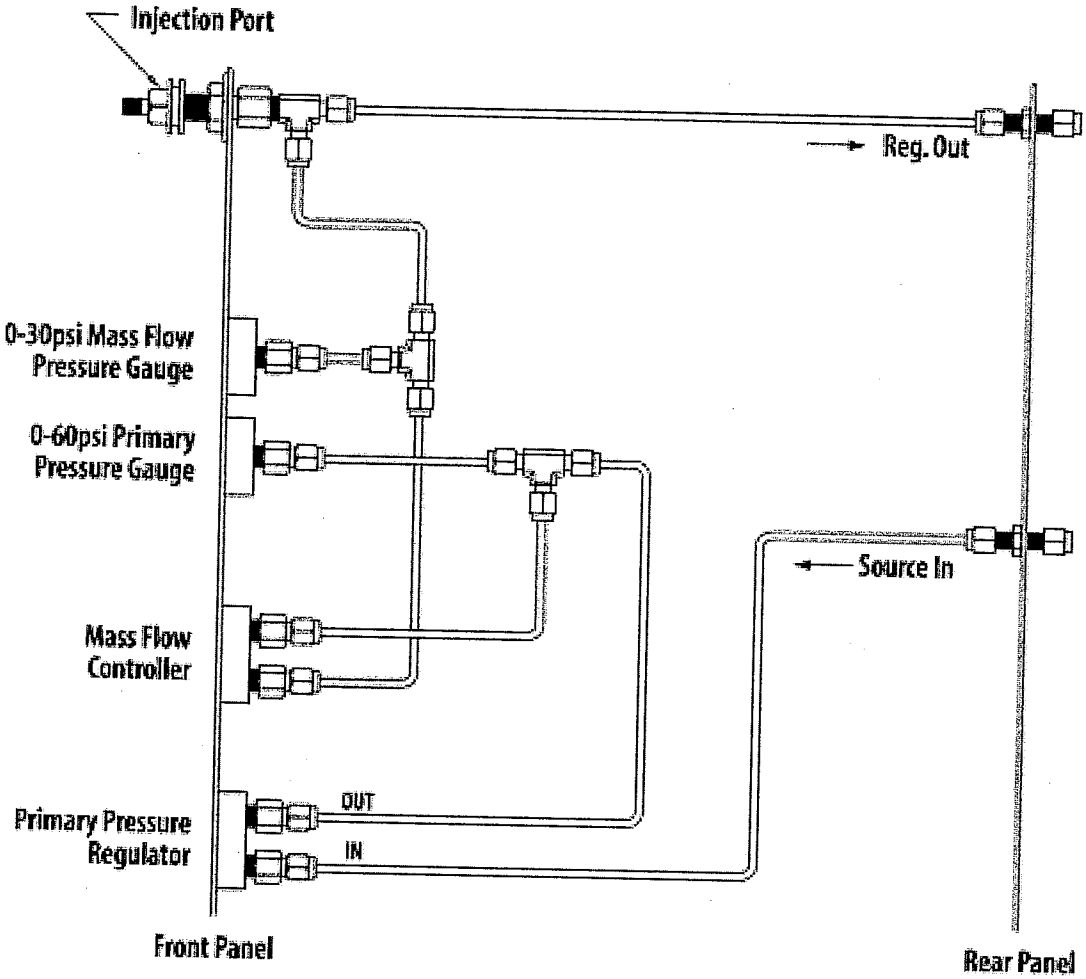


Figure AIII-1
MP3500 Internal Carrier Gas Plumbing

APPENDIX IV
MIP Logging Screen Key Commands

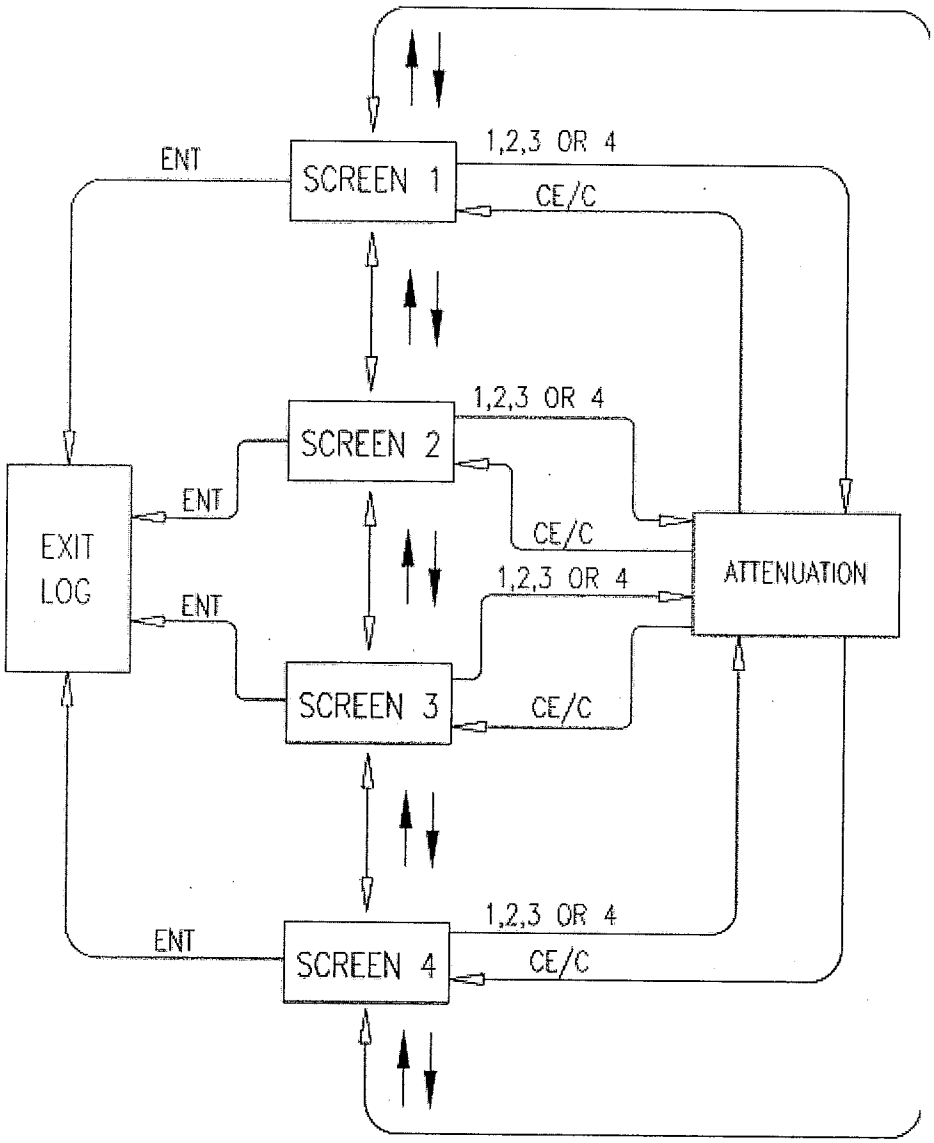


Figure AIV-1
MIP Logging Key Navigation

APPENDIX V
MIP Time Log Key Commands

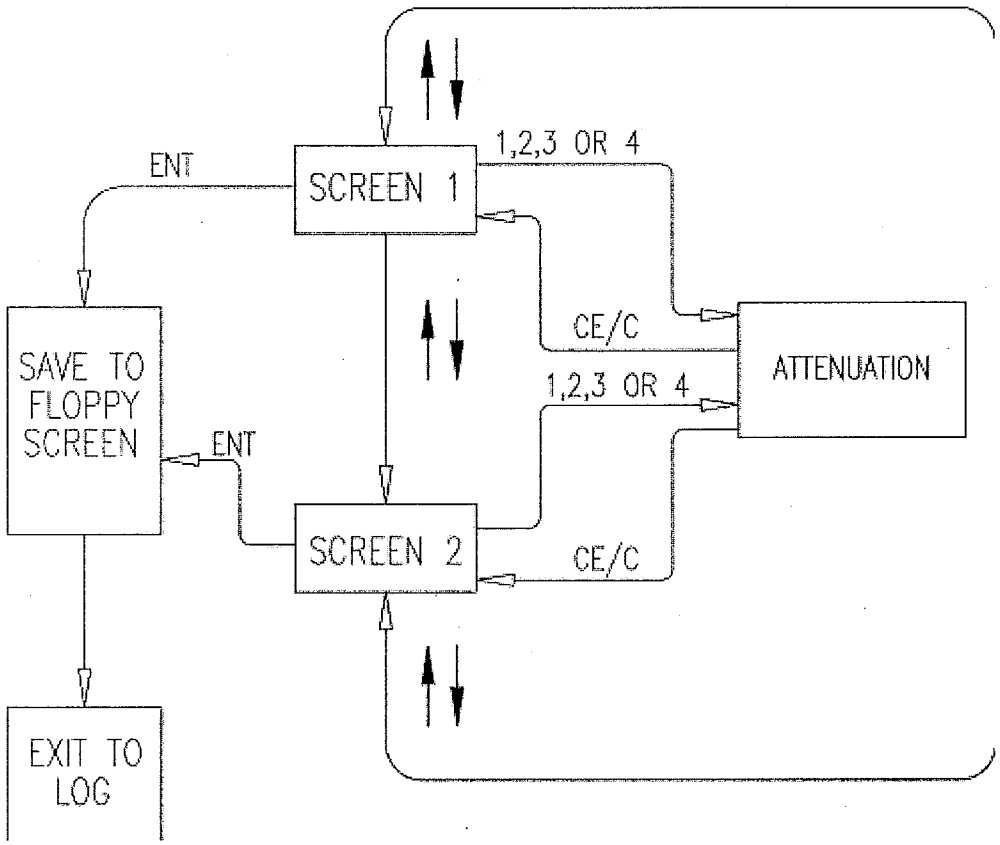


Figure AV-1
MIP Time Logging Key Navigation

APPENDIX VI
EC Logging Screen Key Commands

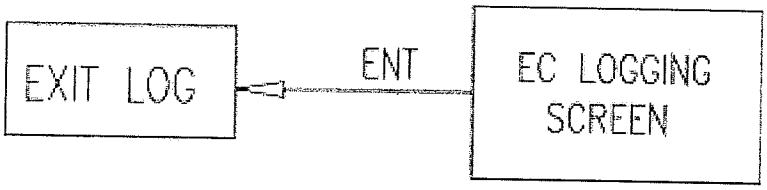


Figure AVI-1
EC Logging Key Commands

APPENDIX VII
MP3500 Transformer Configuration

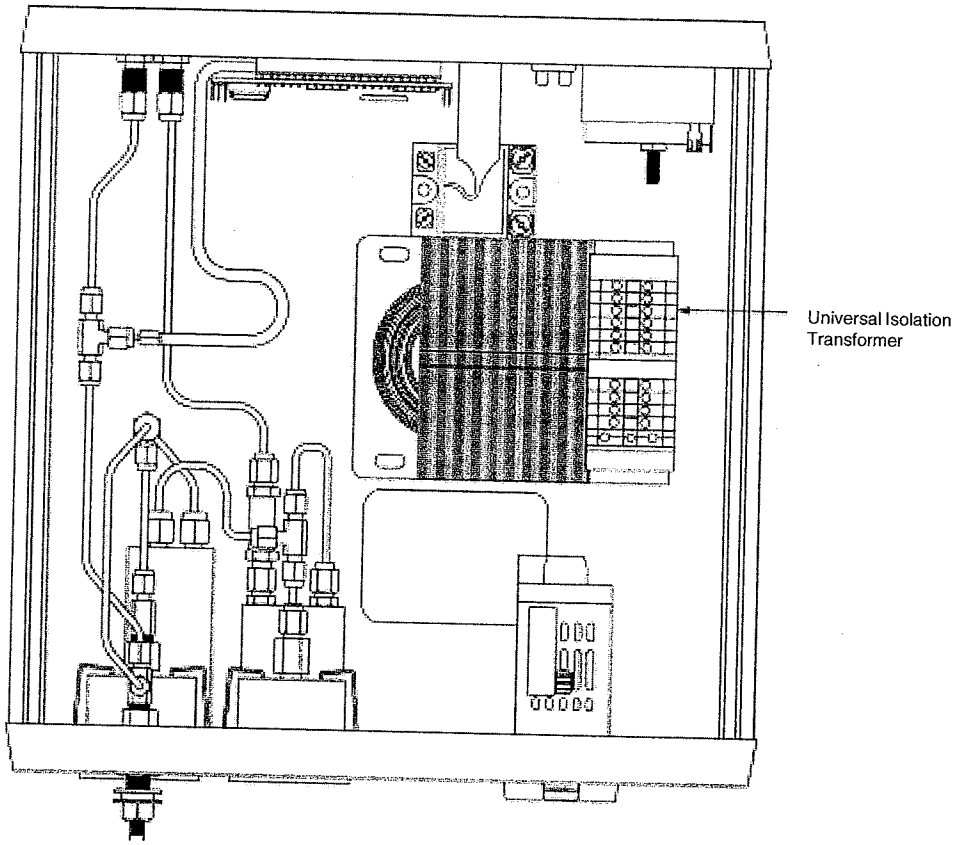


Figure AVII-1
MP3500 Transformer Configuration

Introduction

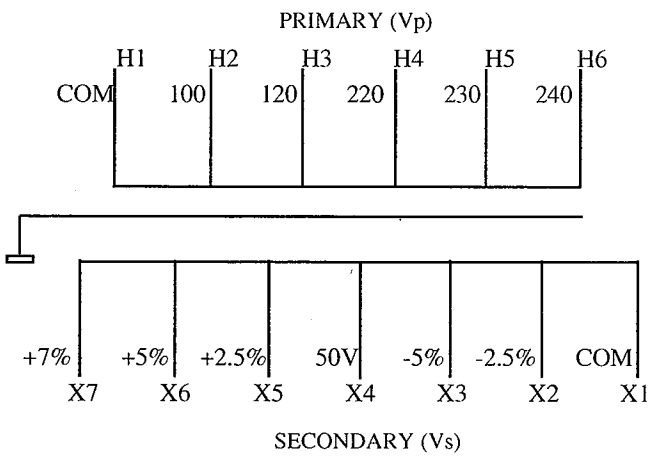
This section is intended to assist in field communication between customer and technical support personnel to re-wire the heater transformer to accommodate installation input voltage levels. The heater transformer may need to be re-wired whenever deemed necessary by the manufacturer or authorized European representative.

Procedure

The transformer needs to be wired to fit the following guidelines:

1. Input power sourced does NOT match the name-plate input value.
2. Measured output to heater is NOT 50 VAC (± 2%).

Step 1: Use the following terminal pin-out to properly wire the MP3500 Series controller to suit the power source. The "COM" or common ground will remain connected. Shift H2 – H6 to match the power source.



Step 2: Use the following chart to help set up the correct scheme. With this system, it is required that the heater output to the probe is 50VAC (± 1.5%). This means one lead will be connected to "X1" (COM) for common ground on the secondary side. The other is connected to "X4" which is the 50V tap.

Table AVII-1
Primary Voltage Compensation

TAP	-5%	-2.5%	BASE	2.5%	5%	7%
Vp	241.5	235.75	230	224.25	218.50	213.90
Vs (Adjusted)	50	50	50	50	50	50

It is possible that the output may read less than the desired 50VAC. Under loaded conditions, the transformer Secondary (output) can drop in proportion to a drop in the Primary (input).

An example of how this chart can be used is as follows:

Condition: Input to the module is designed for 230 VAC in Europe.
Input measured under load with the heater running reads 224 VAC.
Output will therefore read 40 to 48.75 VAC.

Solution: Re-wire the secondary of the transformer to the +2.5% tap. This will shift the secondary (output) voltage to 50 VAC.

Vp	Primary	Secondary
100	H2-H1	X4-X1
120	H3-H1	X4-X1
200	H4-H1	X6-X1
220	H4-H1	X4-X1
230	H5-H1	X4-X1
240	H6-H1	X4-X1

Table AVII-2
Standard Voltage Connections

Voltages above 240 VAC or below 100 VAC require a customer-supplied, properly-sized, step-up or step-down transformer to supply the MP3500 instrument with the proper primary voltage.

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—— Notes ——

Geoprobe® Systems

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GEOPROBE® DT21 DUAL TUBE SOIL SAMPLING SYSTEM

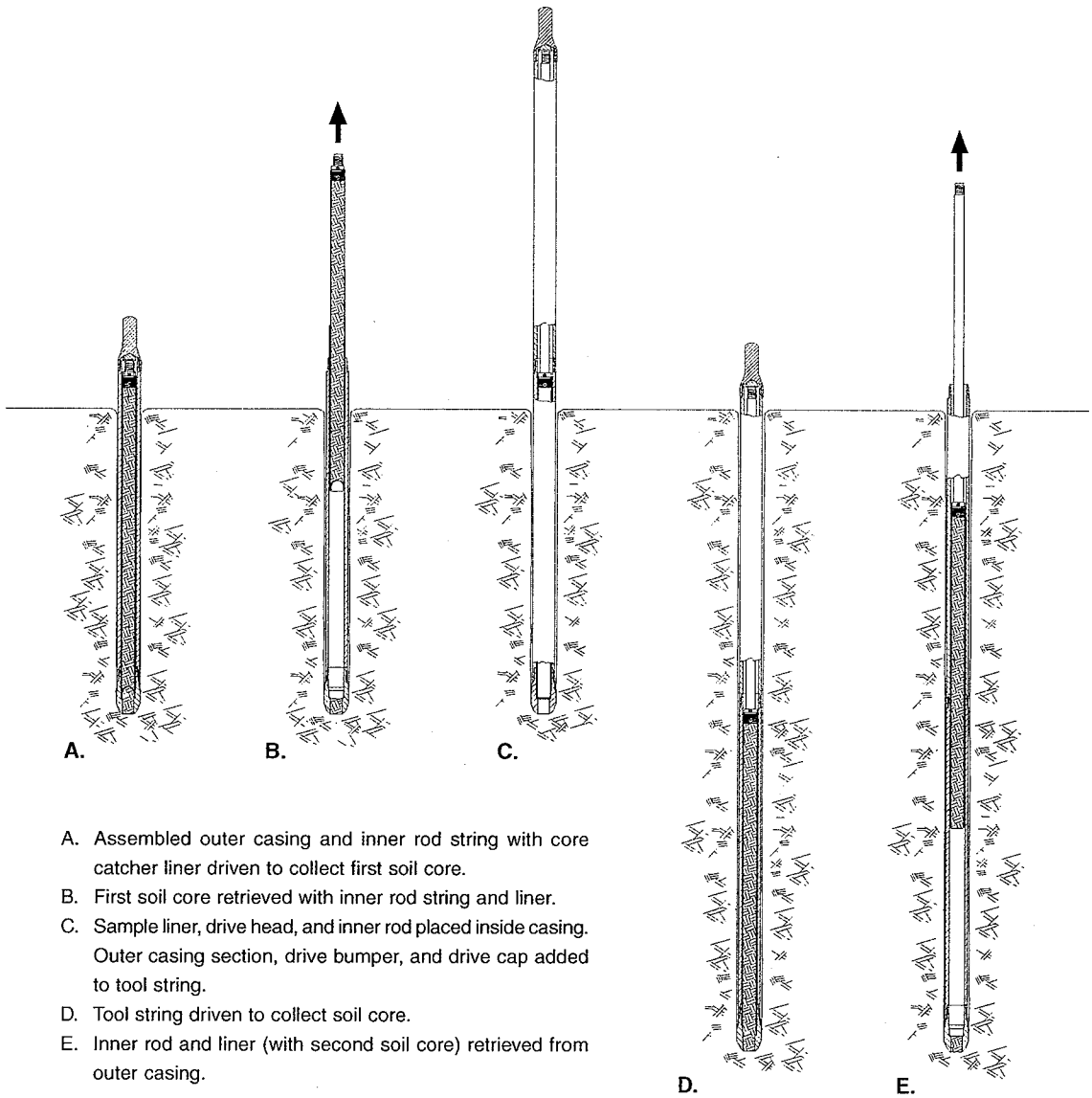
CONTINUOUS CORE SOIL SAMPLER

STANDARD OPERATING PROCEDURE

Technical Bulletin No. 982100

PREPARED: September, 1998

REVISED: September, 2003



A. Assembled outer casing and inner rod string with core catcher liner driven to collect first soil core.
B. First soil core retrieved with inner rod string and liner.
C. Sample liner, drive head, and inner rod placed inside casing. Outer casing section, drive bumper, and drive cap added to tool string.
D. Tool string driven to collect soil core.
E. Inner rod and liner (with second soil core) retrieved from outer casing.

OPERATION OF THE DUAL TUBE 21 SOIL SAMPLING SYSTEM

Geoprobe Systems®

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1.0 OBJECTIVE

The objective of this procedure is to collect a representative soil sample at depth through an enclosed casing and recover it for visual inspection and/or chemical analysis.

2.0 BACKGROUND

2.1 Definitions

Geoprobe®: A brand name of high quality, hydraulically-powered machines that utilize both static force and percussion to advance sampling and logging tools into the subsurface. The Geoprobe® brand name refers to both machines and tools manufactured by Geoprobe Systems®, Salina, Kansas. Geoprobe® tools are used to perform soil core and soil gas sampling, groundwater sampling, soil conductivity and contaminant logging, grouting, and materials injection.
** Geoprobe® is a registered trademark of Kejr Engineering, Inc., Salina, Kansas*

Dual Tube 21 Soil Sampling System: A direct push system for collecting continuous core samples of unconsolidated materials from within a sealed casing of Geoprobe® 2.125-inch (54 mm) OD probe rods. Samples are collected and retrieved within a liner that is threaded onto the leading end of a string of Geoprobe® 1.0-inch (25 mm) OD probe rods and inserted to the bottom of the outer casing. Collected samples measure up to approximately 800 ml in volume in the form of a 1.125-inch x 48-inch (29 mm x 1219 mm) core.

Liner: A 1.375-inch (35 mm) OD thin-walled, PETG tube that is inserted into the outer casing on the leading end of the inner rod string for the purpose of containing and retrieving core samples. Liners are available in two configurations; a simple open tube or a tube with a core catcher permanently attached to the leading end. Nominal liner lengths include 24 inches, 36 inches, 1 meter, and 48 inches.
***Nominal liner length identifies the length of tools with which the liner is used (see Page 8). The actual end-to-end lengths of the various DT21 liners will differ from the specified nominal lengths.*

Core Catcher: A dome-shaped device positioned at the leading end of a liner to prevent loss of collected soil during retrieval of the liner and soil core. Flexible fingers at the top of the core catcher are pushed outward by soil entering the liner during advancement of the tool string. As the filled liner is subsequently retrieved, the fingers of the core catcher move back inward, effectively closing off the end of the liner and limiting soil loss. The core catcher designed for the DT21 system is made of PETG material and is permanently fused to the liner.

2.2 Discussion

Dual tube sampling gets its name from the fact that two sets of probe rods are used to retrieve continuous soil core samples from the subsurface. One set of rods is driven into the ground as an outer casing (Fig. 2.1). These rods receive the driving force from the hammer and provide a sealed casing through which soil samples may be recovered. The second, smaller set of rods are placed inside the outer casing with a sample liner attached to the leading end of the rod string (Fig. 2.1). These smaller rods hold the liner in place as the outer casing is driven to fill the liner with soil. The inner rods are then retracted to retrieve the full liner.

Standard Geoprobe® 2.125-inch OD probe rods provide the outer casing for the DT21 Dual Tube Soil Sampling System. A cutting shoe is threaded into the leading end of the rod string. When driven into the subsurface, the cutting shoe shears a 1.125-inch OD soil core which is collected inside the casing in a clear plastic liner.

The second set of rods in the DT21 system are standard Geoprobe® 1.0-inch OD probe rods. A sample liner is attached to the end of these smaller rods and then inserted into the casing. The 1.0-inch rods hold the liner tight against the cutting shoe as the outer casing is driven to collect the soil core. Once filled with soil, the liner is removed from the bottom of the outer casing by lifting out the 1.0-inch rods.

The outer, 2.125-inch probe rods provide a cased hole through which to sample. The main advantage of sampling through a cased hole is that there is no side slough to contend with. In addition, the outer casing effectively seals the probe hole when sampling through perched water tables. These factors mean that sample cross-contamination is eliminated. The DT21 sampling system is therefore ideal for continuous coring in both saturated and unsaturated zones.

Solid Drive Tip

A Solid Drive Tip (DT4070) can be placed on the leading end of the 1.0-inch probe rod string in place of a sample liner (Fig. 2.2). When installed in the outer casing, the drive tip firmly seats within the cutting shoe and effectively seals the tool string as it is driven into the subsurface. This enables the operator to advance the outer casing to the bottom of a pre-cored hole or through undisturbed soil to reach the top of the sampling interval.

Grouting

The DT21 system allows bottom-up grouting through the primary tool string. This means that a cement or bentonite grout mix can be pumped through the outer casing as it is withdrawn from the ground. This is in contrast to most other soil samplers which require driving a second set of tools back down the probe hole in order to deliver the grout mix.

Monitoring Well Installation

An expendable cutting shoe enables the operator to install a Geoprobe® Prepacked Screen Monitoring Well through the outer casing of the DT21 Dual Tube System. After the collection of continuous soil cores to the desired depth, prepacked screens can be inserted to the bottom of the outer casing on the leading end of a PVC riser string. The well is finished, complete with grout barrier, bentonite well seal, and a high-solids bentonite slurry/neat cement grout, during retrieval of the outer casing.

Groundwater Profiler

The DT21 Groundwater Profiler Kit (GW2100) brings groundwater sampling to the dual tube system. With these tools, operators can perform soil sampling, groundwater sampling, and slug testing at multiple depth intervals in one probe push.

The DT21 profiler utilizes a 3/4-inch Schedule 40 PVC screen with a slotted length of 12 inches. Standard Geoprobe® extension rods are used to install and retrieve the screen through the cutting shoe on the leading end of the outer casing. This ability to install and retrieve the screen with the outer casing in place allows for alternating of soil and groundwater sampling and slug testing in a single probe push.

For specific information on using the DT21 profiler, refer to *Dual Tube (DT21) Groundwater Profiler Kit - Installation and Operation Instructions* (Geoprobe® Instructional Bulletin No. 19275).

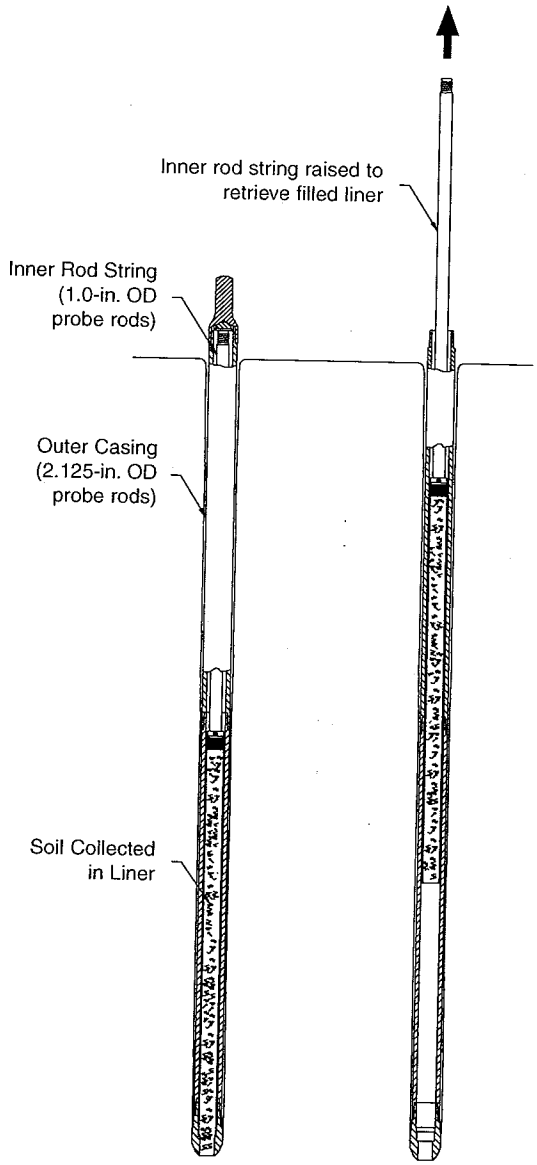

FIGURE 2.1
Outer Casing Driven With Liner

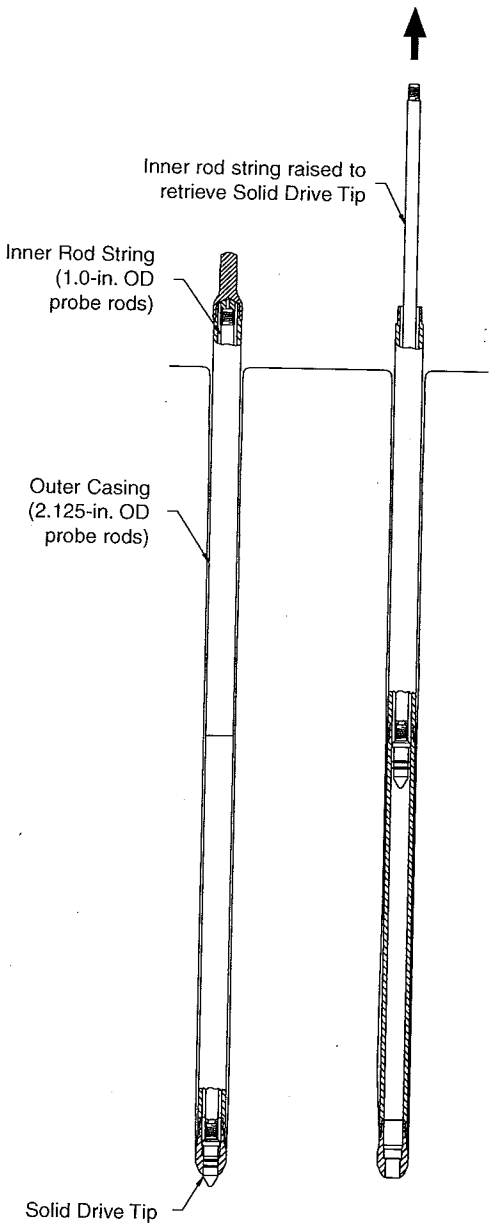

FIGURE 2.2
Outer Casing Driven With Solid Drive Tip

3.0 REQUIRED EQUIPMENT

The following equipment is used to recover samples with the Geoprobe® Dual Tube 21 Soil Sampling and probing systems. Note that the operator may choose to utilize 2.125-inch probe rods in lengths of 36 inches (914 mm), 1 meter, or 48 inches (1219 mm). It is not necessary to have all three rod lengths on hand. Refer to Figure 3.1 for parts identification.

DUAL TUBE 21 SAMPLER PARTS	QUANTITY	PART NUMBER
DT21 Drive Bumper, Pkg. of 5	-1-	DT4010K
DT21 Threadless Drive Cap, for 1.0-inch rods	-1-	DT4020
DT21 Cutting Shoe, Large, 2.375 in. OD	-1-	DT4030
DT21 Cutting Shoe, Small, 2.205 in. OD	-1-	DT4035
DT21 Expendable Cutting Shoe Holder	-1-	DT4040
DT21 Expendable Cutting Shoe, 2.375 in. OD	variable	DT4045
O-rings for Expendable Cutting Shoe, Pkg. of 50	variable	DT4045R
DT21 Liner Drive Head Assembly, for 1.0-inch rods	-1-	DT4050
Rebuild Kit for DT21 Liner Drive Head	-1-	DT4051K
DT21 Rod Clamp Assembly, for 1.0-inch rods	-1-	DT4060
DT21 Solid Drive Tip, for 1.0-inch rods	-1-	DT4070
O-rings for Solid Drive Tip, Pkg. of 25	variable	DT4070R
DT21 Liners, PETG, 24-inch, Box of 50*	variable	DT4024K
DT21 Vinyl End Caps, Pkg. of 100 (50 pair)	variable	DT4026K
DT21 Liner, PETG, 36-inch, Box of 50*	variable	DT4036K
DT21 Liner, PETG, 1-meter, Box of 50*	variable	DT4039K
DT21 Liner, PETG, 48-inch, Box of 50*	variable	DT4048K
DT21 Liner With Core Catcher, PETG, 36-inch, Box of 50*	variable	DT4136K
DT21 Liner With Core Catcher, PETG, 1-meter, Box of 50*	variable	DT4139K
DT21 Liner With Core Catcher, PETG, 48-inch, Box of 50*	variable	DT4148K
DT21 Liner Grooving Tool	-1-	18170
MC Liner Holder, for use with 1-meter and shorter liners	-1-	AT8020
MC Liner Holder, for use with 48-inch and shorter liners	-1-	15679

GEOPROBE TOOLS AND EQUIPMENT	QUANTITY	PART NUMBER
Probe Rod, 1.0 inch OD x 36 inches*	variable	AT10B
Probe Rod, 1.0 inch OD x 1 meter (39 inches)*	variable	AT1039
Probe Rod, 1.0 inch OD x 48 inches*	variable	AT104B
Probe Rod, 1.0 inch OD x 24 inches*	variable	AT105B
O-rings for 2.125-inch Probe Rods	variable	AT2100R
GH40 Threaded Drive Cap, for 2.125-inch rods	-1-	AT2101
GH60 Threaded Drive Cap, for 2.125-inch rods**	-1-	15673
Pull Cap, for 2.125-inch rods	-1-	AT2104
Probe Rod, 2.125 inches OD x 36 inches*	variable	AT2136
Probe Rod, 2.125 inches OD x 1 meter (39 inches)*	variable	AT2139
Probe Rod, 2.125 inches OD x 48 inches*	variable	AT2148
MC Combination Wrench	-1-	AT8590
Rod Grip Pull System, for GH40 Hammer	-1-	GH3000K
Rod Grip Pull Handle, for GH60 Hammer and 2.125-inch rods**	-1-	9640
Rod Grip Pull Handle, for GH60 Hammer and 1.0-/1.25-inch rods**	-1-	9641

ADDITIONAL TOOLS	QUANTITY
Hex Key, 3/32 in.	-1-
Utility Knife (with straight blade)	-1-
Pipe Wrench	-2-

* Match length of probe rods to desired liner length. Use 36-inch probe rods with 36-inch liners, 1-meter probe rods with 1-meter liners, and 48-inch probe rods with 24- and 48-inch liners. A 1.0 inch x 24 inches probe rod is also required when utilizing 24-inch sample liners.

** The considerable percussive force of the GH60 Hydraulic Hammer may result in reduced tool life for components such as the DT21 Drive Bumper and rod string as compared to operation with the GH40 Hydraulic Hammer.

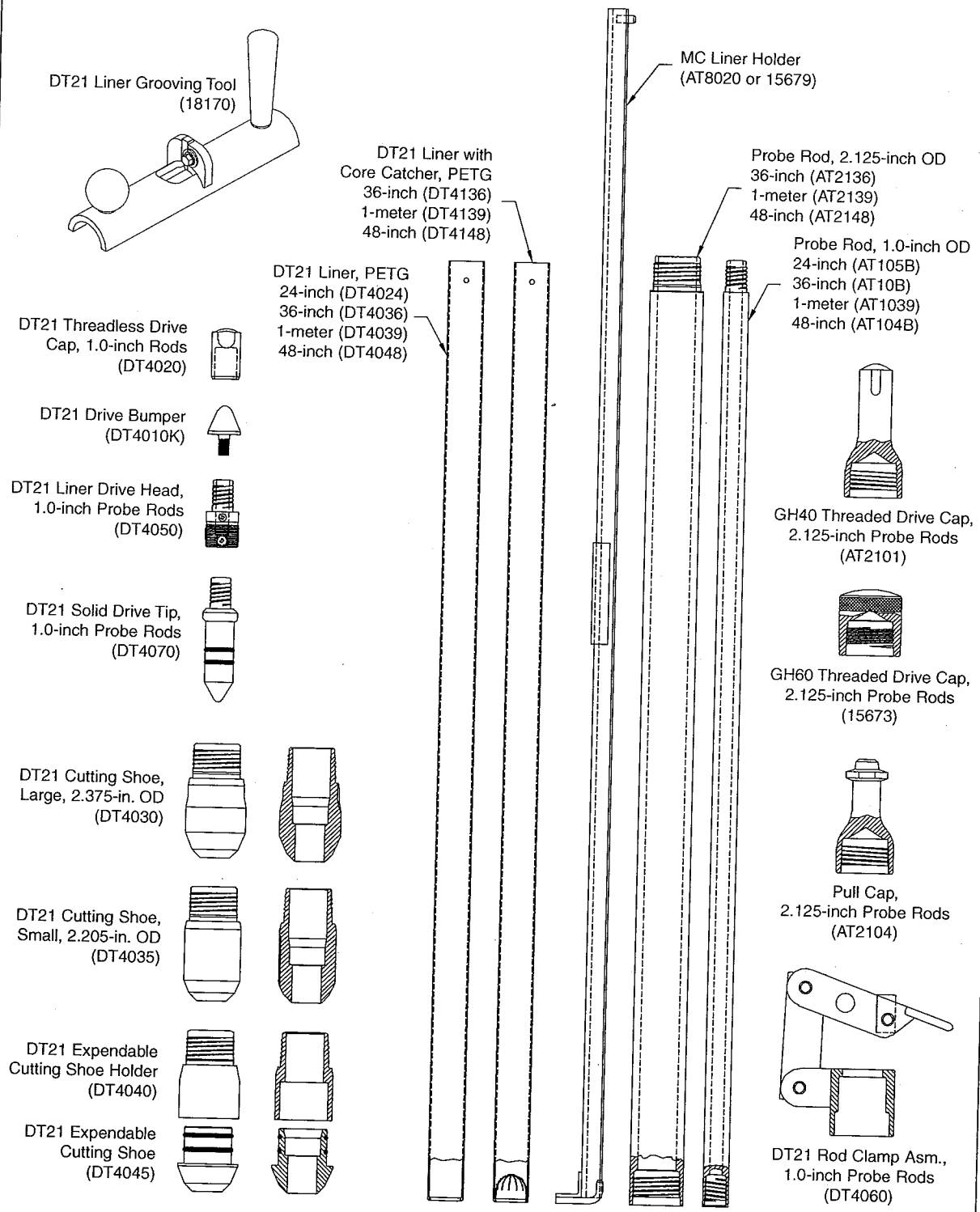

FIGURE 3.1
Dual Tube 21 Soil Sampling System Parts

3.1 Tool Options

Three major components of the DT21 Soil Sampling System are probe rods, sample liners, and cutting shoes. These items are manufactured in a variety of sizes to fit the specific needs of the operator. This section identifies the specific tool options available for use with the DT21 Dual Tube System.

Probe Rods

Standard Geoprobe® 1.0-inch (25 mm) OD and 2.125-inch (54 mm) OD probe rods are required to operate the DT21 Soil Sampling System. The specific length of rods may be selected by the operator. Available rod lengths are 36 inches (914 mm), 48 inches (1,219 mm), and 1 meter. Both rod sets (1.0-inch and 2.125-inch) must be of the same length.

Sample Liners

Sample liners are made of a heavy-duty clear plastic for convenient inspection of the soil sample. Nominal lengths of 24 inches, 36 inches, 1 meter, and 48 inches are available with an OD of 1.375 inches (35 mm). Choose the liner length corresponding to the length of probe rods used (e.g. 36-inch liners with 36-inch probe rods).

The shorter length of the 24-inch liners helps to recover samples from flowing sands and highly expansive clays. These liners are used with 48-inch probe rods, but also require a single 1.0-inch x 24-inch probe rod.

Sample liners with integral core catchers are available in nominal lengths of 36 inches, 1 meter, and 48 inches. Utilize the core catcher liners when sampling flowing sands, noncohesive soils, extremely dry soils, or any other materials that fall from the liner during retrieval. DT21 core catcher liners are used with the same equipment as open sample liners. No special tooling or adapters are required.

Cutting Shoes

Three cutting shoes are available for use with the DT21 Dual Tube System (Fig. 3.2). The DT21 Large Cutting Shoe (DT4030) and DT21 Small Cutting Shoe (DT4035) thread into the leading end of the 2.125-inch probe rods and are recovered after sampling. Dimensions for the large cutting shoe are 1.125 inches (29 mm) ID and 2.375 inches (60 mm) OD. The small cutting shoe also has an ID of 1.125 inches (29 mm) but the OD is only 2.205 inches (56 mm). To reduce side friction and make driving easier, the large cutting shoe (DT4030) is oversized to provide a small annulus between the outer casing and soil. By contrast, the small cutting shoe (DT4035) is for use in formations where an annulus is undesirable or where expansive soil conditions exist.

The DT21 sampling system may also employ an expendable cutting shoe. In this arrangement, a DT21 Expendable Cutting Shoe Holder (DT4040) is threaded into the leading end of the outer casing. A DT21 Expendable Cutting Shoe (DT4045) is then inserted into the holder. Upon completion of soil sampling, the outer casing is withdrawn slightly. The expendable cutting shoe detaches from the holder, leaving an open casing through which a prepacked screen monitoring well may be installed. Dimensions for the expendable cutting shoe are the same as the large cutting shoe (ID = 1.125 in. (29 mm) and OD = 2.375 in. (60 mm)).

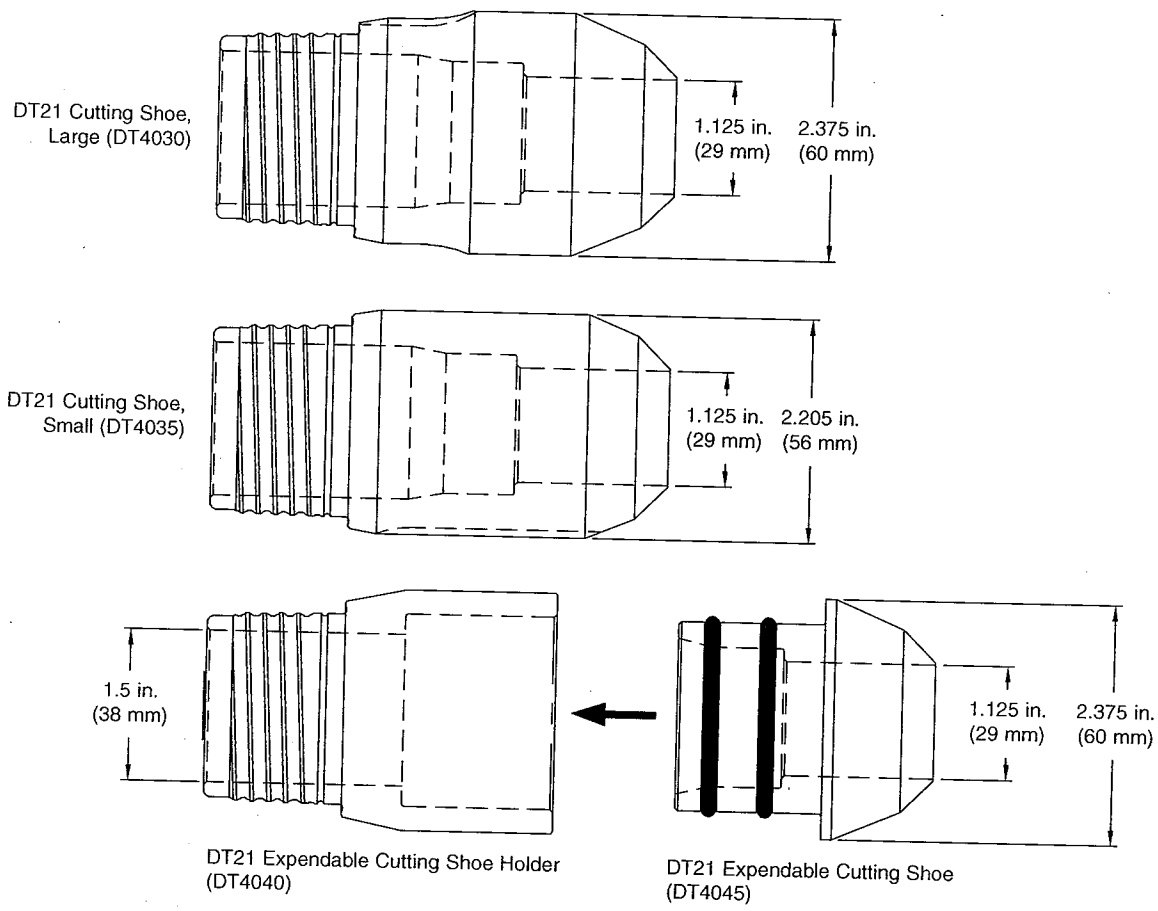

FIGURE 3.2
Cutting Shoe Options for the DT21 Dual Tube Soil Sampling System

4.0 OPERATION

4.1 Decontamination

Before and after each use, thoroughly clean all parts of the soil sampling system according to project requirements. Parts should also be inspected for wear or damage at this time. During sampling, a clean new liner is used for each soil core.

4.2 Operational Overview

The DT21 Soil Sampling System is designed to collect continuous soil cores. Sampling may begin either from ground surface or a predetermined depth below ground. Once sampling begins, consecutive soil cores must be removed as the outer casing is advanced to greater depths

(continued on following page)

When sampling is to begin at the ground surface, the first soil core should be collected using a core catcher liner to maximize sample recovery (Fig. 4.1-A). This is especially true when the first core is composed of dry, loose soil. Upon removal of the first liner and soil core (Fig. 4.1-B), a new liner is inserted to the bottom of the outer casing on the end of an inner rod. A section of outer casing is added to the tool string (Fig. 4.1-C) and the entire tool string is driven to fill the liner with soil (Fig. 4.1-D). The filled liner is removed from the outer casing to retrieve the second soil core (Fig. 4.1-E). A new liner is then inserted to the bottom of the outer casing and the process is repeated over the entire sampling interval.

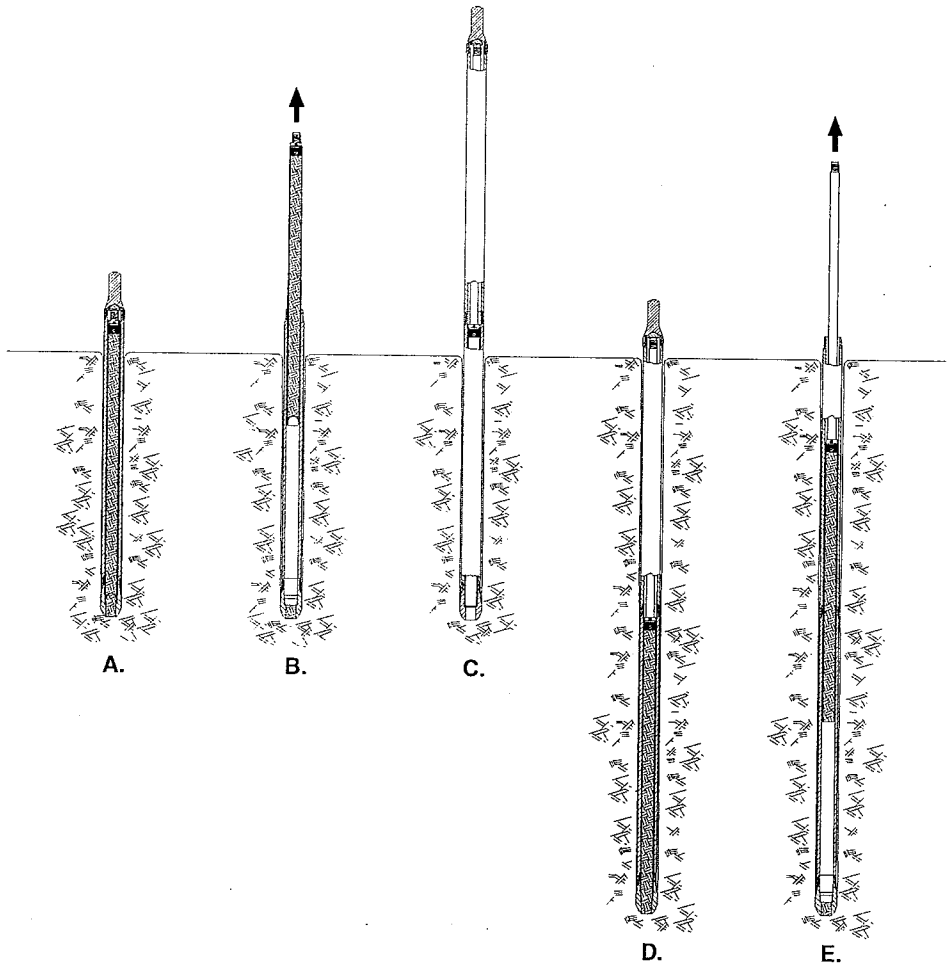

FIGURE 4.1
Continuous Core Sampling From Ground Surface with Dual Tube 21 System

When the sampling interval begins at some depth below ground surface, a DT21 Solid Drive Tip is installed in the outer casing and the entire assembly is driven from ground surface directly through undisturbed soil (Fig. 4.2-A). This enables the operator to reach the top of the sampling interval without stopping to remove unwanted soil cores. Once the interval is reached, the solid drive tip is removed (Fig 4.2-B) and sampling continues as described in the preceding paragraphs (Fig. 4.2-C, Fig. 4.2-D, and Fig. 4.2-E).

Specific instructions for the assembly and operation of the DT21 Dual Tube Soil Sampling System are given in the following sections.

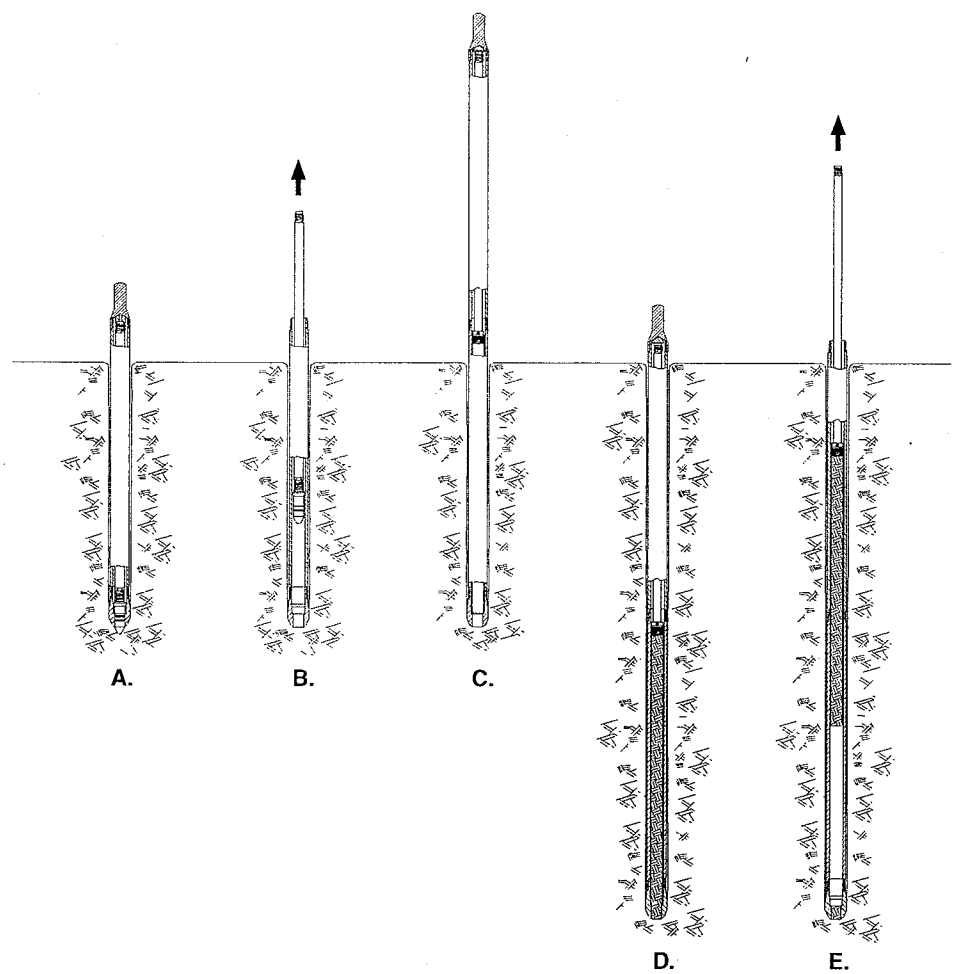

FIGURE 4.2
Outer Casing Driven Through Undisturbed Soil to Begin Sampling with DT21 System

4.3 Assembling and Driving the Outer Casing Using a DT21 Solid Drive Tip

If soil sampling is to begin at some depth below ground surface, the outer casing of the DT21 Dual Tube System can be driven to the top of the sampling interval with a DT21 Solid Drive Tip installed in the leading end. The solid drive tip seals the outer casing as it is driven to depth. Once the desired sampling interval is reached, the solid drive tip is removed to allow collection of the first soil core. This section describes assembling and driving the outer casing using the DT21 Solid Drive Tip.

1. If using a DT21 Large or Small Cutting Shoe (DT4030 or DT4035) install an O-ring (AT2100R) at the base of the threads as shown in Figure 4.3. If using an expendable cutting shoe, install an AT2100R O-ring on the DT21 Expendable Cutting Shoe Holder (DT4040) and two DT4045R O-Rings on the DT21 Expendable Cutting Shoe (DT4045) (Fig. 4.3).

2. Thread the DT21 Cutting Shoe or DT21 Expendable Point Holder into the leading end of a 2.125-inch OD Probe Rod (AT2136, AT2139, or AT2148). Completely tighten the cutting shoe or cutting shoe holder using a machine vise and MC Combination Wrench (AT8590) as shown in Figure 4.4.

3. Install an O-ring (DT4070R) in both grooves of the DT21 Solid Drive Point (DT4070) (Fig.4.5).

4. Thread the solid drive point into the female end of a 1.0-inch OD probe rod of the same length as the 2.125-inch probe rod (outer casing).

5. Lubricate the O-rings on the solid drive point with a small amount of deionized water. Insert the point and probe rod into the outer casing until the point partially extends from the bottom of the cutting shoe.

6. Place a DT21 Threadless Drive Cap (DT4020) on top of the inner rod (Fig. 4.6). This drive cap is threadless for quick installation/removal, yet still provides protection for the probe rod threads.

7. Thread a GH40 Drive Cap (AT2101) or GH60 Threaded Drive Cap (15673) onto the 2.125-inch probe rod (outer casing) as shown in Figure 4.6.

   For GH40 Users: Current AT2101 Drive Caps are manufactured using a process that leaves an angled surface inside the cap (Fig. 4.7). This provides room for the Threadless Drive Cap (DT4020). Older AT2101 Drive Caps were manufactured using a process that leaves a flat surface inside the cap (Fig. 4.7). These older drive caps cannot be used with the DT21 Dual Tube System.

   For GH60 Users: A GH60 Threaded Drive Cap (15673) is recommended for the 2.125-inch probe rods when driving the DT21 system. The threaded drive cap secures the inner rod string during percussion so that the outer casing and inner rod string are advanced as one assembly. The standard threadless drive cap may allow the outer casing to advance slightly ahead of the inner rod string, resulting in poor sample recovery and alignment problems when adding rods to the tool string.

   NOTE: Do not allow the threaded drive cap to unthread more than approximately 1/8 inch while driving the tool string. Failure to keep the drive cap tight during percussion will fuse the drive cap to the outer casing and permanently damage the threads of both the drive cap and top probe rod.

(continued on Page 14)

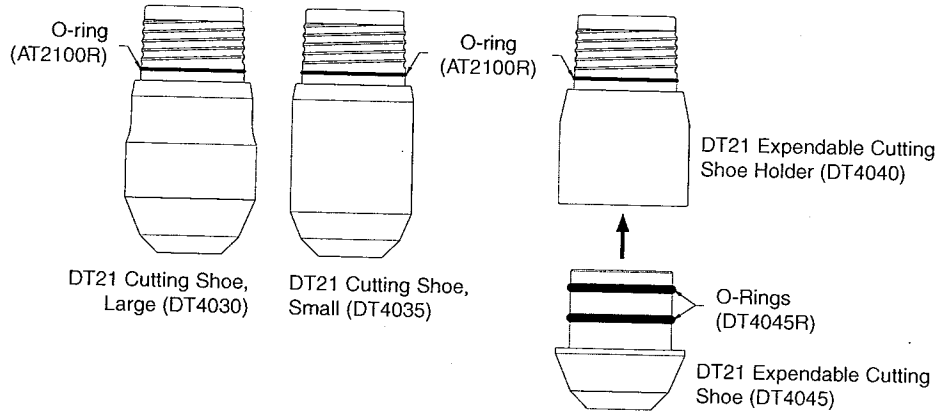

FIGURE 4.3
DT21 Cutting Shoes Utilize O-ring Seals

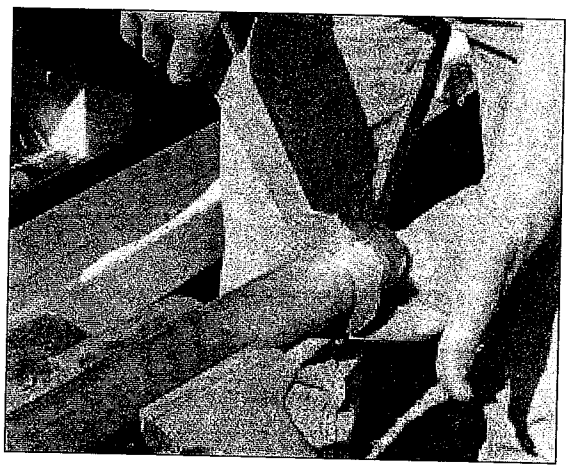

Figure 4.4. Place probe rod (outer casing) in vise and tighten cutting shoe with MC Combination Wrench.

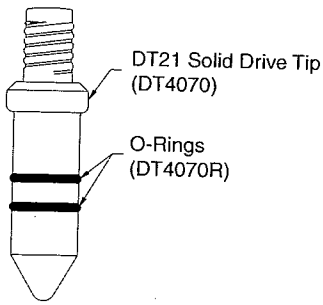

FIGURE 4.5
Solid Drive Point Utilizes Two O-Rings

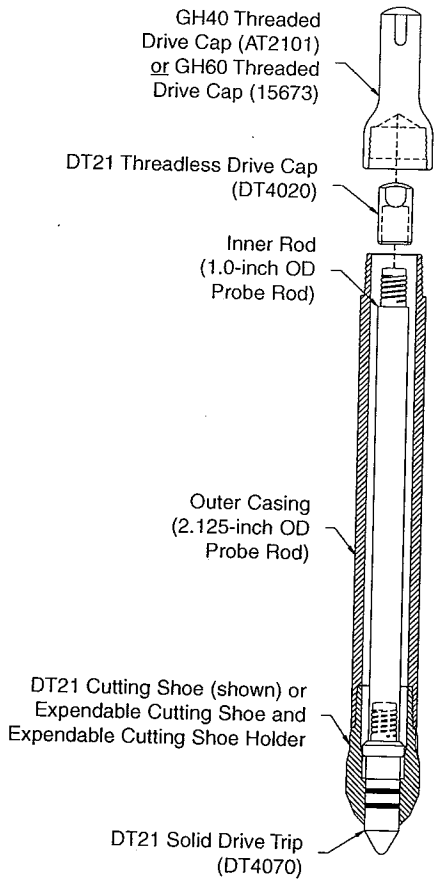

FIGURE 4.6
Outer Casing with Solid Drive Tip

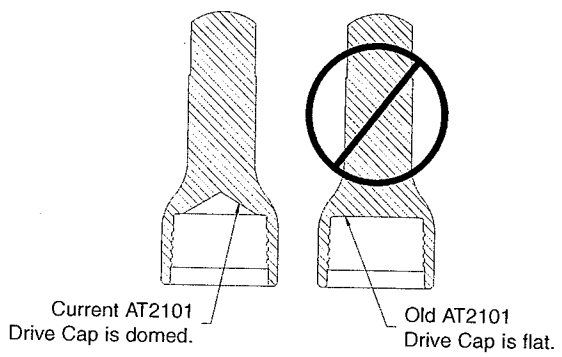

FIGURE 4.7
Comparison of AT2101 Drive Caps

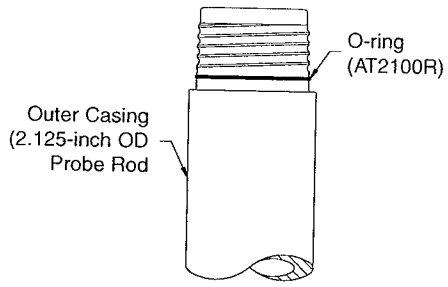

FIGURE 4.8
Outer Casing Joints are Sealed with O-Rings

8. Place the assembled outer casing section under the direct push machine for driving. Position the casing directly under the hammer with the cutting shoe centered between the toes of the probe foot.

9. Lower the hydraulic hammer onto the drive cap and advance the outer casing into the subsurface.

10. Raise the hydraulic hammer and remove the drive cap from the outer casing and the threadless drive cap from the inner rod string.

11. Place an O-ring (AT2100R) on the outer casing section that extends from the ground (Fig. 4.8).

12. Thread a 1.0-inch probe rod onto the inner rod string. Place a 2.125-inch probe rod over the inner rods and thread it onto the outer casing (Fig. 4.9). Completely tighten the outer casing using a pipe wrench.

13. Place the threadless drive cap on top of the inner rod. Thread the 2.125-inch drive cap over the threadless drive cap and onto the outer casing.

14. Lower the hydraulic hammer onto the drive cap and advance the outer casing into the subsurface.

    Repeat Steps 10-13 until the leading end of the outer casing is at the top of the proposed sampling interval. Continue with Step 15 to remove the DT21 Solid Drive Point for sampling.

15. Raise the hydraulic hammer and retract the probe derrick to provide access to the top of the tool string.

16. Unthread the 2.125-inch drive cap and remove the threadless drive cap from the inner rods.

17. Thread a 1.0-inch OD probe rod onto the inner rod string. Lift and remove the inner rods from the outer casing. The DT21 Solid Drive Point is removed from the leading end of the casing with the inner rods.

    NOTE: Hold the inner rod string with a DT21 Rod Clamp Assembly (DT 4060) while unthreading the retracted rods (4.10). Use the probe hammer and Rod Grip Pull System (GH3000K) to pull the inner rods if they are too heavy to lift comfortably by hand (Fig. 4.11).

The outer casing is now ready for sampling. Continue with Section 4.4 for more instructions.

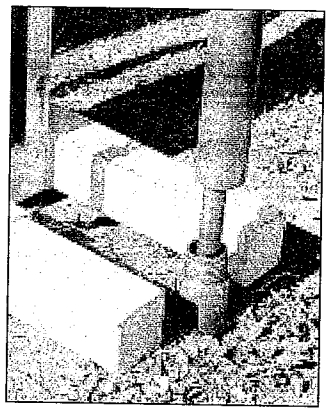

Figure 4.9. Place a 2.125-inch probe rod over the 1.0-inch rod and thread it onto the outer casing string.

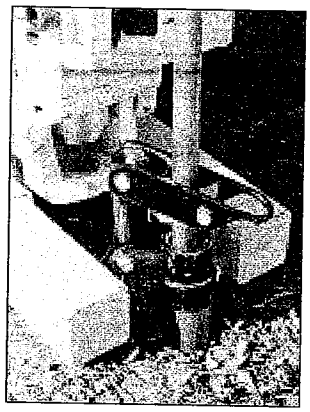

Figure 4.10. The DT21 Rod Clamp Assembly holds the inner rod string while adding or removing 1.0-inch rods.

Figure 4.11. Raise the inner rod string using the probe hammer and a Rod Grip Pull System if rods are too heavy to lift by hand.

4.4 Liner Drive Head Assembly

The main function of the DT21 Liner Drive Head Assembly (DT4050) is to connect a liner to the leading end of the inner rod string. This enables the inner rods to hold the liner tight against the cutting shoe to fill the liner with soil as the outer casing is driven. The inner rods are then used to retrieve the liner and soil core from within the outer casing.

The liner drive head assembly includes an internal check ball to improve sample recovery (Fig. 4.12) A considerable vacuum is created below the filled liner as it is lifted from the bottom of the outer casing. Because the inner rod string and liner drive head are hollow, atmospheric air can travel through the rods creating a positive pressure differential above the soil core during retrieval. The check ball seals the liner drive head to eliminate air flow into the liner which could otherwise push the soil sample out of the liner. The check ball also allows air to escape up through the liner drive head and inner rod string when lowering a new liner down the outer casing and as soil enters the liner during sampling.

Saturated conditions can also challenge sample recovery. Water enters the outer casing either from the saturated formation or is deliberately poured from the ground surface to keep flowing sands out of the casing. As with air in unsaturated formations, the check ball lets water pass through the liner drive head as a new liner is lowered to the bottom of the casing and during sampling as the liner is filled with soil. The check ball then seals the drive head during retrieval so that water draining from the inner rods does not wash the sample out the bottom of the liner. A drain hole located on the side of the liner drive head (Fig. 4.12) allows water to exit the inner rods and travel harmlessly along the outside of the liner.

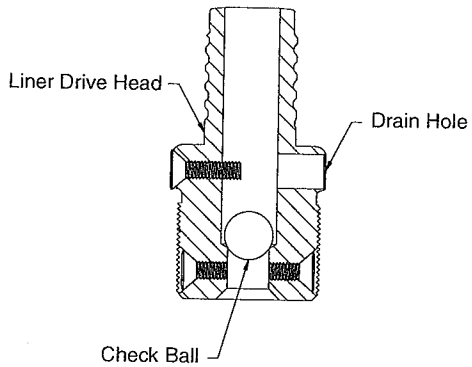

FIGURE 4.12
Cross-Section of Liner Drive Head

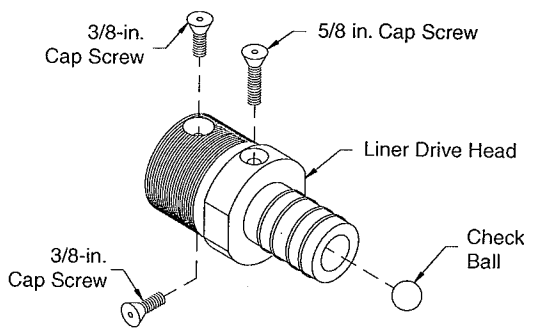

FIGURE 4.13
Liner Drive Head Assembly Components

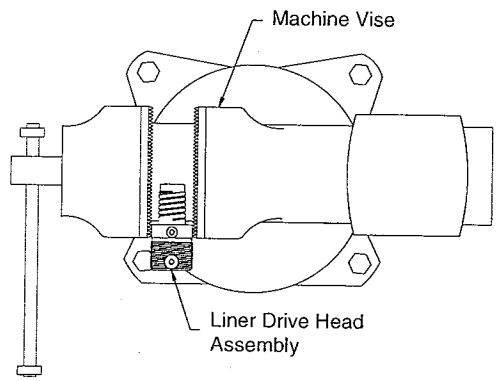

FIGURE 4.14
Place Liner Drive Head in Vise

The liner drive head assembly is made up of five parts as shown in Figure 4.13. The two 3/8-inch flat head socket cap screws are used to attach liners to the liner drive head. The longer 5/8-inch flat head socket cap screw holds the stainless steel check ball within the liner drive head. To disassemble the liner drive head for cleaning, simply unthread the 5/8-inch cap screw and remove the check ball.

Instructions for attaching a liner to the DT21 Liner Drive Head Assembly (DT4050) are given below.

1. Visually inspect the liner drive head assembly to ensure that the check ball moves freely within the drive head and the drain hole is unobstructed.

2. Place the liner drive head assembly in a machine vise so that either one of the 3/8-inch caps screws is on top as shown in Figure 4.14.

   NOTE: Only one 3/8-inch cap screw is used to attach a liner to the liner drive head assembly. Two 3/8-inch cap screws are included on the drive head to provide a backup in case one incurs thread damage. Either cap screw may be used to attach the liner.

3. Remove the 3/8-inch cap screw using a 3/32-inch hex key.

4. Place the open end of a DT21 Liner against the bottom of the liner drive head. Align the hole in the liner with the hole in the liner drive head as shown in Figure 4.15. Wiggle the free end of the liner back-and-forth while pushing the liner onto the drive head (Fig. 4.15).

   NOTE: Use the DT21 Liner that matches the length of your probe rods (36 inches, 1 meter, or 48 inches). The 24-inch DT21 Liners (DT4024K) are to be used with 48-inch probe rods only. One 1.0-inch x 24-inch probe rod (AT105B) is also required when using the DT4024K liners.

5. Thread the 3/8-inch cap screw through the liner and back into the liner drive head (Fig. 4.16). Tighten the cap screw with the 3/32-inch hex key.

   The DT21 Liner is now attached to the DT21 Liner Drive Head Assembly (Fig. 4.17).

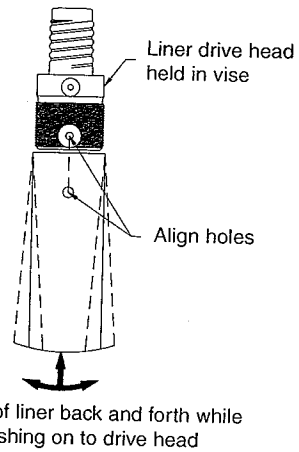

FIGURE 4.15
Push Liner Onto Liner Drive Head

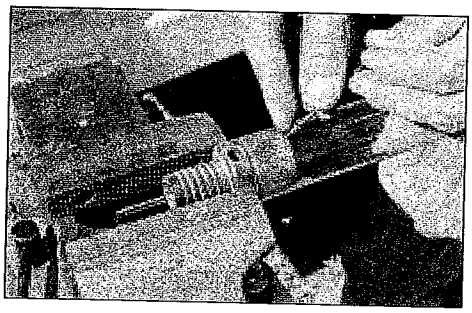

Figure 4.16. Thread cap screw into liner drive head to secure liner.

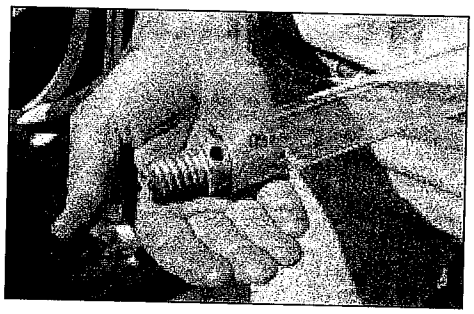

Figure 4.17. Liner attached to liner drive head and ready for sampling.

4.5 Soil Core Collection

This section describes collection of continuous soil core samples from within the sealed outer casing of the DT21 Dual Tube Soil Sampling System. The procedure is written for a sampling series that begins at the ground surface. If sampling is to begin after driving the outer casing through undisturbed soil using a DT21 Solid Drive Tip, skip ahead to Step 13 of this section.

1. Select a DT21 Large Cutting Shoe (DT4030), DT21 Small Cutting Shoe (DT4035), or DT21 Expendable Cutting Shoe (DT4045) and DT21 Expendable Cutting Shoe Holder (DT4040).

   If using a large or small cutting shoe, install an O-ring (AT2100R) at the base of the threads as shown in Figure 4.18. If using an expendable cutting shoe, install an AT2100R O-ring on the expendable cutting shoe holder and two DT4045R O-Rings on the expendable cutting shoe (Fig. 4.18).

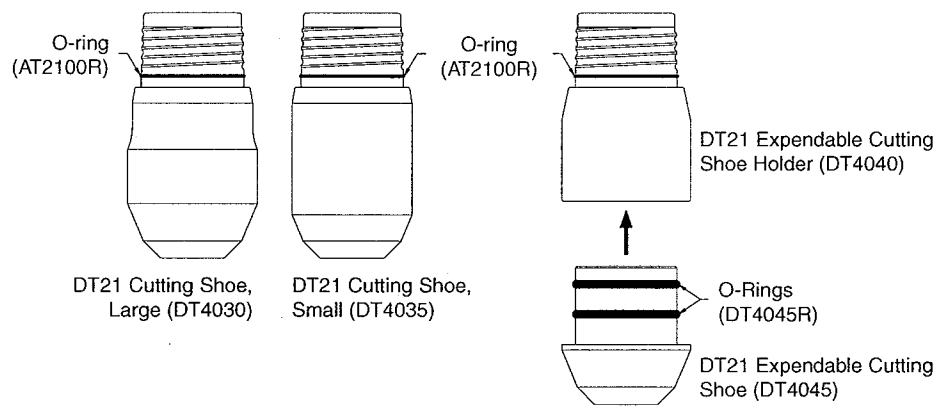

FIGURE 4.18
DT21 Cutting Shoes Utilize O-ring Seals

2. Thread the DT21 Cutting Shoe or DT21 Expendable Point Holder into the leading end of a 2.125-inch OD Probe Rod (AT2136, AT2139, or AT2148). Completely tighten the cutting shoe or cutting shoe holder using a machine vise and MC Combination Wrench (AT8590) as shown in Figure 4.19.

3. Attach a DT21 Liner Drive Head Assembly (DT4050) to a new liner as described in Section 4.4. A core catcher liner (DT4136, DT4139, or DT4148) is recommended for when the first soil core begins at ground surface as this configuration will provide maximum sample recovery.

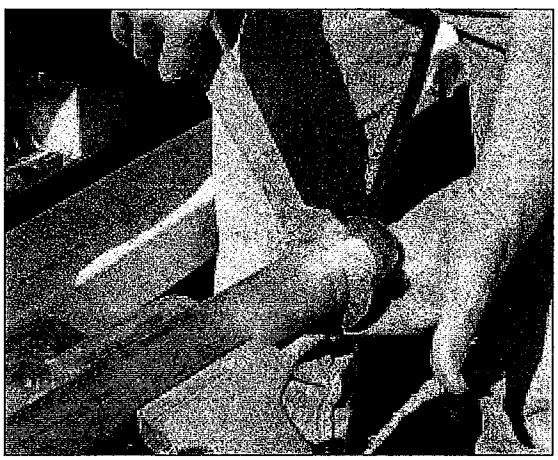

Figure 4.19. Place probe rod (outer casing) in vise and tighten cutting shoe with MC Combination Wrench.

4. Insert the liner and drive head into the 2.125-inch OD probe rod such that the core catcher contacts the cutting shoe as shown in Figure 4.20.

5. Place a DT21 Drive Bumper (DT4010K) on top of the liner drive head (Figure 4.20).

6. Thread a GH40 Drive Cap (AT2101) or GH60 Threaded Drive Cap (15673) onto the 2.125-inch probe rod (outer casing) as shown in Figure 4.20.

   For GH40 Users: Current AT2101 Drive Caps are manufactured using a process that leaves an angled surface inside the cap (Fig. 4.21). This provides room for the Threadless Drive Cap (DT4020). Older AT2101 Drive Caps were manufactured using a process that leaves a flat surface inside the cap (Fig. 4.21). These older drive caps cannot be used with the DT21 Dual Tube System.

   For GH60 Users: A GH60 Threaded Drive Cap (15673) is recommended for the 2.125-inch probe rods when driving the DT21 system. The threaded drive cap secures the inner rod string during percussion so that the outer casing and inner rod string are advanced as one assembly. The standard threadless drive cap may allow the outer casing to advance slightly ahead of the inner rod string, resulting in poor sample recovery and alignment problems when adding rods to the tool string.

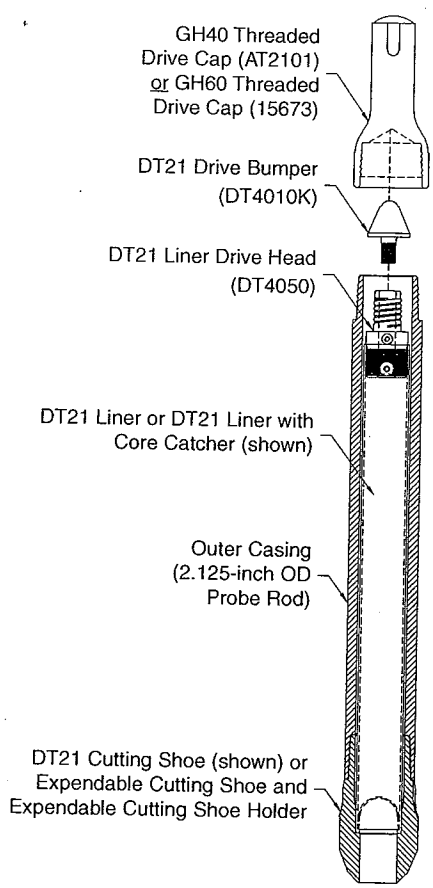

FIGURE 4.20
Outer Casing with DT21 Liner

NOTE: Do not allow the threaded drive cap to unthread more than approximately 1/8 inch while driving the tool string. Failure to keep the drive cap tight during percussion will fuse the drive cap to the outer casing and permanently damage the threads of both the drive cap and top probe rod.

7. Place the assembled outer casing section under the direct push machine for driving. Position the casing directly under the hydraulic hammer with the cutting shoe centered between the toes of the probe foot.

8. Lower the hydraulic hammer onto the drive cap and advance the outer casing into the subsurface using continuous percussion.

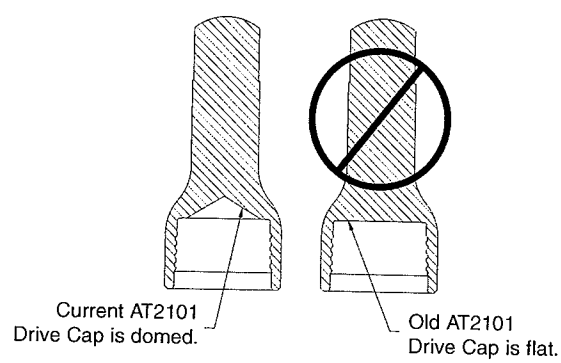

FIGURE 4.21
Comparison of AT2101 Drive Caps

9. Raise the hydraulic hammer and move the probe assembly back to provide access to the top of the tool string.

10. Remove the drive cap and drive bumper

11. Thread a 1.0-inch probe rod onto the liner drive head. Rotate the probe rod and liner assembly two or three revolutions to shear the soil core at the bottom of the liner. Lift the probe rod and filled liner from the outer casing to retrieve the first soil core.

12. Remove the filled liner from the liner drive head as described in Section 4.6. Prepare the soil core for subsampling or storage as specified by the project plan.

13. Place an O-ring (AT2100R) in the groove just below the male threads on the top section of the outer casing (Fig. 4.22).

14. Thread a 1.0-inch probe rod onto an assembled DT21 Liner Drive Head (DT4050) and DT21 Liner (Fig. 4.23).

NOTE: A 1.0-inch x 24-inch Probe Rod (AT105B) is first threaded onto the DT21 Liner Drive Head Assembly (DT4050) if using a 24-inch DT21 Liner (DT4024K).

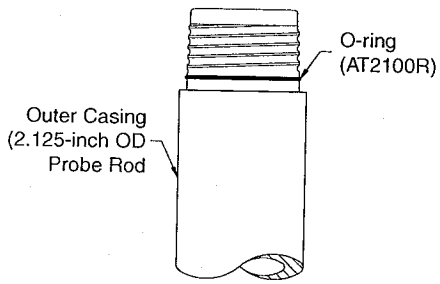

FIGURE 4.22
Outer Casing Joints are Sealed with O-Rings

Figure 4.23. Thread liner and liner drive head into 1.0-inch probe rod.

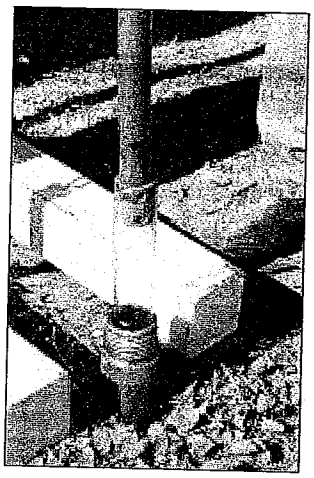

Figure 4.24. Lower liner to bottom of outer casing on leading end of inner rods.

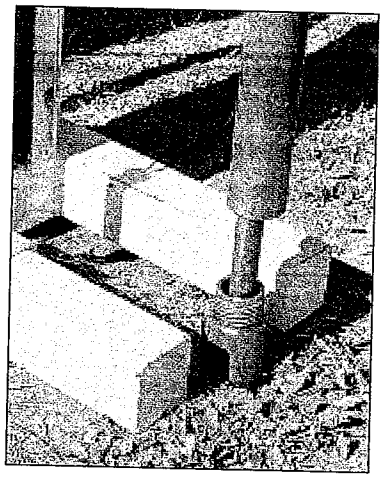

Figure 4.25. Place a 2.125-inch probe rod over the 1.0-inch probe rod and thread it onto the outer casing string.

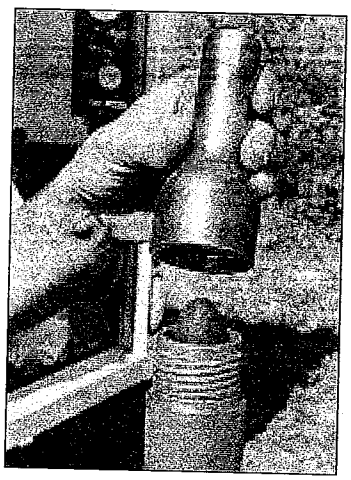

Figure 4.26. Place a drive bumper on top of the inner rods and thread a drive cap onto the outer casing.

15. Insert the liner and probe rod into the outer casing (Fig. 4.24).

The inner rod will extend past the top of the outer casing if only one section of casing was previously driven into the ground. If the casing was driven to a greater depth, continue adding 1.0-inch probe rods until the last rod extends from the casing. Use the DT21 Rod Clamp Assembly to hold the inner tool string while adding rods if desired.

16. Place a 2.125-inch probe rod over the inner rods and thread it onto the outer casing (Fig. 4.25). Completely tighten the outer casing using a pipe wrench.

17. Place a DT21 Drive Bumper (DT4010K) on top of the inner rod as shown in Figure 4.26.

18. Thread a Drive Cap (AT2101 or 15673) onto the 2.125-inch probe rod (Fig. 4.26). Completely tighten the drive cap with a pipe wrench. **(Refer to notes in Step 6 regarding Drive Caps.)**

19. Lower the hydraulic hammer onto the drive cap and advance the outer casing one liner length into the subsurface to collect the first soil core. Apply hammer percussion to the tool string as this helps move soil through the cutting shoe and into the liner for increased sample recovery.

20. Raise the hydraulic hammer and retract the probe derrick to provide access to the top of the tool string.

21. Unthread the drive cap and remove the drive bumper.

(continued on following page)

22. Thread a 1.0-inch probe rod onto the inner rod (Fig. 4.27). Rotate the inner rods two or three revolutions to shear the soil core at the bottom of the liner. Raise the inner rods to retrieve the filled liner.

NOTE: Hold the inner rod string with a DT21 Rod Clamp Assembly (DT 4060) while unthreading the retracted rods (Fig. 4.28). Use the probe hammer and Rod Grip Pull System (GH3000K) to pull the inner rods if they are too heavy to lift comfortably by hand (Fig. 4.29).

23. Remove the filled liner from the liner drive head as described in Section 4.6.

(Repeat Steps 13-23 to collect consecutive soil core samples.)

Figure 4.27. Thread a 1.0-inch probe rod onto inner rod string to retrieve filled liner.

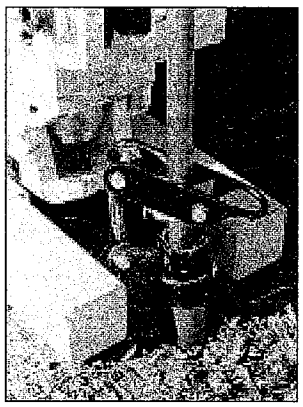

Figure 4.28. The DT21 Rod Clamp Assembly holds the inner rod string while adding or removing 1.0-inch rods.

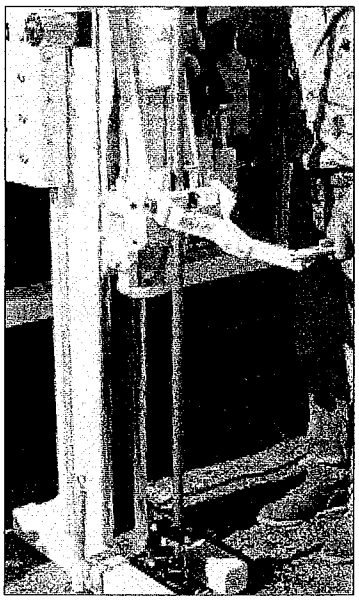

Figure 4.29. Raise the inner rod string with the Rod Grip Pull System if rods are too heavy to lift by hand.

4.6 Removing the DT21 Liner Drive Head from a Filled DT21 Liner

The liner drive head assembly remains attached to the filled liner after retrieval from the outer casing (Fig. 4.30). In order to decontaminate the drive head for further sampling, it must first be removed from the filled liner. This process is easily accomplished using a machine vise and sharp utility knife.

Place the liner drive head in the machine vise such that the 3/8-inch cap screw threaded through the liner is positioned on top. Remove the cap screw with a 3/32-inch hex key.

Using a utility knife, score a line from the top of the liner to the bottom of the drive head (Fig. 4.31). Move the free end of the liner side-to-side until the top of the liner splits and releases from the drive head (Fig. 4.32). The soil core may now be prepared for storage or analysis according to project guidelines.

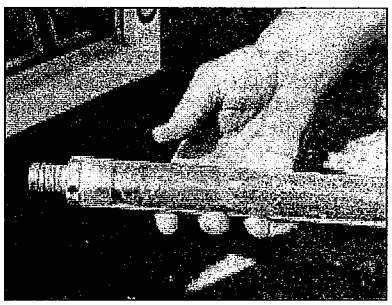

Figure 4.30. Liner drive head remains attached to filled liner after retrieval from outer casing.

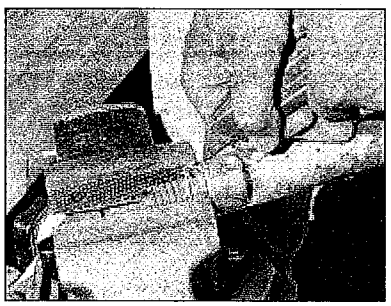

Figure 4.31. Score a line from top of liner to base of liner drive head using a utility knife.

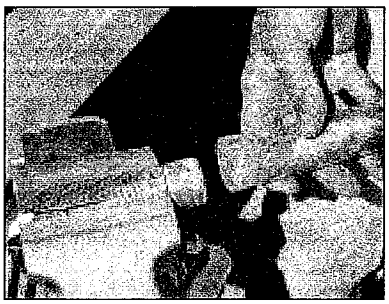

Figure 4.32. Move free end of liner back-and-forth to split liner and free it from the liner drive head.

4.7 Removing a Section of Liner with a DT21 Liner Grooving Tool and Utility Knife

A special tool is needed to help efficiently and safely cut through the thick DT21 liners in order to access the soil sample. The DT21 Liner Grooving Tool (P/N 18170, Fig. 4.33) greatly improves the liner cutting process by creating a deep longitudinal groove in the sample liner (Fig. 4.34). The sample liner is then easily cut along the groove with a normal utility knife.

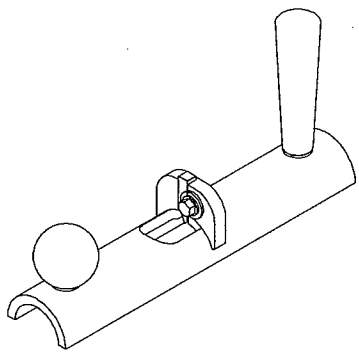

FIGURE 4.33
DT21 Liner Grooving Tool

Figure 4.34. Groove in liner created by DT21 Grooving Tool.

Grooving the Liner

Because DT21 liners are flexible and will bend under load, a means of holding the liner is absolutely necessary in order to successfully operate the grooving tool. An MC Liner Holder works well for this application. Liners up to the 1-meter length will fit in an AT8020 holder while the 15679 holder will accept all DT21 liner lengths.

1. Secure the liner holder in a machine vise or place it on a sturdy table or platform.

2. Lay the filled liner in the holder with the top (drive head) end of the liner toward the "hook" stop and the downhole end toward the "pin" stop (Fig. 4.35).

3. The grooving tool is pulled along the top of the liner to cut a single longitudinal groove. Right-handed operators will generally pull the tool beginning at the left end of the liner while left-handed operators will generally pull the tool beginning at the right end.

   Place the grooving tool on top of the liner with the cutting insert positioned approximately 0.25 inches (6 mm) in from the appropriate end as determined by the operator's dominant hand (Fig. 4.36).

4. Pull the grooving tool across the entire length of the liner by applying steady force to the front (spherical) handle. **For the grooving tool to function correctly, it is very important to put the majority of the pulling force on the front handle.** Only use the back (tapered) handle to guide the tool. When used correctly, the grooving tool will remove material in a continuous string as shown in Figure 4.37.

5. Repeat Steps 3 and 4 to cut a second groove that will allow removal of a section of liner for easier access to the soil sample.

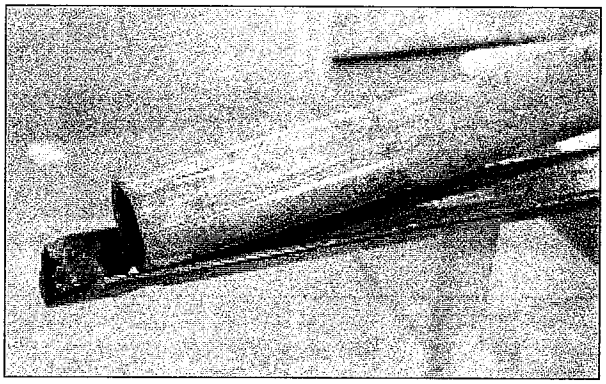

Figure 4.35. Lay the liner in the holder tray.

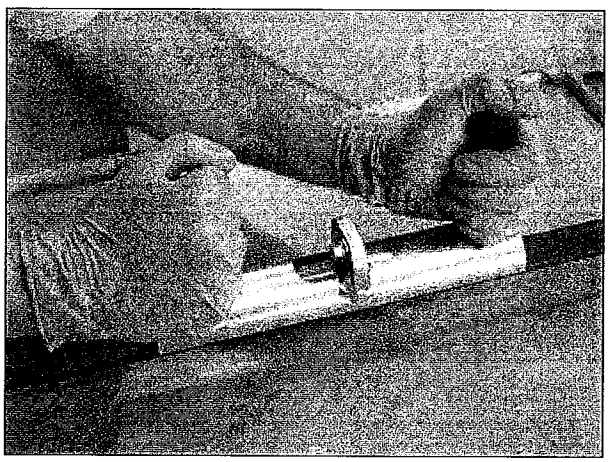

Figure 4.36. Place the grooving tool on top of the liner with the cutting insert positioned approximately 0.25 inches (6 mm) in from the end.

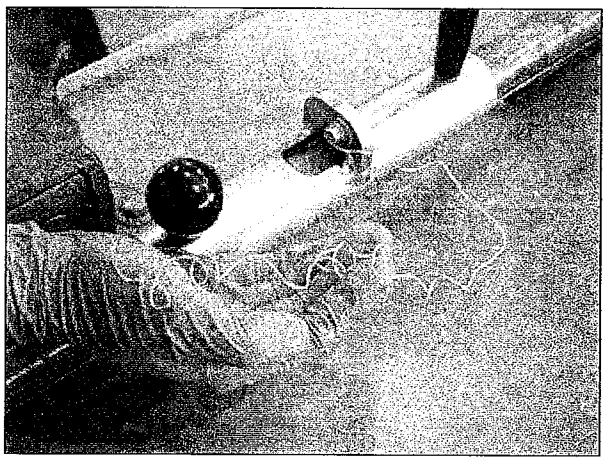

Figure 4.37. A thin ribbon of material will be cut as the tool is drawn down the length of the liner.

Cutting the Liner Open

Once the liner is grooved with the DT21 Grooving Tool, the groove is cut using a utility knife. **Make sure no one is standing near you before starting this procedure!**

1. Holding the utility knife perpendicular to the liner, place the blade in the groove to be cut (Fig. 4.38).

2. Push the blade into the groove by pushing straight down on the knife. Push down until the blade is completely through the liner wall.

3. With the knife blade still in the liner, rotate the knife approximately 90° until the blade is almost tangent to the liner (Fig. 4.39).

4. Grasp the knife such that it is held with the palm of the hand facing up as shown in Figure 4.39.

5. Place your other hand on the liner for support (Fig. 4.39). Make sure the knife blade faces away from this hand to avoid injury should the knife slip out of the liner while cutting.

6. Stand close to the liner holder to keep the hand with the utility knife close to your body for maximum control. Carefully pull the knife completely down the groove in the liner. Move your entire body with the knife to help keep your arm tucked against your body.

7. Repeat Steps 1-6 for the second liner groove.

8. Once both grooves are cut, pull the liner section away to access the soil sample (Fig. 4.40).

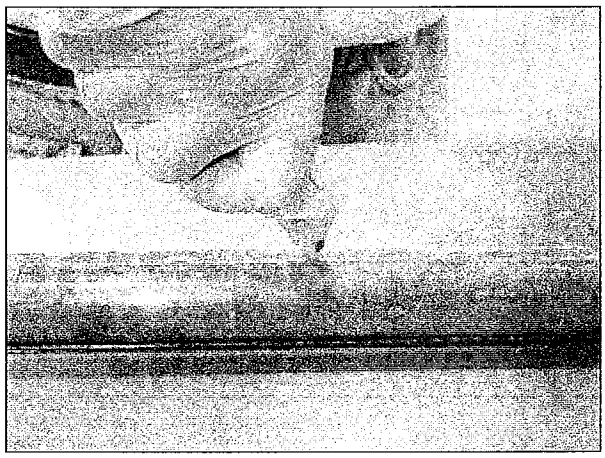

Figure 4.38. Insert the knife blade straight down into the groove.

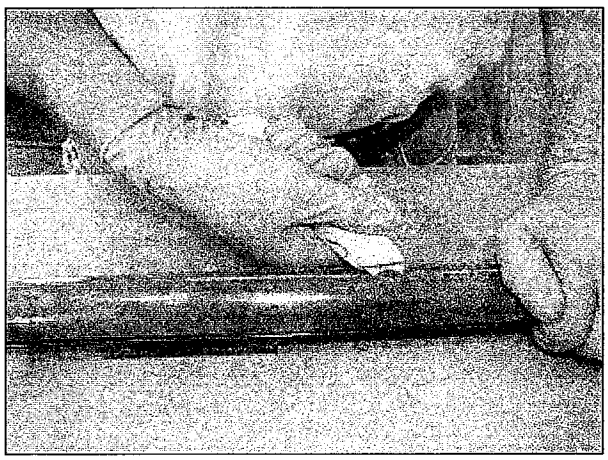

Figure 4.39. Rotate the knife until it is almost tangent to the liner. Gripping the knife palm up and keeping your arm close to your body for control, pull the knife along the groove.

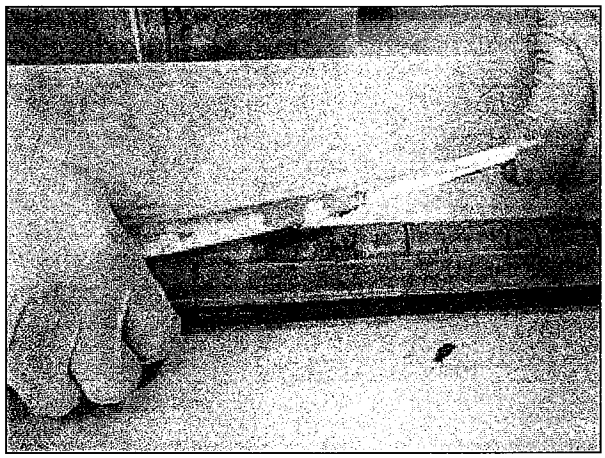

Figure 4.40. After cutting along the two grooves, peel away the liner section to access the soil sample.

4.8 Dual Tube Soil Sampling Tips

Saturated sands are the most difficult formations to sample with the DT21 system. Saturated conditions place positive pressure on the soil outside of the outer casing. When sampling in noncohesive formations (e.g. sands) below the water table, it may be necessary to add water to the outer casing to prevent formation heave. Adding water to the probe rods puts a positive head on the system and may keep formation material from flowing into the rods as the liner and soil sample are retracted. If a small amount of formation material is still drawn into the outer casing as the soil core is retrieved, the material may be displaced by slightly raising the outer casing while lowering the next new liner to depth. Water must be maintained within the outer casing during this process to overcome the hydraulic head imparted by the formation fluid.

When utilizing a solid drive tip in heaving sand formations, a ported probe rod (such as the 1.0-inch x 12.0-inch Groundwater Profiler Center Stem - PN: 12566) is suggested for use as the first rod above the drive tip of the inner rod string. A ported rod helps minimize the suction effect created when the inner rod string and solid drive tip are withdrawn from the water-filled outer casing prior to sampling. The ports allow water (added to the outer casing to control formation heave) to drain from the inner rods as they are withdrawn. The main benefit is a reduction in the amount of water removed with the inner rod string - water that offsets formation fluid pressure to minimize heaving. Another benefit is a cleaner work site as water is no longer captured within the rods and subsequently dumped as the inner rods are unthreaded at ground surface.

DT21 core catcher liners will provide the best sample recovery in saturated noncohesive formations. Additionally, 24-inch open (no core catcher) liners may be used to collect samples under these conditions. The shorter sample interval may enhance sample recovery in sandy materials. Collecting a shorter sample will also minimize the vacuum created as the sample is retracted inside the rod bore. This may help minimize the heave of noncohesive materials into the rods.

DT21 core catcher liners will help considerably with sample recovery in noncohesive soils and other materials that do not fill the liner diameter. Core catcher liners are not recommended for cohesive or expansive soils as the core catchers may actually inhibit soil movement into the liner.

Some clay materials will expand during sampling. Under these conditions, using a shorter sample interval (24-inch liners) may improve sample recovery by minimizing the wall friction as the material is sampled.

4.9 Outer Casing Retrieval

The outer casing of the DT21 Dual Tube System may be retrieved in one of three ways:

1. Casing pulled then probe hole sealed from ground surface with granular bentonite.

   The outer casing may be pulled from the ground with the probe machine and a Pull Cap (AT2104) or a Rod Grip Pull System (GH3000K) if the probe hole is to be sealed with granular bentonite from the ground surface (Fig. 4.41). This method is used for shallow probe holes in stable formations only. Such conditions allow the entire probe hole to be sealed with granular bentonite.

2. Casing pulled with probe hole sealed from bottom-up during retrieval.

   Bottom-up grouting should be performed during casing retrieval in unstable formations where side slough is probable. Such conditions create void spaces in the probe hole if granular bentonite is installed from the ground surface.

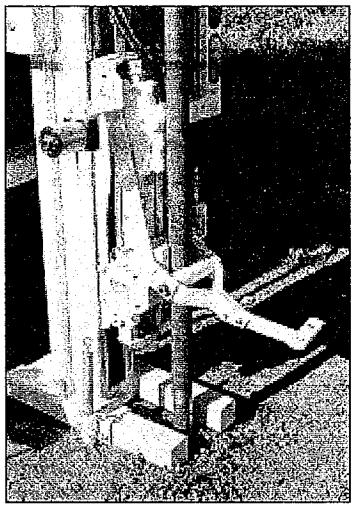

Figure 4.41 Outer casing may be retrieved with a pull cap or rod grip pull system if the probe hole is sealed with granular bentonite.

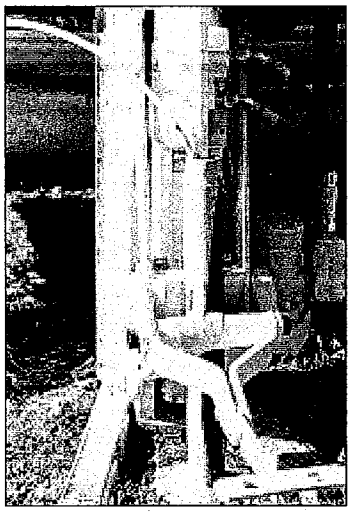

Figure 4.42. A grout machine and flexible tubing allow bottom-up grouting as the outer casing is retrieved.

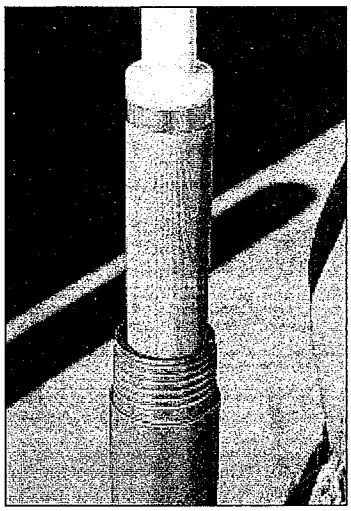

Figure 4.43. Geoprobe® prepacked screens may be installed through the outer casing when an expendable cutting shoe is used.

A GS500 or GS1000 Grout Machine is used to deliver a sealing material (high-solids bentonite slurry or neat cement grout) to the bottom of the outer casing through flexible tubing. The grout mix is pumped through the tubing to seal the void remaining as the outer casing is retrieved (Fig. 4.42). This is an advantage of the DT21 Dual Tube System as other soil samplers require a second set of tools to deliver grout to the bottom of the probe hole. Contact Geoprobe Systems® for more information on bottom-up grouting with the GS500 and GS1000 Grout Machines.

3. Casing pulled with Geoprobe Prepacked Screen Well installed during retrieval.

   The final option is to install a 1.4-inch OD Geoprobe® Prepacked Screen Monitoring Well in the probe hole during retrieval of the outer casing. A DT21 Expendable Cutting Shoe Holder (DT4040) and a DT21 Expendable Cutting Shoe (DT4045) allow the operator to collect continuous soil cores as the outer casing is driven to depth. When sampling is complete, the outer rods are raised and the expendable cutting shoe is removed from the leading rod. This leaves an open casing through which a set of prepacked screens is lowered on the leading end of a PVC riser string (Fig. 4.43). The well is finished, complete with grout barrier, bentonite well seal, and a high-solids bentonite slurry/neat cement grout, during retrieval of the outer casing.

   Refer to *Geoprobe® 0.5-in. x 1.4-in. OD and 0.75-in. x 1.4-in. OD Prepacked Screen Monitoring Wells Standard Operating Procedure* (Geoprobe® Technical Bulletin No. 962000) for specific information on well installation.

5.0 REFERENCES

Geoprobe Systems®, *Dual Tube (DT21) Groundwater Profiler Kit - Installation and Operation Instructions.* Instructional Bulletin No. 19275, 2001

Geoprobe Systems®, *Geoprobe® 0.5-in. x 1.4-in. OD and 0.75-in. x 1.4-in. OD Prepacked Screen Monitoring Wells - Standard Operating Procedure.* Technical Bulletin No. 962000, 2002.

Geoprobe Systems®, *Geoprobe Systems® Tools Catalog,* Vol. 6, 2003.

Equipment and tool specifications, including weights, dimensions, materials, and operating specifications included in this brochure are subject to change without notice. Where specifications are critical to your application, please consult Geoprobe® Systems.

Geoprobe Systems®

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WATER QUALITY PROGRAMS DIVISION

Standard Operating Procedure for the Installation of Nonrecording Gages and Measurement of Stage in Streams

Revised and Adopted October 2004

Draft Copy

OWRB

the water agency

OKLAHOMA WATER RESOURCES BOARD
WATER QUALITY PROGRAMS DIVISION
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STANDARD OPERATING PROCEDURE FOR THE INSTALLATION OF NONRECORDING GAGES AND MEASUREMENT OF STAGE IN STREAMS REVISED AND ADOPTED OCTOBER 2004

1.0 Introduction

Continuous measurements of stream stage are used in determining records of stream discharge. In stream gaging, gage heights are used as the independent variable in a stage discharge relation to derive discharges. The stage of a stream is the height of the water surface above an established datum plane while the gage height is a water-surface elevation referenced from an arbitrary point to the gage datum. Reliability of the discharge record is therefore dependent on the reliability of the gage height record as well as the stage-discharge relation. Gage height records may be obtained through various means. They may include a water-stage recorder, by systematic observation of a non-recording gage, or by noting only peak stages with a crest-stage gage. This SOP describes the installation and operation of Nonrecording gages commonly used in obtaining a record of stream stage.

2.0 Definitions/ Terms (UNDER CONSTRUCTION)

Datum plane
Stage height
Reference Mark
Reference point

3.0 Safety

Upon reaching the sampling location, site safety determinations should be made before proceeding. These will be different for wadeable and bridge sites. Please refer to the OWRB safety manual for instructions on how to sample both kinds of sites. When regulating the flow of traffic is necessary, please refer to the portion of the safety manual outlining "Traffic Safety Protocols".

4.0 Quality of the Measurement

When sampling for all programs, Quality Assurance/Quality Control (QA/QC) samples will be routinely collected to assure that environmental samples meet the Data Quality Objectives (DQO's) that are outlined in the controlling Quality Assurance Project Plan (QAPP). QA/QC sampling is designed to control each step of the sampling process. Blanks are collected to ensure that field personnel are properly cleaning the plastics and glassware used in field sampling. Duplicate samples are collected to ensure that composite samples are properly processed. Replicate samples may be collected to ensure that the sampling methodology employed is collecting a representative sample. Spike or known samples may be submitted to test the efficacy of the analytical laboratory.

For all measurements of stage, quality assurance is measured through several means. First of all, all surveys to set datums should be periodically checked. Secondly, replicate measurements should be taken from outside reference gages when available. Lastly, duplicate stage measurements should be made under extreme hydrological or meteorological conditions and periodically (1 in 10) for each measurement taken.

5.0 Personnel and Equipment

Principle investigators for the OWRB are required to have degrees and/or experience with biological or other applicable sciences. Principle investigators are defined as crew leaders, and this designation may be made upon the leader of a multi- or a one-person crew. Training is required for all SOPs dealing with water quality and quantity collections and measurements as well as habitat assessments and biological collections. In-house training will be conducted for the use of all meters and digital titrators used for water quality or quantity measurements. Investigators must be familiar with OWRB SOP document and all training will follow the methods outlined in that document. Extra training will be provided when new SOPs are developed. Training of field crews will be done through dry run exercises in the laboratory to familiarize field crews with sample collection, sample preservation, instrument operation, calibration, and maintenance. In addition, when new personnel are hired or new methods developed, qualified staff will train on sample collection, measurement, and field analysis methods through side-by-side field trips. These trips will familiarize staff with SOP requirements. When training is considered adequate, a qualified staff member will check field staff for adherence to SOPs.

In most instances, the collection of water quality samples requires only one field person. However, depending on the safety requirements of a particular station, additional crewmembers may be necessary to ensure a safe work zone. Equipment used to collect the chlorophyll-a sample are described in the document "Standard Operating Procedure for the Collection of Water Quality Samples".

6.0 Installation of Gages and Collection of Stage Data

6.1 Gage Datum

The gage datum is the level surface that is at the zero elevation of all the gages at a gaging station. This point can be tied to mean sea level or can be an arbitrary datum plane. Arbitrary datums are often established so that numbers are more manageable. A permanent datum must be maintained so that only one datum for the gage-height record is used for the life of the station. Though a benchmark is preferable, each gaging station requires at least two to four reference marks that are independent of the gage structure in order to maintain a permanent datum. The gage height is then established from the change in elevation between the gage datum and the gage. All gages are periodically checked under the procedures required and outlined by the Surveying Gaging Stations SOP. An individual Station Plan will outline in detail the frequency of surveys, the survey circuit, and the maintenance required to protect the gage datum.

6.2 Non Recording Gages

The types of non-recording gages generally used are staff, wire-weight, and manual tapedown. Staff gages are read directly whereas the other two types are read by measurement to the water surface from a fixed point. For the OWRB, nonrecording gages serve several purposes including:

1. A reference gage at nonrecording water quality stations
2. Useful when a recording gage is not practical or feasible
3. Outside reference gage at recording water quality stations
4. Inexpensive screening tool to determine best location for recording gages in watershed or aquifer specific studies.

6.2.1 Accuracy of Measurements at Nonrecording Gages

The level of measurement accuracy needed depends on the use of the data. In general, gages maintained to 0.01-0.05 foot have a high level of accuracy, and the stage-discharge rating that is developed may be used for all purposes. Accuracies maintained in a range of 0.05-0.20 foot may have limited utility but are still appropriate for certain purposes. Except in rare cases when data is used only as a screening tool, accuracy should never exceed 0.20 foot.

The most important parts of maintaining accuracy are attention to detail and consistency of effort. A long-term schedule for surveying should be made when the station is installed and adhered to over the period of record. Station characteristics should be noted when making discharge measurements. For example, comparison of inside and outside gage readings or comparison of flood marks with recorded peak gage heights should be made at every opportunity.

The management decisions outlined in workplans and individual Station Plans are the final guide on accuracy to be maintained. In addition, the OWRB's "Levels and Gaging Stations SOP" Section 6.3 makes general suggestions for the frequency of levels. However, rules of thumb concerning management decisions may be followed to assist in the determination of what level of accuracy is needed.

1. When minimal shifts in stage can dramatically influence management decisions, high levels of measurement accuracy are required. Examples include decisions made at the lower end of the rating curve (e.g., characterizing surface-ground water interactions), a small stream with relatively small medium to peak discharges, or a rating maintained to determine surface water yield.
2. When minimal shifts in stage will not dramatically influence management decisions, moderate levels of measurement accuracy are acceptable. For example, ratings used to assist in the calculation of surface water loadings because the great majority of constituent loading occurs during medium to peak discharges. Another example is when data are used to assist in trend analysis or comparison of a standard to a concentration.
3. If relative stage height is the only data need, a low level of accuracy may be sufficient. This is usually the case when using stage as a screening. However, it

should be kept in mind that the data gathered during the screen might be of limited utility once a station is put in place.

6.3 Manual Tapedown Gages

6.3.1 Accuracy and Use of Manual Tapedown Gages

Manual tapedown gages are the easiest and least expensive gages to install, measure, and maintain. The gage is placed directly in wing wall or a pier and usually consists of a chiseled mark or a pin. Accuracy and precision are normally low and are adversely effected by several things including: 1) difficult to accurately read and repeat reading of steel tape to a hundredth because of placement, 2) tape often not vertical due to winds and/or the lip of the bridge deck, and 3) susceptibility of marks and pins to movement and vandalism. By using a graduated engineer's tape, tapedowns can be accurate to 0.005 foot under perfect conditions. However, perfect conditions do not exist, and actual accuracy may be 0.01-0.10 foot. Therefore, they have limited utility. In certain instances (e.g., screening or starting a gage when limited funds are available), they can be used as the sole reference gage at a non-recording station. Tapedown stations are normally used in conjunction with another nonrecording gage as an index reference point. They should not be used as the outside reference gage for a recording station.

6.3.2 Installation and Maintenance of Manual Tapedown Gages

The chiseled arrow is simply a reference point that has been etched into the bridge wingwall or deck on the downstream side over the main channel. It is the most cost effective but least accurate method of stage measurement. The marks should be elevated if possible as well as labeled and painted. Tapedown should be free of any obstructions below the mark and in an area readily accessible by personnel. When placing marks in concrete, avoid scaling concrete. Because of the solid surface of angle or tubular iron, these often work best in steel bridges

Steel pins can also be installed as a reference point for tapedown gages. These are the more advanced cousin of the chiseled arrow because they have a more defined surface from which to measure and allow for a vertical measurement. However, they still yield low accuracy. Pins are installed by placing an anchor, bolt or nail into the bridge wingwall or pier on the downstream side over the main channel. They should be of sufficient length and set deeply enough to remain firmly fixed in place. Tapedown should be free of any obstructions below the pin and in an area readily accessible by personnel.

The marks should be consistently inspected for damage and, at a minimum, be surveyed annually to account for movement and loss of accuracy. If the gage is serving as a reference point for another type of nonrecording gage, it may be surveyed on the same schedule. Because they are inexpensive and easy to install, several should be installed at each station above all possible channels and labeled as primary (RP-1), secondary (RP-2), etc.

6.3.3 Measurement of Stage at Tapedown Gages

Stage is measured by taping down from the gage to surface of the water using a weighted, calibrated steel tape graduated in tenths and hundredths. The weight, known as a Louisiana ding-wop, is made of 2 inch tubular brass with a turned I-bolt sunk in the top and secured to the tape with plastic zip ties. The length of the dingwop can be changed by tightening the I-bolt and should be measured to one foot, or another known length. This length should be checked periodically using the steel tape. With the dingwop barely touching the surface of the water (look for slight ripples immediately around the weight or have a spotter), the steel tap is read at the pointed end of the chiseled arrow or the top of steel pin. After adding in the length of the ding-wop, stage is recorded to the nearest thousandth. If wind is affecting the measurement, attempt to measure stage between gusts, but if wind is constant, note the affect of wind in the discharge notes. Steel tapes are easily deformed and should be straightened before measurement.

6.4 Staff Gages

6.4.1 Accuracy and Use of Staff Gages

Vertical staff gages (Figure 1) are used as a stand-alone outside gage, an outside gage at a recording station, or a reference gage to another nonrecording device. Vertical gages are available in a variety of lengths, widths, and increments in both U.S. and metric scales. However, because of the necessary accuracy, comparability, and ruggedness, gages should be a Style A gage, which is made of porcelain enameled iron sections measuring 3 1/3' by 4" and graduated at every foot, tenth, and 0.02 foot. They are accurate only to 0.02 foot and can be damaged or lost due to high flows or ice. They have a tendency to drift if not consistently kept free of debris. Accuracies to 0.01 foot may be obtained if needed by reading the gage with a point gage.

Because of their uniqueness to each station, inclined gages are normally not available commercially for stream gaging. They are used in situations where placement of another type of outside gage is not possible. They can be used as stand-alone outside gages or as an outside gage at a recording station. Inclined gages have a low level of accuracy of up to 0.10 foot. Because of issues with installation, maintenance, and accuracy, inclined staff gages should be used only when other options are not feasible.

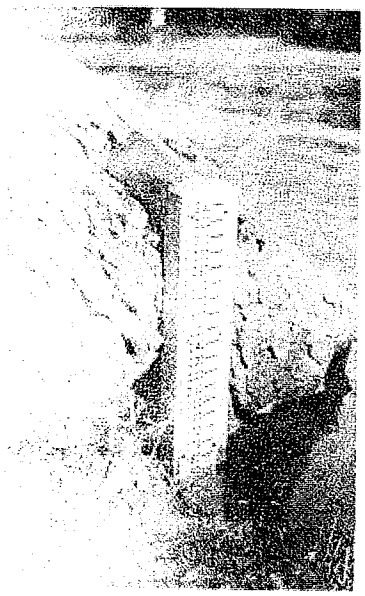


Figure 1. Vertical staff gage

6.4.2 Installation and Maintenance of Staff Gages

A vertical gage is mounted to permanently placed structure such as a piers or beam sunk to bedrock. If mounted in a stable location, a number of plates may be mounted one on top of the other. When mounting, find the high water bench and attempt to place the top of gage above that mark. Vertical height of gages should be kept to one plate and may require the installation of two or more to account for the vertical height of banks and potential high water level. When mounting to a pier, special equipment may be needed. The gage should be kept free of obstructions and be in an area readily accessible by personnel. A reference point should be placed on the backing of the gage plates and tied into the level circuit, and an RP reading should be noted by measuring against the steel plate using a steel tape. During subsequent surveys, new RP readings should be made and compared to the elevation for that RP. If these are different, a correction should be made to the gage datum. If a series of gages are used, each should be tied to the level circuit with a separate RP.

Inclined staff gages are usually made of a graduated heavy timber securely attached to a permanent foundation and are built flush with the stream bank The position of installation should be that the gage might be observed from a safe place during floods and high water conditions. To tie the gage into the level circuit, place an RP near the gage, and from the RP, make several side shots to footmarks throughout the range of the gage. Corrections should be made only if errors exceed 0.10 foot.

6.4.3 Measurement of Stage at Staff Gages

The measure is rather straightforward. The water level is read by a three-step method. As an example, 20.64 is used as a measurement. The footmark, 20.0, is noted. The next mark (6) is the inch mark. The next step is to locate the hundredth mark and count

the marks backward from the next highest inch mark. Using the example, the water level is at 0.64, and to determine the hundredth mark (0.04), the technician would count back from 0.7 to the water level. In this instance, the count was 0.06. By subtracting 0.06 from the inch mark, the hundredth mark (0.04) is obtained.

6.5 Wire Weight Gages

6.5.1 Accuracy and Use of Wire Weight Gages

The type A wire weight gage (Figure 2) consists of a drum wound with a single layer of cable, a bronze weight attached to the end of the cable, a graduated disc, and a Veeder counter, all within a cast-aluminum box. It is used as a stand-alone outside gage or an outside gage at a recording station. A full description of the gage is made in Section 4.4.3 of this document. The gage has a high level of accuracy ranging from 0.005 to 0.01 foot depending upon use. Several sources of error are associated with the use of the gage. The elevation of the gage may vary considerably due to placement along a bridge span. As top surface temperatures rise above bottom surface temperatures (hotter months), the bridge will arch upward and vice-versa when bottom surface temperatures are higher (cooler months). To avoid this potential source of error, every attempt should be made to place the gage near the end of a long span. Also, variations in the drum or cable diameter may lead to errors of up to 0.10 foot over 60 feet of spooled out cable.

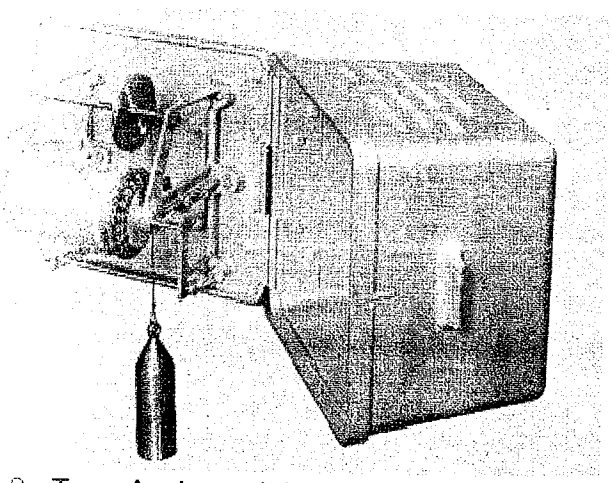


Figure 2. Type A wire weigh gage

6.5.2 Installation and Maintenance of Wire Weight Gages

A wire weight gage should be placed on the bridge wing wall directly above the deepest portion of the main channel. However, the area should be free of disturbance from piers, obstructions, and bridge supports so that the wire weight may be lowered freely without interference from the bridge and associated objects. Debris piles around piers can drastically reduce the accuracy of the measurement. Therefore, moving the weight away from the immediate vicinity of a pier is preferable. To ensure the gage can rotate freely to the water's surface, tapedown with a steel tape at an appropriate horizontal distance from the wing wall. The gage can be affixed to the wing wall through a variety

of means. However, the horizontal check bar must be parallel to the water surface and level, and metal shims, brackets, and/or washers may be required.

Levels should be run at several points when installing a wire weight gage. At a minimum, the wire weight should be leveled at the check bar in its outer position, and the gage datum should be leveled to the stream bottom. This requires two level circuits, but both begin at the same starting point allowing the wire weight to be tied to the gage datum. When possible, a reference gage (see tapedown measurements) should be established beside the wire weight. By tying the two marks together, changes in gage height may be more easily detected during normal measurements. An electric tape gage can also be mounted as the reference gage. Furthermore, if a gage is mounted near the center of long bridge span, placing reference points on both ends of the span may assist in determining the change of the gage datum that can be attributed to arching and sagging of the bridge. When possible, a reference point should also be established near the gage datum in a bridge pier or other permanent structure.

So that the gage height may be read, the dial is set to zero at the gage datum, and as the reel is brought back to the bridge, the gage height will gradually increase until it reaches the check bar reading. For streams with unstable substrates, the gage datum is often set above zero to allow for scour. When surveying, the gage height is calibrated and adjusted if needed. Follow these steps (an example in Table 1):

1. Survey the elevation of the check bar and the reference gage.
2. Tapedown to the water surface from the reference gage and correct gage height if necessary. A spotter should always be used and should not be done during windy conditions.
3. Read the check bar to the nearest thousandth.
4. Using a steel tape, tape to the water's surface from the check bar.
5. Using a steel tape, tape to the water's surface from the reference point.
6. Survey the elevation of the water surface.
7. Take a gage reading.
8. Adjust the check bar reading up or down so that the gage reading and the water surface elevation are equivalent to at least the hundredths.

The wire weight gauge should kept closed from the elements. The cable assembly should be periodically checked for kinks, twists and fraying. The drum assembly should be periodically lubricated to ensure smooth operation.

6.5.3 Measurement of Stage at Wire Weight Gages

A wire weight gage is simply a cabled drum attached to a counter. The diameter of the drum of the reel is such that each complete turn represents a 1-foot movement of the weight. The disc attached to the drum is graduated in tenths and hundredths of a foot and is permanently connected to the counter and to the shaft of the drum. The reel is equipped with a pawl and ratchet for holding the weight at any desired elevation. A horizontal checking bar is mounted at the lower edge of the instrument so that when it is moved to the forward position the bottom of the weight will rest on it.

A wire weight gage is rather simple to use. Before lowering the weight, ensure that the cable is fed through the wheel guide above the drum. The check bar located beneath the weight should be moved to the rear of the bracket. On the side of the drum, there is pawl with toothed gear. Holding the drum handle, ease the pawl away from the gear. The weight is now free to be lowered slowly to the water's surface. **DO NOT FREEWHEEL THE DRUM**. Upon contact with the water, the measurement can be made by looking at two counters. The Veeder counter, located on top of the instrument, represents the footmark. The tenths and hundredths footmarks are located on the disc attached to the drum. The arrow or pointer going from the drum to the disc represents the tenth mark. The hundredth mark is read going down from the tenth mark. Rewind the drum to the top and place the weight on the forward placed check bar. Lock the weight in place by locking the pawl. **LOCK THE COVER WHEN MEASUREMENT IS COMPLETED**.

6.6 Crest Gages

The crest-stage gage is a device for obtaining the elevation of the flood crest of streams. The gage is receiving widespread use because it is simple, economical, reliable, and easily installed. The one found most satisfactory is a vertical piece of " 2-inch galvanized pipe containing a wood or aluminum staff held in a fixed position with relation to a datum reference. Crest gages should be cleaned and reset after periodic floods. The crest gage should also be inspected for damage and level errors.

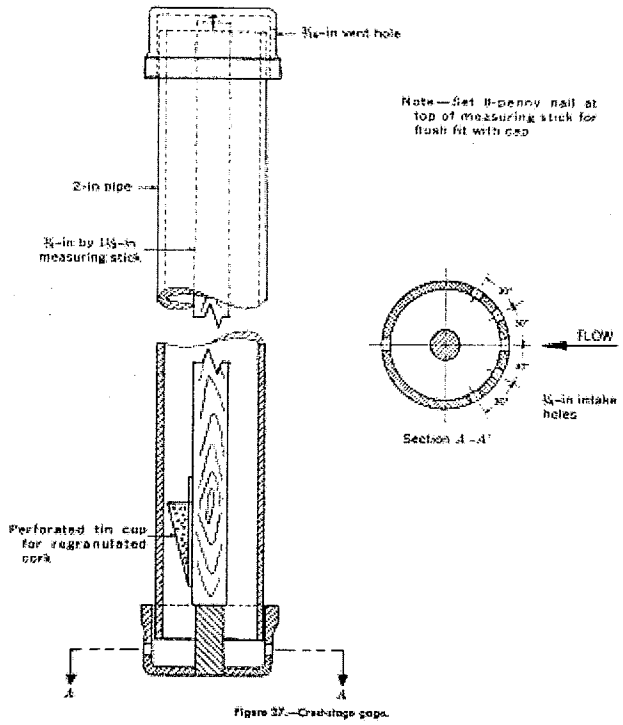


Figure 3. Crest gage

The bottom cap has six intake holes located so as to keep the non-hydrostatic draw down or super elevation inside the pipe to a minimum. Tests have shown this arrangement of intake holes to be effective with velocities up to 10 feet per second, and at angles up to 30 degrees with the direction of flow. The top cap contains - one small vent hole. The bottom cap or a perforated tin cup or copper screening in cup shape attached to the lower end of the staff contains regranulated cork. As the water rises inside the pipe the cork floats on its surface. When the water reaches its peak and starts to recede the cork adheres to the staff inside the pipe, thereby retaining the crest stage of the flood. The gage height of a peak is obtained by measuring the interval on the staff between the reference point and the flood mark. Scaling can be simplified by graduating the staff.

A Crest gage should placed in location that allows personnel to readily access the gage and reset the cork. Locations usually are along the bridge abutments, downstream in a protected location, or suspended from the bridge. If suspending from a bridge, the scale inside should be built to allow the scale to be removed and reset

7.0 Forms

7.1 Field Notes

Field notes are documents used to annotate and record information that is gathered at the project site. They are a data sheet and should be treated as such. Therefore, they should be written, legible, and complete. To avoid confusion and loss of data, a new sheet should be used at each new project site. Field notes should be initialed and dated by the collecting personnel and data entry personnel. For guidance on proper procedure to complete the field notes, refer to your supervisor and or FTE. Field notes can be found at S:\Monitoring\STREAMS\forms\Field Notes.doc.

8.0 Data Storage

All completed paper copies of forms and data sheets should be maintained with the appropriate station notebook. The data from the field notes and laboratory data sheets should be either entered into or uploaded to the Water Quality Database or other approved electronic format. Each sample should be maintained electronically in the database under a unique sample number.

9.0 References

Stage measurement at gaging stations, by T.J. Buchanan and W.P. Somers: USGS–TWRI Book 3, Chapter A7. 1968.

Measurement and computation of streamflow Volume 1. Measurement of stage and discharge Volume 2. Computation of discharge, By S. E. Rantz, et al.: USGS—Water Supply Paper 2175.

APPENDIX E

FIELD FORMS

Tetra Tech NUS, Inc.

BORING LOG

Page ___ of ___

PROJECT NAME: _____ BORING No.: _____
PROJECT NUMBER: _____ DATE: _____
DRILLING COMPANY: _____ GEOLOGIST: _____
DRILLING RIG: _____ DRILLER: _____

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: Soil Density/ Consistency or Rock Hardness	Color	Material Classification	USCS *	Remarks	PID/FID Reading (ppm): Sample	Sampler BZ	Borehole**	Driller BZ**

* When rock coring, enter rock brokeness.

** Include monitor reading in 6 foot intervals @ borehole. Increase reading frequency if elevated reponse read.

Drilling Area Background (ppm): ____

Remarks: _____

Converted to Well: Yes _____ No _____ Well I.D. #: _____

Tetra Tech NUS, Inc.

DAILY ACTIVITIES RECORD

PROJECT NAME: _____ **PROJECT NUMBER:** _____

CLIENT: _____ **LOCATION:** _____

DATE: _____ **ARRIVAL TIME:** _____

Tt NUS PERSONNEL: _____ **DEPARTURE TIME:** _____

CONTRACTOR: _____ **DRILLER:** _____

ITEM	QUANTITY ESTIMATE	QUANTITY TODAY	PREVIOUS TOTAL QUANTITY	CUMULATIVE QUANTITY TO DATE

COMMENTS: _____

APPROVED BY:

_____ _____

Tt NUS REPRESENTATIVE DRILLER

DATE: _____

Tetra Tech NUS, Inc.

PROJECT: _____ JOB #: _____
LOCATION: _____ DATE: _____
PROJECT MANAGER: _____ FOL: _____

DAILY ACTIVITIES CHECKLIST

Startup Checklist

Activity	Yes	No	N/A
Pertinent site activities/information entered into site logbook			
All onsite personnel listed in logbook			
Required medical information onsite for all workers (TtNUS and Subcontractors)			
Required MSDS's onsite			
Proper equipment calibrations performed (list equipment)			
1			
2			
3			
4			
Calibration logs filled out			
Tailgate H&S meeting held prior to beginning field activities			
Required work permits filled out/signed			
Required utility clearances obtained			
Required PPE onsite and in use			
Information required to be posted is in place			
(OSHA poster, hospital route, key phone numbers, etc.)			

Exit Checklist

Activity	Yes	No	N/A
Logbooks completely and comprehensively filled out			
Field forms complete and accounted for/properly filed			
Samples properly packaged/shipped			
COCs faxed to appropriate in-house personnel			
All equipment accounted for, on charge if needed, and properly secured			
All personnel accounted for			
Arrangements made for upcoming work (permits, clearances, equipment, etc.)			
Site properly secured			

Note - not all items listed apply to every job, and some additional requirements may apply on a job-specific basis.

Tetra Tech NUS, Inc.

EQUIPMENT CALIBRATION LOG

PROJECT NAME : _____ INSTRUMENT NAME/MODEL: _____

SITE NAME: _____ MANUFACTURER: _____

PROJECT No.: _____ SERIAL NUMBER: _____

Date of Calibration	Instrument I.D. Number	Person Performing Calibration	Instrument Settings		Instrument Readings		Calibration Standard (Lot No.)	Remarks and Comments
			Pre-calibration	Post-calibration	Pre-calibration	Post-calibration		

FIELD ANALYTICAL LOG SHEET
GEOCHEMICAL PARAMETERS

Tetra Tech NUS, Inc.

Project Site Name: _____ Sample ID No.: _____

Project No.: _____ Sample Location: _____

Sampled By: _____ Duplicate: ☐

Field Analyst: _____ Blank: ☐

Field Form Checked as per QA/QC Checklist (initials): _____

SAMPLING DATA:

Date: / Time:	Color (Visual)	pH (S.U.)	S.C. (mS/cm)	Temp. (^{0}C)	Turbidity (NTU)	DO (mg/l)	Salinity (%)	Other
Method:								

SAMPLE COLLECTION/ANALYSIS INFORMATION:

ORP (Eh) (+/- mv):_____ Electrode Make & Model:_____

Reference Electrode (circle one): Silver-Silver Chloride / Calomel / Hydrogen

Dissolved Oxygen:

Equipment: HACH Digital Titrator OX-DT CHEMetrics (Range: _____mg/L) Analysis Time: _____

Range Used:	Range	Sample Vol.	Cartridge	Multiplier
☐	1-5 mg/L	200 ml	0.200 N	0.01
☐	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

CHEMetrics: _____mg/L

Notes:

Alkalinity:

Analysis Time: _____

Equipment: HACH Digital Titrator AL-DT CHEMetrics (Range: _____mg/L) Filtered: ☐

Range Used:	Range	Sample Vol.	Cartridge	Multiplier	Titration Count	Multiplier		Concentration
☐	10-40 mg/L	100 ml	0.1600 N	0.1	_____ & _____	x 0.1	=	mg/L
☐	40-160 mg/L	25 ml	0.1600 N	0.4	_____ & _____	x 0.4	=	mg/L
☐	100-400 mg/L	100 ml	1.600 N	1.0	_____ & _____	x 1.0	=	mg/L
☐	200-800 mg/L	50 ml	1.600 N	2.0	_____ & _____	x 2.0	=	mg/L
☐	500-2000 mg/L	20 ml	1.600 N	5.0	_____ & _____	x 5.0	=	mg/L
☐	1000-4000 mg/L	10 ml	1.600 N	10.0	_____ & _____	x 10.0	=	mg/L

Parameter:	Hydroxide	Carbonate	Bicarbonate
Relationship:			

CHEMetrics: _____mg/L

Notes:

Standard Additions: ☐ Titrant Molarity:_____ Digits Required: 1st.:_____ 2nd.:_____ 3rd.:_____

Carbon Dioxide:

Equipment: HACH Digital Titrator CA-DT CHEMetrics (Range: _____mg/L) Analysis Time: _____

Range Used:	Range	Sample Vol.	Cartridge	Multiplier
☐	10-50 mg/L	200 ml	0.3636 N	0.1
☐	20-100 mg/L	100 ml	0.3636 N	0.2
☐	100-400 mg/L	200 ml	3.636 N	1.0
☐	200-1000 mg/L	100 ml	3.636 N	2.0

Titration Count			Concentration
_____	x 0.1	=	mg/L
_____	x 0.2	=	mg/L
_____	x 1.0	=	mg/L
_____	x 2.0	=	mg/L

CHEMetrics: _____mg/L

Notes:

Standard Additions: ☐ Titrant Molarity:_____ Digits Required: 1st.:_____ 2nd.:_____ 3rd.:_____

Tetra Tech NUS, Inc.

FIELD ANALYTICAL LOG SHEET
GEOCHEMICAL PARAMETERS

Project Site Name: _____ Sample ID No.: _____

Project No.: _____ Sample Location: _____

Sampled By: _____ Duplicate: ☐

Field Analyst: _____ Blank: ☐

Field Form Checked as per QA/QC Checklist (initials): ☐

SAMPLE COLLECTION/ANALYSIS INFORMATION:

Sulfide (S^{2-}):

Equipment: DR-700 DR-8 _ _ HS-WR Color Wheel Other: _____ Analysis Time: _____

Program/Module: 610nm 93

Concentration: _____ mg/L Filtered: ☐

Notes: _____

Sulfate ($S0_4^{2-}$):

Equipment: DR-700 DR-8 _ _ Other: _____ Analysis Time: _____

Program/Module: 91

Concentration: _____ mg/L Filtered: ☐

Standard Solution: ☐ Results: _____

Standard Additions: ☐ Digits Required: 0.1ml:_____ 0.2ml:_____ 0.3ml:_____

Notes: _____

Nitrite (NO_2^--N):

Analysis Time: _____

Equipment: DR-700 DR-8 _ _ Other: _____ Filtered: ☐

Program/Module: 60

Concentration: _____ mg/L Reagent Blank Correction: ☐

Standard Solution: ☐ Results: ☐

Notes: _____

Nitrate (NO_3^--N):

Analysis Time: _____

Equipment: DR-700 DR-8 _ _ Other: _____ Filtered: ☐

Program/Module: 55

Concentration: _____ mg/L

Nitrite Interference Treatment: ☐

Standard Solution: ☐ Results: _____ Reagent Blank Correction: ☐

Standard Additions: ☐ Digits Required: 0.1ml:_____ 0.2ml:_____ 0.3ml:_____

Notes: _____

Tetra Tech NUS, Inc.

FIELD ANALYTICAL LOG SHEET
GEOCHEMICAL PARAMETERS

Project Site Name: _____ Sample ID No.: _____

Project No.: _____ Sample Location: _____

Sampled By: _____ Duplicate: ☐

Field Analyst: _____ Blank: ☐

Field Form Checked as per QA/QC Checklist (initials): ☐

SAMPLE COLLECTION/ANALYSIS INFORMATION:

Manganese (Mn^{2+}):

Equipment: DR-700 DR-8 _ _ HACH MN-5 Other: _____ Analysis Time: _____

Program/Module: 525nm 41

Concentration: _____ mg/L Filtered: ☐

Digestion: ☐

Standard Solution: ☐ Results: _____ Reagent Blank Correction: ☐

Standard Additions: ☐ Digits Required: 0.1ml:_____ 0.2ml:_____ 0.3ml:_____

Notes: _____

Ferrous Iron (Fe^{2+}):

Equipment: DR-700 DR-8 _ _ IR-18C Color Wheel Other: _____ Analysis Time: _____

Program/Module: 500nm 33

Concentration: _____ mg/L Filtered: ☐

Notes: _____

Hydrogen Sulfide (H_2S):

Equipment: HS-C Other: _____ Analysis Time: _____

Concentration: _____ mg/L Exceeded 5.0 mg/L range on color chart: ☐

Notes: _____

QA/QC Checklist:

All data fields have been completed as necessary: ☐

Correct measurement units are cited in the SAMPLING DATA block: ☐

Values cited in the SAMPLING DATA block are consistent with the Groundwater Sample Log Sheet: ☐

Mulitplication is correct for each *Multiplier* table: ☐

Final calulated concentration is within the appropriate *Range Used* block: ☐

Alkalinity *Relationship* is determined appropriatly as per manufacturer (HACH) instructions: ☐

QA/QC sample (e.g., Std. Additions, etc.) frequency is appropriate as per the project planning documents: ☐

Nitrite Interference treatment was used for Nitrate test if Nitrite was detected: ☐

Title block on each page of form is initialized by person who performed this QA/QC Checklist: ☐

Tetra Tech NUS, Inc.

GROUNDWATER LEVEL MEASUREMENT SHEET

Project Name: _____ **Project No.:** _____

Location: _____ **Personnel:** _____

Weather Conditions: _____ **Measuring Device:** _____

Tidally Influenced: Yes ____ No ____ **Remarks:** _____

Well or Piezometer Number	Date	Time	Elevation of Reference Point (feet)*	Total Well Depth (feet)*	Water Level Indicator Reading (feet)*	Thickness of Free Product (feet)*	Groundwater Elevation (feet)*	Comments

* All measurements to the nearest 0.01 foot

Tetra Tech NUS, Inc.

GROUNDWATER SAMPLE LOG SHEET

Page___ of ___

Project Site Name: _____
Project No.: _____

[] Domestic Well Data
[] Monitoring Well Data
[] Other Well Type: _____
[] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____
Type of Sample:
[] Low Concentration
[] High Concentration

SAMPLING DATA:

Date:	Color	pH	S.C.	Temp.	Turbidity	DO	Salinity	Other
Time:	(Visual)	(S.U.)	(mS/cm)	(^{0}C)	(NTU)	(mg/l)	(%)	
Method:								

PURGE DATA:

Date:	Volume	pH	S.C.	Temp.	Turbidity	DO	Salinity	Other
Method:								
Monitor Reading (ppm):								
Well Casing Diameter & Material								
Type:								
Total Well Depth (TD):								
Static Water Level (WL):								
One Casing Volume(gal/L):								
Start Purge (hrs):								
End Purge (hrs):								
Total Purge Time (min):								
Total Vol. Purged (gal/L):								

SAMPLE COLLECTION INFORMATION:

Analysis	Preservative	Container Requirements	Collected

OBSERVATIONS / NOTES:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

Tetra Tech NUS, Inc.

HYDRAULIC CONDUCTIVITY TESTING DATA SHEET

PROJECT NAME: ..... WELL/BORING NO.: .....

PROJECT NO.: ..... GEOLOGIST: .....

WELL DIAMETER: ..... SCREEN LENGTH/DEPTH: ..... TEST NO.: .....

STATIC WATER LEVEL (Depth/Elevation): ..... DATE: .....

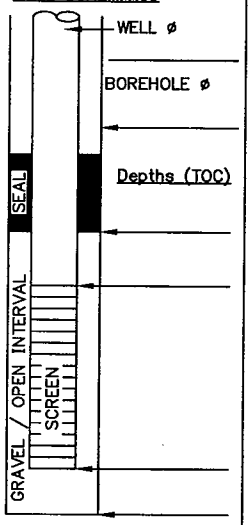
TEST TYPE (Rising/Falling/Constant Head): ..... CHECKED: .....

METHOD OF INDUCING WATER LEVEL CHANGE: ..... PAGE ..... OF .....

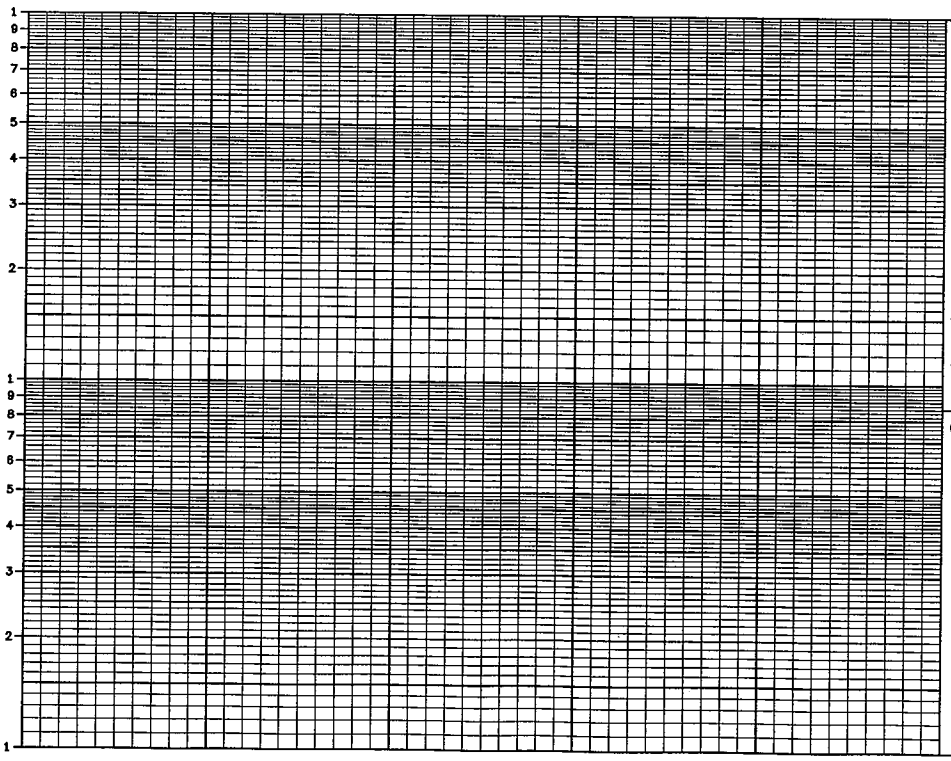
REFERENCE PT. FOR WL MEAS. (Top of Casing, Transducer, etc.): .....

ELAPSED TIME (min. or sec.)	MEASURED WATER LEVEL (feet)	DRAWDOWN OR HEAD (ΔH) (feet)	ELAPSED TIME (min. or sec.)	MEASURED WATER LEVEL (feet)	DRAWDOWN OR HEAD (ΔH) (feet)

WELL SCHEMATIC



▽ Indicate SWL Depth on Drawing



REMARKS:

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CALCS,SKETCH MAPS, ETC.:

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LOW FLOW PURGE DATA SHEET

PROJECT SITE NAME: _____

PROJECT NUMBER: _____

WELL ID.: _____

DATE: _____

Time (Hrs.)	Water Level (Ft. below TOC)	Flow (mL/Min.)	pH (S.U.)	S. Cond. (mS/cm)	Turb. (NTU)	DO (mg/L)	Temp. (Celcius)	ORP mV	Salinity % or ppt	Comments

SIGNATURE(S): _____

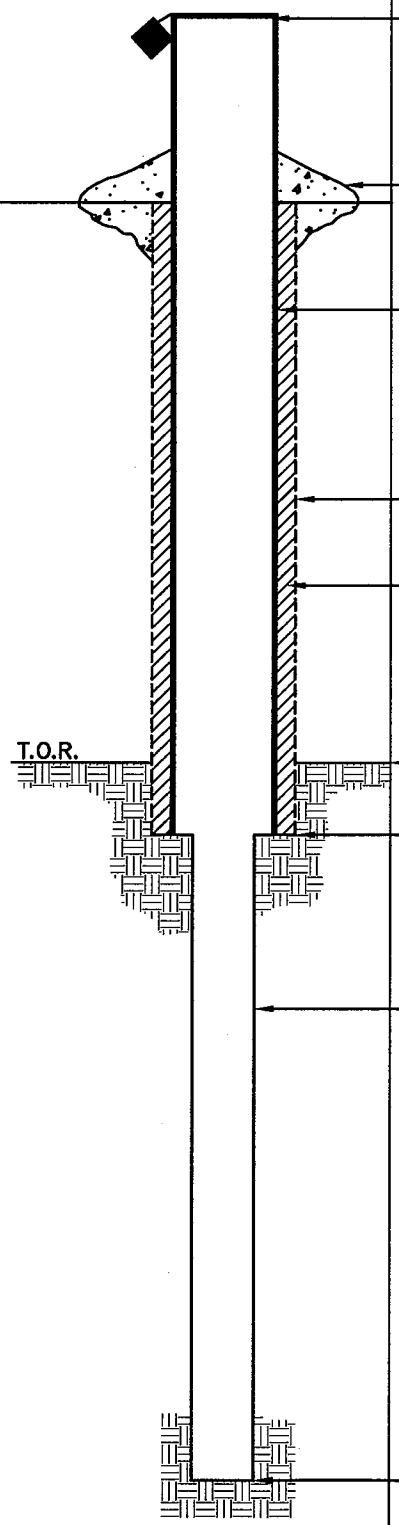
PAGE___OF___

WELL NO.: _____

Tetra Tech NUS, Inc.

BEDROCK
MONITORING WELL SHEET
OPEN HOLE WELL

PROJECT _____	LOCATION _____	DRILLER _____
PROJECT NO. _____	BORING _____	DRILLING METHOD _____
DATE BEGUN _____	DATE COMPLETED _____	
FIELD GEOLOGIST _____		DEVELOPMENT METHOD _____
GROUND ELEVATION _____	DATUM _____	



ELEVATION/HEIGHT OF TOP OF SURFACE CASING: ____/____

TYPE OF SURFACE SEAL: _____

I.D. OF CASING: _____

TYPE OF CASING: _____

TEMP./PERM.: _____

DIAMETER OF HOLE: _____

TYPE OF CASING SEAL: _____

DEPTH TO TOP OF ROCK: _____

DEPTH TO BOTTOM CASING: _____

DIAMETER OF HOLE IN BEDROCK: _____

DESCRIBE IF CORE/REAMED WITH BIT:

DESCRIBE JOINTS IN BEDROCK AND DEPTH:

ELEVATION/DEPTH OF HOLE: ____/____

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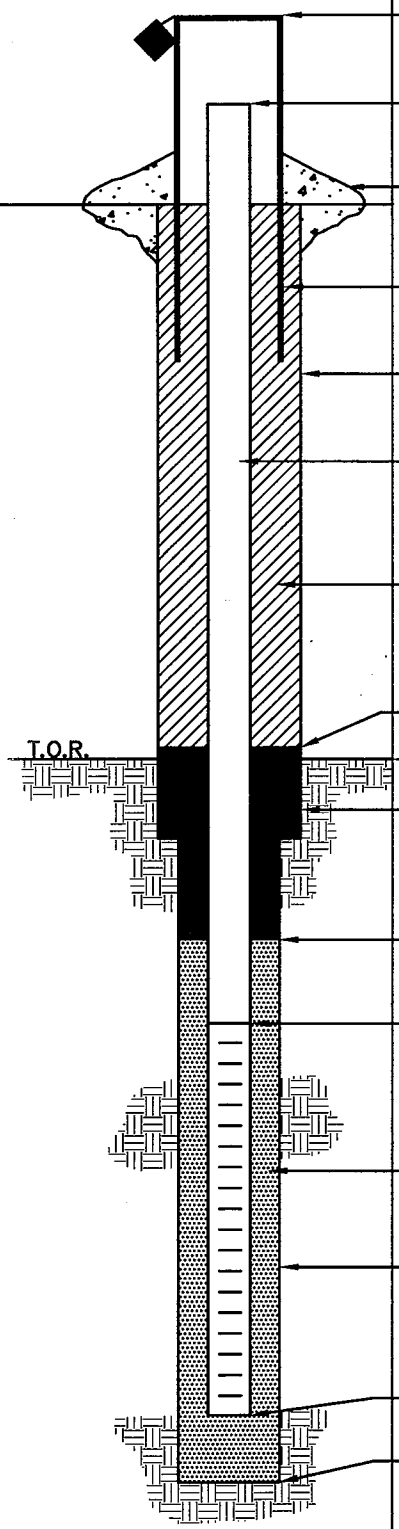
WELL NO.: _____

Tetra Tech NUS, Inc.

BEDROCK
MONITORING WELL SHEET
WELL INSTALLED IN BEDROCK

PROJECT _____	LOCATION _____	DRILLER _____
PROJECT NO. _____	BORING _____	DRILLING METHOD _____
DATE BEGUN _____	DATE COMPLETED _____	
FIELD GEOLOGIST _____		DEVELOPMENT METHOD _____
GROUND ELEVATION _____	DATUM _____	



ELEVATION/HEIGHT OF TOP OF SURFACE CASING: ____/____

ELEVATION/HEIGHT TOP OF RISER: ____/____

TYPE OF SURFACE SEAL: _____

I.D. OF SURFACE CASING: _____

DIAMETER OF HOLE: _____

RISER PIPE I.D.: _____
TYPE OF RISER PIPE: _____

TYPE OF BACKFILL: _____

ELEVATION/DEPTH TOP OF SEAL: ____/____

ELEVATION/DEPTH TOP OF BEDROCK: _____

TYPE OF SEAL: _____

ELEVATION/DEPTH TOP OF SAND: ____/____

ELEVATION/DEPTH TOP OF SCREEN: ____/____
TYPE OF SCREEN: _____
SLOT SIZE x LENGTH: _____
I.D. SCREEN: _____

TYPE OF SAND PACK: _____

DIAMETER OF HOLE IN BEDROCK: _____
CORE/REAM: _____

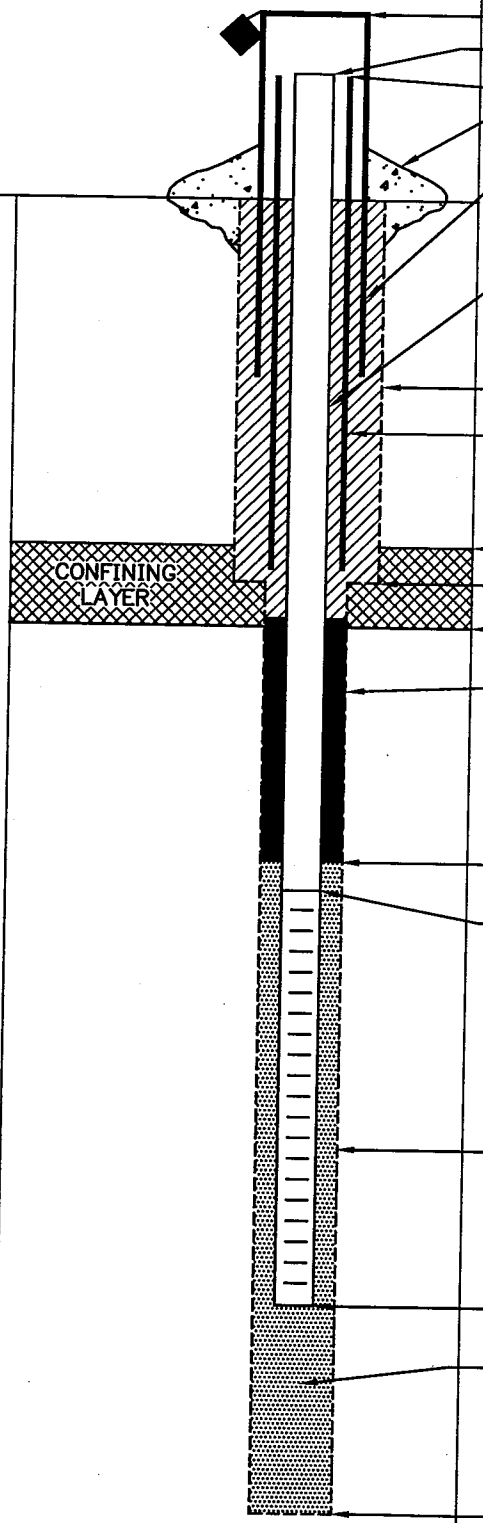
ELEVATION/DEPTH BOTTOM SCREEN: ____/____
ELEVATION/DEPTH BOTTOM OF SAND: ____/____
ELEVATION/DEPTH BOTTOM OF HOLE: ____/____
BACKFILL MATERIAL BELOW SAND: _____

WELL NO.: _____

Tetra Tech NUS, Inc.

CONFINING LAYER
MONITORING WELL SHEET

PROJECT _____	LOCATION _____	DRILLER _____
PROJECT NO. _____	BORING _____	DRILLING METHOD _____
DATE BEGUN _____	DATE COMPLETED _____	
FIELD GEOLOGIST _____		DEVELOPMENT METHOD _____
GROUND ELEVATION _____	DATUM _____	



ELEVATION/HEIGHT OF TOP OF SURFACE CASING: ____/____

ELEVATION/HEIGHT OF TOP OF RISER PIPE: ____/____

ELEVATION/HEIGHT OF TOP OF PERM. CASING: ____/____

TYPE OF SURFACE SEAL: _____

I.D. OF SURFACE CASING: _____

TYPE OF SURFACE CASING: _____

RISER PIPE I.D.: _____

TYPE OF RISER PIPE: _____

BOREHOLE DIAMETER: _____

PERM. CASING I.D.: _____

TYPE OF CASING AND BACKFILL: _____

ELEVATION/DEPTH TOP CONFINING LAYER: ____/____

ELEVATION/DEPTH BOTTOM OF CASING: ____/____

ELEVATION/DEPTH BOTTOM CONFINING LAYER: ____/____

ELEVATION/DEPTH TOP OF SEAL: ____/____

TYPE OF SEAL: _____

DEPTH TOP OF SAND PACK: _____

ELEVATION/DEPTH TOP OF SCREEN: ____/____

TYPE OF SCREEN: _____

TYPE OF SAND PACK: _____

BOREHOLE DIA. BELOW CASING: _____

ELEVATION/DEPTH BOTTOM OF SCREEN: ____/____

ELEVATION/DEPTH BOTTOM OF SAND PACK: ____/____

BACKFILL MATERIAL BELOW SAND: _____

ELEVATION/DEPTH OF HOLE: ____/____

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Tetra Tech NUS, Inc.

MONITORING WELL DEVELOPMENT RECORD

Site: _____ Depth to Bottom (ft.): _____ Project Name: _____
Well: _____ Static Water Level Before (ft.): _____ Project Number: _____
Date Installed: _____ Static Water Level After (ft.): _____ Site Geologist: _____
Date Developed: _____ Screen Length (ft.): _____ Drilling Co.: _____
Dev. Method: _____ Specific Capacity: _____
Pump Type: _____ Casing ID (in.): _____

Time	Estimated Sediment Thickness (Ft.)	Cumulative Water Volume (Gal.)	Water Level Readings (Ft. below TOC)	Temperature (Degrees C)	pH	Specific Conductance (Units _____)	Turbidity (NTU)	Remarks (odor, color, etc.)

MONITORING WELL MATERIALS
CERTIFICATE OF CONFORMANCE

Well Designation: _____

Site Name: _____

Date Installed: _____

Project Name: _____

Site Geologist: _____

Drilling Company: _____

Driller: _____

Project Number: _____

Material	Brand/Description	Source/Supplier	Sample Collected ?
Well Casing			
Well Screen			
End Cap			
Drilling Fluid			
Drilling Fluid Additives			
Backfill Material			
Annular Filter Pack			
Bentonite Seal			
Annular Grout			
Surface Cement			
Protective Casing			
Paint			
Rod Lubricant			
Compressor Oil			

To the best of my knowledge, I certify that the above described materials were used during installation of this monitoring well.

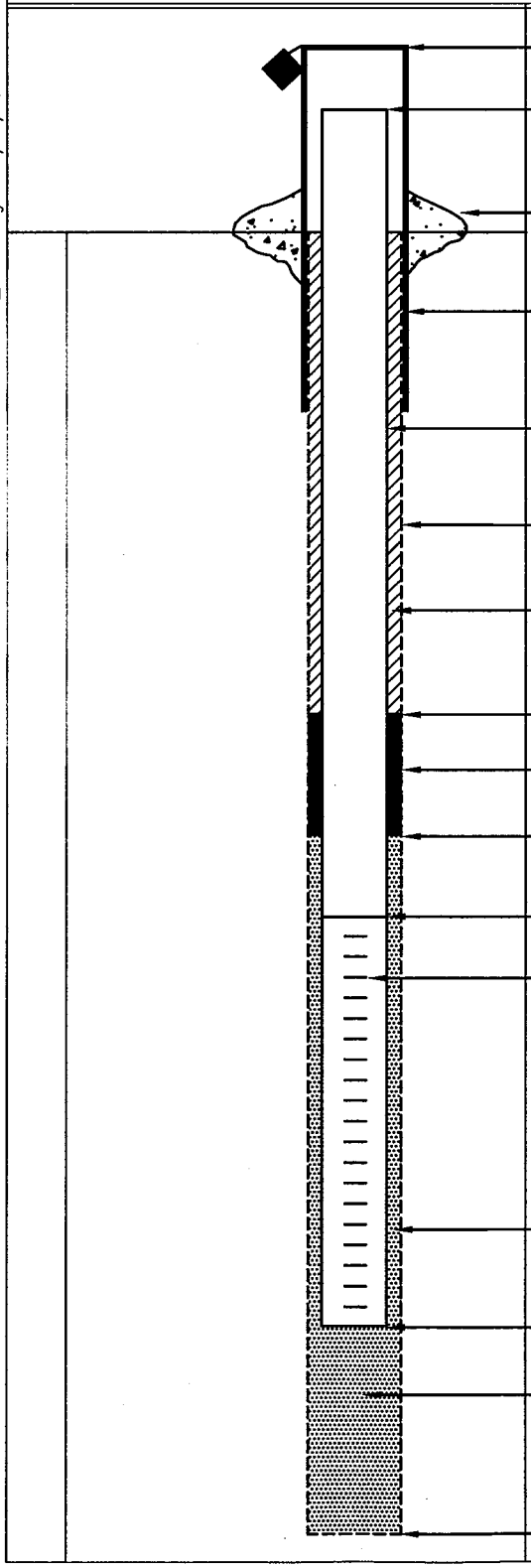
Signature of Site Geologist: _____

WELL NO.: _____

Tetra Tech NUS, Inc.

OVERBURDEN MONITORING WELL SHEET STICK-UP

PROJECT _____	LOCATION _____	DRILLER _____
PROJECT NO. _____	BORING _____	DRILLING METHOD _____
DATE BEGUN _____	DATE COMPLETED _____	
FIELD GEOLOGIST _____		DEVELOPMENT METHOD _____
GROUND ELEVATION _____	DATUM _____	



ELEVATION/HEIGHT OF TOP OF SURFACE CASING: ____/____

ELEVATION/HEIGHT OF TOP OF RISER PIPE: ____/____

TYPE OF SURFACE SEAL: _____

I.D. OF SURFACE CASING: _____
TYPE OF SURFACE CASING: _____

RISER PIPE I.D.: _____
TYPE OF RISER PIPE: _____

BOREHOLE DIAMETER: _____

TYPE OF BACKFILL: _____

ELEVATION/DEPTH TOP OF SEAL: ____/____

TYPE OF SEAL: _____

DEPTH TOP OF SAND PACK: _____

ELEVATION/DEPTH TOP OF SCREEN: ____/____

TYPE OF SCREEN: _____

SLOT SIZE x LENGTH: _____

I.D. OF SCREEN: _____

TYPE OF SAND PACK: _____

ELEVATION/DEPTH BOTTOM OF SCREEN: ____/____

ELEVATION/DEPTH BOTTOM OF SAND PACK: ____/____
BACKFILL MATERIAL BELOW SAND: _____

ELEVATION/DEPTH OF HOLE: ____/____

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Tetra Tech NUS, Inc.

PUMPING TEST DATA SHEET

PROJECT NAME: _____ PUMPING WELL NUMBER: _____

PROJECT NUMBER: _____ MEASURED WELL NUMBER: _____

[ ] PUMPING TEST [ ] STEP DRAW DOWN TEST

DATE(s): MONITORING POINT:

MEASUREMENT METHOD: DEPTH CORRECTION (ft):

TEST NUMBER: PUMP SETTING (Ft. below monitoring point):

STATIC WATER LEVEL (ft): DISTANCE FROM PUMPING WELL (ft):

PERSONNEL:

REMARKS:

MILITARY TIME	ELAPSED TIME SINCE PUMP START OR STOP (Min.)	WATER LEVEL (Ft.)	CORRECTION (Ft.)	DRAW DOWN OR RECOVERY (Ft.)	PUMPING RATE (GPM)	FLOW METER READING (Gals.)	REMARKS

NOTE: All measurements to nearest 0.01 foot measured from top of well riser pipe unless otherwise noted.

Tetra Tech NUS, Inc.

QA SAMPLE LOG SHEET

Page___ of ___

Project Site Name: _____ Sample ID Number: _____
Project Number: _____ Sampled By: _____
Sample Location: _____ C.O.C. Number: _____
QA Sample Type:

[] Trip Blank [] Rinsate Blank
[] Source Water Blank [] Other Blank _____

SAMPLING DATA:

Date: _____
Time: _____
Method: _____

WATER SOURCE:

[] Laboratory Prepared [] Tap
[] Purchased [] Fire Hydrant
[] Other _____

PURCHASED WATER INFORMATION (If Applicable as Source or Rinsate Water):

Product Name: _____
Supplier: _____
Manufacturer: _____
Order Number: _____
Lot Number: _____
Expiration Date: _____

RINSATE INFORMATION (If Applicable):

Media Type: _____
Equipment Used: _____
Equipment Type:

[] Dedicated
[] Reusable

SAMPLE COLLECTION INFORMATION:

Analysis	Preservative	Container Requirements	Collected
Volatiles	Cool 4°C & HCl		YES / NO
Semivolatiles	Cool 4°C		YES / NO
Pesticide / PCB	Cool 4°C		YES / NO
Metals	Cool 4°C & HNO_3		YES / NO
Cyanide	Cool 4°C & NaOH		YES / NO

OBSERVATIONS / NOTES:

Signature(s):

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SURFACE WATER SAMPLE LOG SHEET

Page___ of ___

Project Site Name: _____
Project No.: _____

[] Stream
[] Spring
[] Pond
[] Lake
[] Other: _____
[] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____

Type of Sample:
[] Low Concentration
[] High Concentration

SAMPLING DATA:

	Color (Visual)	pH (S.U.)	S.C. (mS/cm)	Temp. (^{0}C)	Turbidity (NTU)	DO (mg/l)	Salinity (%)	Other
Date:								
Time:								
Depth:								
Method:								

SAMPLE COLLECTION INFORMATION:

Analysis	Preservative	Container Requirements	Collected

OBSERVATIONS / NOTES:

MAP:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

TETRA TECH NUS
FIELD TASK MODIFICATION REQUEST FORM

Project/Installation Name _____ CTO & Project Number _____ Task Mod. Number _____

Modification To (e.g. Work Plan) _____ Site/Sample Location _____ Date _____

Activity Description: _____

Reason for Change: _____

Recommended Disposition: _____

Field Operations Leader (Signature) _____ Date _____

Approved Disposition:

Project/Task Order Manager (Signature) _____ Date _____

Distribution:

Program/Project File –
Project/Task Order Manager –
Field Operations Leader –

Other: _____

APPENDIX F

ANALYTICAL LABORATORY STANDARD OPERATING PROCEDURES
(provided on compact disk)

ANALYTICAL STANDARD OPERATING PROCEDURES
NORTHEAST LABORATORIES, INC
FOR SUPPLEMENTAL RI OF 1,4-DIOXANE IN THE EASTERN PLUME AND BEDROCK
NAS BRUNSWICK, MAINE

SOP 3.2.1	Metals Analysis by 6010B
SOP 3.2.3	Mercury in Liquid Waste EPA 7470A
SOP 3.2.5	Acid Digestion of Aqueous Sample & Extracts for Total metals for Analysis by FLAA or ICP Spectroscopy EPA 3010A
SOP 3.2.8	Toxicity Characteristic Leaching Procedure Method 1311
SOP 3.3.5	Determination of Chlorinated Acids in Water (8151A)
SOP 3.3.13	Volatile Organic Compounds (8260B)
SOP 3.3.14	Organochlorine Pesticides (8081)
SOP 3.3.15	Semivolatile Organic Compounds EPA 8270C
SOP 3.3.29	Separatory Funnel Liquid - Semivolatile Organics
SOP 3.3.30	Separatory Funnel Liquid-Liquid Extraction SW-846 Method 3510C – Pesticides and PCBs
SOP 3.3.50	1,4-Dioxane 8260
SOP 3.3.52	1,1-DCE by GC/MS – Modified EPA Method 8260B-SIM
SOP 3.2.83	TOC in Solid Matrices – EPA Method 9060 (Modified) and Lloyd Kahn (1988)
SOP 3.4.0	Procedure for Proper Sample Receipt and Log-In of Analytical Samples
SOP 3.7.11	Standard Operating Procedure for the Proper Disposal of Samples

Laboratory Quality Assurance Manual

REV:	K
PAGE:	1 of 23

TITLE:	Metals Analysis (Inductively Coupled Plasma-Atomic Emissions Spectrometry) EPA 6010B and 6010C
DEPARTMENTS AFFECTED:	Metals Chemistry Section

ISSUE DATE:	REVISION CODE:	SOP #:	REVISION CHANGES:
2/6/02	-	3.2.1	Update to NELAC form
5/29/02	A	3.2.1	Update SOP to comply with client audit
5/29/03	B	3.2.1	Client Project Plan
5/12/04	C	3.2.1	Add Peak Profilling & Dilution Procedure
3/31/05	D	3.2.1	Annual Review & Update
01/31/06	E	3.2.1	Annual Review & Update
09/25/06	F	3.2.1	Update to include Navy Assessment Findings
10/24/06	G	3.2.1	Update to include Additional Navy Assessment Findings
12/11/06	H	3.2.1	Update
12/14/06	I	3.2.1	Update to include Method 6010C
11/1/07	J	3.2.1	Addition of lead paint procedure and Annual Review and Update
08/01/08	K	3.2.1	Annual Update and Review

		DOCUMENT APPROVAL
Originator: Andrew McCaw	Signature/Date: Andrew McCaw 8.6.08	Title Inorganics Supervisor
Approved by: Jim Galasyn	J Galasyn 080608	Chemistry Lab Manager
Reviewed by: Pamela Doughty	Pamela Doughty 8/6/08	Total Quality Manager

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NEL	STANDARD OPERATING PROCEDURE		
Metals Analysis (Inductively Coupled Plasma-Atomic Emissions Spectrometry EPA 6010B)			
SOP# 3.2.1	REV# K	EFFECTIVE 08/01/08	PAGE 2 of 23
WRITTEN BY: Andrew McCaw	DEPARTMENTS AFFECTED: Metals Chemistry Section		

1.0 Test Method:

This standard operating procedure (SOP) is for EPA Method 6010B; metal analysis by inductively coupled plasma-atomic emissions spectroscopy.

2.0 Applicable Matrix or Matrices

All matrices, including ground water, aqueous samples, TCLP extracts, industrial and organic wastes, soils, sludges, sediments, wipes, paint chips and other solid wastes, that require digestion prior to analysis.

3.0 Detection Limit

Detection limits vary per element. See table 1.0. Annual Method Detection Limit (MDL) studies are performed and all data is stored in the QA Manager's office.

Table 1: Wavelengths, PQL's and 5x concentrations

Analyte	wavelength		PQL	5x concentration
Ag	328.068	mg/L	0.005	0.001
Ag	338.289	mg/L	0.005	0.001
Al	237.313	mg/L	0.02	
As	188.979	mg/L	0.01	0.005
B	182.528	mg/L	0.05	
B	249.772	mg/L	0.05	
Ba	233.527	mg/L	0.01	
Be	313.107	mg/L	0.005	0.002
Ca	317.933	mg/L	1.0	
Cd	228.802	mg/L	0.01	0.002
Co	230.786	mg/L	0.05	
Cr	267.716	mg/L	0.01	0.005
Cu	324.752	mg/L	0.01	0.003
Fe	238.204	mg/L	0.01	
K	766.490	mg/L	1.0	
Li	670.784	mg/L	0.020	

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Mg	280.271	mg/L	1.0	
Mg	279.077	mg/L	1.0	
Mn	257.610	mg/L	0.01	
Mo	202.031	mg/L	0.05	
Na	589.592	mg/L	1.0	
Na	330.237	mg/L	1.0	
Ni	231.604	mg/L	0.01	0.003
P	178.221	mg/L	0.02	
Pb	220.353	mg/L	0.01	0.003
Sb	217.582	mg/L	0.05	
Se	196.026	mg/L	0.05	0.01
Si	288.158	mg/L	0.01	
Sn	283.998	mg/L	0.01	
Sr	460.733	mg/L	0.01	
Ti	336.121	mg/L	0.01	
Tl	190.801	mg/L	0.05	
V	292.402	mg/L	0.05	
W	207.912	mg/L	0.01	
Zn	202.548	mg/L	0.01	

4.0 Scope and Application

4.1 Inductively coupled plasma-atomic emission spectroscopy (ICP-AES determines elements in solution. The method is applicable to all of the elements listed in Table 1.

4.2 Detection limits, sensitivity, and optimum range of the instrument will vary with the matrices and chosen emission line.

4.3 Users of the method should state the data quality objectives prior to analysis and must document and have on file the required initial performance data described in the following sections prior to using the method.

4.4 Use of this method is restricted to spectroscopists who are knowledgeable in the correction of spectral, chemical and physical interferences described in this method.

5.0 Summary of Method

5.1 Prior to analysis, samples must be solubilized or digested using appropriate Sample Preparation Methods (e.g. Methods 3010A/3050). When analyzing for dissolved constituents, acid digestion is not necessary if the samples are filtered and acid preserved prior to analysis.

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5.2 This SOP describes the simultaneous multi-elemental determination of elements by ICP. The method measures element-emitted light by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific atomic-line emission spectra are produced by a radio frequency inductively coupled plasma. Background correction is required for trace element determination. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background-intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interference and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result.

TABLE 2. RECOMMENDED WAVELENGTHS AND ESTIMATED INSTRUMENTAL DETECTION LIMITS

Detection	Wavelength(a)(nm)	Estimated Element Limit(b) (ug/L)
Aluminum	308.215	45
Antimony	206.833	32
Arsenic	193.696	53
Barium	455.403	2
Beryllium	313.042	0.3
Cadmium	226.502	4
Calcium	317.933	10
Chromium	267.716	7
Cobalt	228.616	7
Copper	324.754	6
Iron	259.940	7
Lead	220.353	42
Lithium	670.784	5
Magnesium	279.079	30
Manganese	257.610	2
Molybdenum	202.030	8
Nickel	231.604	15
Potassium	766.491	See note c
Selenium	196.026	75
Silver	328.068	7
Sodium	588.995	29
Strontium	407.771	0.3
Thallium	190.864	40
Vanadium	292.402	8
Zinc	213.856	2

(a) The wavelengths listed are recommended because of their sensitivity and overall acceptance. Other wavelengths may be substituted if they can provide the needed sensitivity and are treated with the same corrective techniques for spectral interference. In time, other elements may be added as more information becomes available and as required.

(b) The estimated instrumental detection limits shown are given as a guide for an instrumental limit. The actual method detection limits are sample dependent analyst instrument, and matrix dependent.

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(c) Background contribution from continuous or recombination phenomena; and stray light from the line emission of high-concentration elements. Spectral overlap can be compensated for by computer-correcting the raw data after monitoring and measuring the interfering element. Unresolved overlap requires selection of an alternate wavelength. Background contribution and stray light can usually be compensated for by a background correction adjacent to the analyte line.

6.0 Definitions

6.1 Instrument Calibration Verification (ICV) Solution – A solution of method analytes, used to evaluate the performance of the instrument system with respect to a defined set of method criteria from a source different than that of the calibration standard.

6.2 Internal Standard – Yitrium is added to a sample, extract, or standard solution in known amount(s) and used to measure the relative responses of other method analytes that are components of the same sample or solution.

6.3 Laboratory Duplicate (LD) – One aliquot of the same sample taken in the laboratory and analyzed separately with identical procedures. Analyses of an LD1 indicates precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures. LD1s are analyzed with every batch of 20 samples.

6.4 Laboratory Control Sample (LCS) – An aliquot of LCS to which known quantities of the method analytes are added in the laboratory. The LCS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements.

6.5 Matrix spike (MS), matrix spike duplicate (MSD), and post digest spike (MSP) – An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS/MSD/MSP is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS/MSD/MSP corrected for background concentrations.

6.6 Laboratory Reagent Blank (LRB) – An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, and internal standards that are used with other samples. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, reagents, or apparatus.

6.7 Quality Control Sample (QCS) – A solution of method analytes of known concentrations which is used to fortify an aliquot of LRB or sample matrix.

6.8 Spectral Interference Check (SIC A & SIC AB) – A solution of selected method analytes of concentrations which is used to evaluate the procedural routine for correcting known inter-element spectral interferences. Analytes should be within 20% of the actual value run at a minimum frequency at the beginning of an analytical run. The absolute value of all non-spiked analytes < 2x MDL for the

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SICA.

7.0 Interferences

7.1 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 contribution from continuum or recombination phenomena; and (4) stray light from the line emission of high-concentration elements. Spectral overlap can be compensated for by computer-correcting the raw data after monitoring and measuring the interfering element. Unresolved overlap requires selection of an alternative wavelength. Background correction adjacent to the analyte line.

Users of all ICP instruments must verify the absence of spectral interference from an element in a sample for which there is no instrument detection channel. Recommended wavelengths are listed in Table 2 and potential spectral interferences for the recommended wavelengths are given in Table 3. The data in Table 2 are intended as rudimentary guides for indicating potential interferences; for this purpose, linear relations between concentration and intensity for the analytes and the interferents can be assumed.

7.1.1 The interference is expressed as analyte concentration equivalents (i.e. false analyte concentrations) arising from 100 mg/L of the interference element. For example, assume that As is to be determined (at 193.696 nm) in a sample containing approximately 10 mg/L of Al. According to Table 3, 100 mg/L of Al would yield a false signal for As equivalent to approximately 1.3 mg/L. Therefore, the presence of 10 mg/L of Al would result in a false signal for As equivalent to approximately 0.13 mg/L. The user is cautioned that other instruments may exhibit somewhat different levels of interference than those shown in Table 2. The interference effects must be evaluated for each individual instrument since the intensities will vary with operating conditions, power, viewing height, argon flow rate, etc.

7.1.2 The dashes in Table 3 indicate that no measurable interferences were observed even at higher interferent concentrations. Generally, interferences were discernible if the produced peaks, or background shifts, corresponding to 2 to 5% of the peaks generated by the analyte concentrations.

TABLE 3. ANALYTE CONCENTRATION EQUIVALENTS ARISING FROM INTERFERENCE AT THE 100-mg/L LEVEL

		Interferent (a,b)									
Analyte	Wavelength (nm)	Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Tl	V
Aluminum	308.215	-	-	-	-	-	-	0.21	-	-	1.4
Antimony	206.833	0.47	-	2.9	-	0.08	-	-	-	0.25	0.45
Arsenic	193.696	1.3	-	0.44	-	-	-	-	-	-	1.1
Barium	455.403	-	-	-	-	-	-	-	-	-	-
Beryllium	313/-42	-	-	-	-	-	-	-	-	0.04	0.05
Cadmium	226.502	-	-	-	-	0.03	-	-	0.02	-	-
Calcium	317.933	-	-	0.08	-	0.01	0.01	0.04	-	0.03	0.03
Chromium	267.716	-	-	-	-	0.003	-	0.04	-	-	0.04
Cobalt	228.616	-	-	0.03	-	0.005	-	-	0.03	0.15	-

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Copper	324.754	-	-	-	-	0.003	-	-	-	0.05	0.02
Iron	259.940	-	-	-	-	-	-	0.12	-	-	-
Lead	220.353	0.17	-	-	-	-	-	-	-	-	-
Magnesium	279.079	-	0.02	0.11	-	0.13	-	0.25	-	0.07	0.12
Manganese	257.610	0.005	-	0.01	-	0.002	0.002	-	-	-	-
Molybdenum	202.030	0.05	-	-	-	0.03	-	-	-	-	-
Nickel	231.604	-	-	-	-	-	-	-	-	-	-
Selenium	196.026	0.23	-	-	-	0.09	-	-	-	-	-
Sodium	588.995	-	-	-	-	-	-	-	-	0.08	-
Thallium	190.864	0.30	-	-	-	-	-	-	-	-	-
Vanadium	292.402	-	-	0.05	-	0.005	-	-	-	0.02	-
Zinc	213.856	-	-	-	0.14	-	-	-	0.29	-	-

(a) Dashes indicate that no interference was observed even when interferents were introduced at the following levels:

Al -	1000 mg/L	Mg -	1000 mg/L
Ca -	1000 mg/L	Mn -	200 mg/L
Cr -	200 mg/L	Tl -	200 mg/L
Cu -	200 mg/L	V -	200 mg/L
Fe -	1000 mg/L		

(b) The figures recorded as analyte concentrations are not the actual observed concentrations; to obtain those figures, add the listed concentration to the interferent figure.

7.2 Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by diluting the sample. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, which affects aerosol flow rate and causes instrumental drift. The problem can be controlled by diluting the sample.

7.3 Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with the ICP technique. If observed, they can be minimized by careful selection of operating conditions (incident power, observation position, etc), by buffering of the sample, by matrix matching, and by standard addition procedures. Chemical interferences are highly dependent on matrix type and the specific analyte element.

7.4 If the correction routine is operating properly, the determined apparent analyte(s) concentration from analysis of each interference solution should fall within a specific concentration range around the calibration blank. The concentration range is calculated by multiplying the concentration of the interfering element by the value of the correction factor being tested and dividing by 10. If after the subtraction of the calibration blank the apparent analyte concentration falls outside this range in either a positive or negative direction, a change in the correction factor of more than 10% should be suspected. The cause of the change should be determined and corrected and the correction factor updated.

8.0 Safety

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8.1 The toxicity or carcinogenicity of the stock standards or reagents in the method have not been precisely determined or defined; however, each compound should be treated as a potential health hazard. Cautions are included for known extremely hazardous materials or procedures.

8.2 The laboratory is responsible for maintaining a current awareness of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) are made available to all personnel involved in the chemical analysis. The preparation and utilization of a formal safety plan has also been established.

9.0 Apparatus and Materials

9.1 Inductively coupled argon plasma emission spectrometer:

9.1.1 Perkin Elmer 3000 DV or 3000XL

9.1.2 Argon gas supply - Welding grade or better

9.1.3 Nitrogen gas – zero grade

9.2 Operating conditions - The analyst should follow the instructions provided by Perkin Elmer. Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects must be established for each individual analyte line on that particular instrument. All measurements must be within the instrument calibration range. The analyst must (1) verify that the instrument configuration and operating conditions satisfy the analytical requirements and (2) maintain quality control data confirming instrument performance and analytical results.

9.3 Class A volumetric flasks

9.4 Class A volumetric pipets

9.5 Analytical balance- capable of accurate measurement to 4 significant figures.

10.0 Reagents

10.1 Trace metals grade shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. If the purity of a reagent is in question analyze for contamination. If the concentration is less than the MDL then the reagent is acceptable.

10.1.1 Hydrochloric acid (conc), HCl.

10.1.2 Hydrochloric acid (1:1), HCl. Add 500 mL concentrated HCl to 400 mL water and dilute to 1 liter in an appropriate beaker.

10.1.3 Nitric acid (conc), HNO3

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10.1.4 Nitric acid (1:1), HNO3. Add 500 mL concentrated HNO3 to 400 mL water and dilute to 1 liter in an appropriate beaker.

10.2 Reagent Water. All references to water in the method refer to reagent water unless otherwise specified. Reagent water will be ASTM Type1 water or equivalent.

10.3 Standard stock solutions will be purchased from a NIST traceable source.

10.4 Mixed calibration standard solutions - Prepare mixed calibration standard solutions by combining appropriate volumes of the stock solutions in volumetric flasks.

Working Stock: A mixture of 3 multi-element standards and 5 single element standards.

10 ml of plasma multi-element 10 ml of custom multi cal 1 10 ml of custom multi cal 2 1 ml of Si, Sn, Sr, Ti & W (1000 ug/L each)	100 ml DI

Calibration Standards 1-5: A dilution of the working stock in increasing concentration

Calibration Standard 1	0.20 ml working stock →	200 ml DI
Calibration Standard 2	1.0 ml working stock →	200 ml DI
Calibration Standard 3	10 ml working stock →	500 ml DI
Calibration Standard 4	10 ml working stock →	200 ml DI
Calibration Standard 5	20 ml working stock →	200 ml DI
Calibration Standard 6	A mix of 6 single element standards 1 ml Al & Fe (10000mg/L) 5 ml Ba, Cu, Mn, Zn (1000 mg/L)	500 ml DI
Calibration Standard 7	20 ml ml working stock Ca, Mg, K, Na (10000 mg/L)	

Calibration standards must be initially verified using a quality control sample and monitored weekly for stability. See Table 7 for ICP calibration units and standard concentrations.

[NOTE: If the addition of silver to the recommended acid combination results in an initial precipitation, add 15 mL of water and warm the flask until the solution clears. Cool and dilute to 100 mL with water. For this acid combination, the silver concentration should be limited to 2 mg/L. Silver under these conditions is stable in a tap-water matrix for 30 days. Higher concentrations of silver require additional HCl.]

10.5 Two types of blanks are required for the analysis. The calibration (rinse) blank is used in establishing the analytical curve, and the reagent blank (LRB) is used to correct for possible contamination resulting from varying amounts of the acids used in the sample processing.

10.5.1 The calibration blank is prepared by acidifying reagent water to the same concentrations of the acids found in the standards and samples. Prepare a sufficient quantity to flush the system between standards and samples.

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10.5.2 The reagent blank must contain all the reagents and in the same volumes as used in the processing of the samples. The reagent blank must be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis.

10.6 The Continuing Calibration Verification (CCV) is prepared by the analyst by combining compatible elements at concentrations equivalent to the midpoint of their respective calibration curves.

10.7 Spectral interference checks (SIC A & SIC AB) is prepared to contain known concentrations of interfering elements that will provide an adequate test of the correction factors. Spike the sample with the elements of interest at approximate concentrations of 10 times the instrumental detection limits. In the absence of measurable analyte, over-correction could go undetected because a negative value could be reported as zero. If the particular instrument will display over-correction as a negative number, this spiking procedure will not be necessary.

10.8 The quality control sample (QCS) should be prepared in the same acid matrix as the calibration standards at 10 times the instrumental detection limits and in accordance with the instructions provided by the supplier.

10.9 The Initial Calibration Verification (ICV) is prepared by the analyst by combining compatible elements from a source different than that of the calibration standard and at concentrations within the linear working range of the instrument.

11.0 Sample Collection, Preservation and Handling

11.1 Prior to the collection of an aqueous sample, consideration should be given to the type of data required (i.e., dissolved or total recoverable), so that appropriate preservation and pretreatment steps can be taken. The pH of all aqueous samples must be tested immediately prior to aliquoting for processing or "direct analysis" to ensure the sample has been properly preserved. If properly acid preserved, the sample can be held up to six months before analysis.

11.2 For the determination of the dissolved elements, the sample must be filtered through a 0.45 um pore diameter membrane filter at the time of collection or as soon thereafter as practically possible. (Glass or plastic filtering apparatus are recommended to avoid possible contamination. Only plastic apparatus should be used when the determinations of boron and silica are critical.) Use a portion of the filtered sample to rinse the filter flask, discard this portion collect the required volume of filtrate. Acidify the filtrate with (1+1) nitric acid immediately following filtration to pH <2.

11.3 For the determination of total recoverable elements in aqueous samples, samples are not filtered but acidified with (1+1) nitric acid to pH < 2 (normally, 3 mL of (1+1) acid per liter of sample is sufficient for most ambient and drinking water samples). Preservation may be done at the time of collection, however, to avoid the hazards of strong acids in the field, transport restrictions, and possible contamination, it is recommended that the samples be returned to the laboratory within two weeks of collection and acid preserved upon receipt in the laboratory. Following acidification, the sample should be mixed, held for 16 hours, and then

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verified to be pH <2 just prior to withdrawing an aliquot for processing or "direct analysis". If for some reason such as high alkalinity the sample pH is verified to be >2, more acid must be added and the sample held for 16 hours until verified to be pH <2.

12.0 Quality Control

12.1 All quality control data should be maintained and available for easy reference or inspection including opening QC, daily calibrations and subsequent CCV;s, blanks and SIC checks.

12.2 Dilute and reanalyze samples that are more concentrated than the calibration curve.

12.3 Employ a minimum of one reagent blank per sample batch to determine if contamination or any memory effects are occurring. A reagent blank is a volume of reagent water acidified with the same amounts of acids as were the standards and samples.

12.4 Employ a minimum of one laboratory control sample (LCS) and one laboratory control sample duplicate (LCSD) in each analytical batch. Recovery is to be within +/- 15% of the actual value. The LCS/LCSD samples are brought through the entire sample preparation and analytical process. A control limit of 20% RPD shall be used.

12.4.1 Client specific acceptance limits:
All Navy DoD LCS/LCSD recoveries for waters and soils shall be 80-120%. A control limit of 20 RPD shall be used.

12.5 Analyze matrix spiked duplicate samples at a frequency of one per matrix batch. A matrix duplicate sample is a sample brought through the entire sample preparation and analytical process in duplicate. Recovery is to be within +/- 25% of the actual value.

12.5.1 Client specific acceptance limits:
All Navy DoD MS/MSD recoveries for waters and soils shall be 80-120%. A control limit of 20% RPD shall be used.

12.5.2 The relative percent difference between matrix duplicate determinations is to be calculated as follows:

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

Where: RPD = relative percent difference
D1 = first sample value
D2 = second sample value (duplicate)

(A control limit of 20% RPD or within the documented historical acceptance limits for each matrix shall be used for sample values greater than ten times the instrument detection limit.)

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12.5.3 The spiked sample or spiked duplicate recovery is to be within +/- 25% of the actual value or within the documented historical acceptance limits for each matrix.

12.6 It is recommended that whenever a new or unusual sample matrix is encountered, a series of tests be performed prior to reporting concentration data for analyte elements. These tests will ensure the analyst that neither positive nor negative interferences are operating on any of the analyte elements to distort the accuracy of the reported values.

12.6.1 Serial dilution: If the analyte concentration is sufficiently high (minimally, a factor of 10 above the instrumental detection limit after dilution), an analysis of a 1:5-dilution should agree within +/- 10% of the original determination. If not, a chemical or physical interference effect should be suspected.

12.6.2 Post digestion spike (MSP): Aliquot of a sample spiked with method analytes after sample digestion. Analyte recoveries should be +/- 25% of actual value.

CAUTION: If spectral overlap is suspected, use of computerized compensation, an alternate wavelength, or comparison with an alternate method is recommended.

12.7 Check the instrument standardization by analyzing appropriate check standards as follows.

12.7.1 Verify calibration immediately after daily calibration, after every 10 samples and at the end of the analytical run, using a calibration blank and a continuing calibration verification standard (CCV).

12.7.1.1 The results of the check standard are to agree within 10% of the expected value; if not, terminate the analysis, correct the problem, and reanalyze the previous ten samples.

12.7.1.2 The results of the calibration blank are to agree within three standard deviations of the mean blank value. If not, repeat the analysis two more times and average the results. If the average is not within three standard deviations of the background mean, terminate the analysis, correct the problem, recalibrate, and reanalyze the previous 10 samples. If the blank is less than 1/10th the concentration of the action level of interest, and no sample is within ten percent of the action limit analyses need not be rerun and re-calibration need not be preformed before continuation of the run.

12.7.2 Verify the inter-element and background correction factors at the beginning of an analytical run. Do this by analyzing the interference check standards. Results should be within +/- 20% of the true value. SICA, SICAB are analyzed at the beginning of an analytical run. The absolute value of all non-spiked analytes < 2x MDL for the SICA.

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12.7.3 One LCS and one MS is to be analyzed at a frequency of 10%.

12.7.3.1 The spiked sample recovery should be within +/- 25% of the actual value.

12.8 Control Charts

Control charts are used to track the performance of LCS recoveries for AFCEE projects over time. NEL will track all analytes spiked into the LCS via control charts. NEL will update these charts on an annual basis and review them on a quarterly basis for possible trends that could compromise data quality. These charts can also be used as a benchmark of NEL performance against AFCEE requirements to determine possible areas for improvement.

As a minimum, NEL will create an LCS control chart for each method of analysis. Where both solid and aqueous LCS standards are analyzed, control charts will be maintained for both solid and aqueous matrices. Laboratory control limits will be equal to or within the AFCEE QAPP limits whenever such limits exist.

Out-of-control events detected in the control charts will be documented in a corrective action report. The following conditions, at a minimum, will be considered out-of-control events for all control charts at NEL:

- Any one point outside control limits.
- Any three consecutive points outside warning limits.
- Any six consecutive points such that each point is larger (or smaller) than its immediate predecessor.
- Any eight consecutive points on the same side of the centerline.

When an analyte in an LCS is outside the recovery control limits, data for that analyte may not be reported. All samples in the analytical batch must be reanalyzed for the out-of-control analyte after system problems have been resolved and system control has been established. When an analyte in an LCS exceeds the upper control limit and the analyte is not detected in the associated samples, no corrective action (reanalysis) is required.

13.0 Calibration and Standardization

13.1 Specific wavelengths used are listed in Table 1.

13.2 The plasma operating conditions should be optimized. The following procedure is recommended for vertically configured plasmas. The purpose of plasma optimization is to provide a maximum signal-to-background ratio for the least sensitive element in the analytical array. The use of a mass flow controller to regulate the nebulizer gas flow rate greatly facilitates the procedure.

13.3 Ignite the plasma and select an appropriate incident rf power with minimum reflected power. Allow the instrument to become thermally stable before beginning. This requires at least 60 minutes of operation. While aspirating the 1000 ug/mL solution of yitrium, follow the instrument manufacturer's instructions and adjust the aerosol carrier gas flow rate through the nebulizer so a definitive blue emission region of the plasma extends approximately from 5-20 mm above

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the top of the work coil.

13.4 After establishing the nebulizer gas flow rate, determine the solution uptake rate of the nebulizer in mL/min. by aspirating a known volume calibration blank for a period of at least three minutes. Divide the spent volume by the aspiration time (in minutes) and record the uptake rate. Set the peristalic pump to deliver the uptake rate in a steady even flow.

13.5 After horizontally aligning the plasma and/or optically profiling the spectrometer, use the selected instrument conditions and aspirate a plasma solution containing 10 ug/mL of Mn. Collect intensity data at the wavelength peak for each analyte at 1 mm intervals from 14-18 mm above the top of the work coil. (This region of the plasma is commonly referred to as the analytical zone.) Determine the net signal to blank intensity ratio for each analyte for each viewing height setting. Choose the height for viewing the plasma that provides the largest intensity ration for the least sensitive element of the four analytes.

13.6 If either the instrument operating conditions, such as incident power and/or nebulizer gas flow rate are changed, or a new torch injector tube having a different orifice i.d. is installed, the plasma and plasma viewing height should be reoptimized.

13.7 Before daily calibration and after the instrument warm-up period, the nebulizer gas flow is reset to the determined optimized flow. If a mass flow controller is being used, it should be reset to the recorded optimized flow rate. In order to maintain valid spectral inter-element correction routines, the nebulizer gas flow rate should be the same from day-to-day (<2% change).

13.8 Before using the procedure to analyze samples, there must be data available documenting initial demonstration of performance. This data must be generated using the same instrument operating conditions and calibration routine to be used for sample analysis. These documented data must be kept on file and be available for review by the data user.

13.9 After completing the initial demonstration of performance, but before analyzing samples, the laboratory must establish and initially verify an inter-element spectral interference correction routine to be used during sample analysis. To determine the appropriate location for background correction and to establish the inter-element interference correction routine, repeated spectral scan about the analyte wavelength and repeated analyses of the single element solutions may be required. Once established, the entire routine must be initially and periodically verified annually, or whenever there is a change in instrument operating conditions. Only a portion of the correction routine must be verified with more frequency or on a daily basis. Initial and periodic verification data of the routine should be kept on file.

14.0 Procedure

14.1 Preliminary treatment of most matrices is necessary because of the complexity and variability of sample matrices. Water samples which have been prefiltered and acidified will not need acid digestion. Solubilization and digestion

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procedures are presented in Sample Preparation Methods (eg. Methods 3005A-3050A).

14.2 Set up the instrument with proper operating parameters established. The instrument must be allowed to become thermally stable before beginning (usually requiring at least 30 minutes of operation prior to calibration).

14.3 Calibrate the instrument according to Perkin Elmers' recommended procedures, using the multi element calibration standard. Flush the system with the calibration blank between each standard. (Use the average intensity of multiple exposures for both standardization and sample analysis to reduce random error.) The calibration curve should consist of a blank and minimum of three standards. After calibration is complete the spectra of each analyte of interest, generated for the calibration shall be assess and profiled in the following manner:

14.3.1 Peak area will be used to generate the calibration curve and all data from the measured spectra.

14.3.2 The peak wavelength assessment point will be the highest point of the peak.

14.3.3 Background correction point(s) will be chosen so as to reflect the trend of the baseline. If the baseline is flat a single point may be used, if there is a slope either positive or negative then multiple points will be used.

14.3.4 Analytical points will be chosen so as to reflect the width of the peak. A narrow, sharp peak will have a single analytical point whereas a broad peak will require more points to assess the peak area.

14.3.5 After all peaks have been examined all changes will be saved and the calibration will be reprocessed to correctly reflect any changes made before continuing with analysis.

14.4 For all analytes and determinations, the laboratory must analyze an ICV, a calibration blank, and a continuing calibration verification (CCV) immediately following daily calibration. A calibration blank and a continuing calibration verification must be analyzed after every tenth sample and at the end of the sample run. Analysis of the check standard and calibration verification must verify that the instrument is within +/-10% of calibration with relative standard deviation <5% from replicate (minimum of two) integrations. If calibration cannot be verifed within the specified limits, the sample analysis must be discontinued, the cause determined and the instrument recalibrated. All samples following the last acceptable CCV or check standard must be reanalyzed. The analysis data of the calibration blank, check standard, and ICV or CCV must be kept on file with the sample analysis data.

14.5 Flush the system with the calibration blank solution for at least 1 minute before the analysis of each sample. Analyze the CCV and the calibration blank after each 10 samples.

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14.6 If an analyte of interest is greater than the highest calibration standard, then a dilution of the sample will be made to bring the analyte recovery within the range of calibration.

14.6.1 Prepared dilutions will be the minimum dilution necessary to bring an analyte within the calibration range.

14.7 The laboratory's reporting limit will be within the calibration range, either at or above the lower quantitation limit. If a client requires a lower reporting limit than supported by the calibration curve, then method modification is required.

15.0 Data Analysis and Calculation

15.1 The calibration curve is established by assuming that the relationship between concentration (X values) and intensity (Y values) is linear and that the following equation describes the relationship.

15.2.1 $Y = MX + B$

Where: X = concentration
Y = intensity
M = slope of the calibration
B = y-axis intercept

15.3 Calculation for solid samples is as follows:

15.3.1 $X * Y * Z = mg/Kg$

Where: X = Raw analyzed value
Y = Dilution factor from preparation
Z = Total solids factor; divide 1 by fractional total solids.

15.4 Calculation for spike recovery is as follows:

15.4.1 $S_R\,\% = (S_S - S) / S_C * 100$

Where: S_R = Percent spike recovery
S_S = Measured spiked sample analyte value
S = Measured sample analyte value
S_C = Spiking concentration.

15.5 Calculation for wipe samples is as follows:

15.5.1 $\frac{\text{Sample concentration}}{100\ cm^2} \times \frac{1\ Ng}{1000\ Ng} \times \frac{10.000\ cm^2}{1\ mL} = \text{Conc. } Ng/m^2$

15.5.2 $\frac{Conc/m^2}{10.76\ ft^2} = \text{Conc. } Ng/ft^2$

16.0 Method Performance

16.1 A method detection Limit (MDL) study is preformed annually for this analysis. The MDL study is conducted by taking seven replicate preparations of a low-concentration standard through all sample preparation steps and analysis for the specified analytical method. MDL values are calculated from the standard deviation of analytical results and the Student's t value for the number of replicate samples analyzed. The resulting calculated MDL values are evaluated by the Laboratory Quality Assurance Officer and Laboratory Manager against criteria to determine their acceptability. The MDL Study for Wipe Samples is pending.

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16.2 An instrument detection limit (IDL) study is performed twice a year for this analysis. The IDL study is conducted by taking seven replicate preparations of a low concentration standard equal to one-half of calibration standard one. IDL values are calculated from the standard deviation of analytical results and the students' true value for the number of replicate samples analyzed. The resulting IDL values are evaluated by the Laboratory Quality Assurance Officer and Laboratory Manager against criteria to determine their acceptability.

16.3 All IDL results are kept on file.

17.0 Pollution Prevention

17.1 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. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the EPA recommends recycling as the next best option.

17.2 Quantity of the chemicals purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18.0 Data Assessment, Acceptance and Correction Action

18.1 In the event that any measurement is out of control, the measurement is reported to the immediate supervisor and then to QA. If the ICP system, for example, is out of control, and the analyst has taken all appropriate steps for repair, the instrument will be taken out of service until the appropriate manufacturer can be contacted for repair. Any data generated from an out of control instrument will be flagged or if holding time permits, reanalyzed on another instrument. All out of control data is documented on corrective action forms and filed in the QA Manager's office.

18.2 In an effort to prevent instrument down-time the following maintenance schedule is used:

18.2.1 Daily check all fluids, chiller water, internal standard and rinse solution. Change peristaltic pump tubing.

18.2.2 Weekly, optimize torch alignment.

18.2.3 Monthly, inspect torch and purge glass(es), inspect O-rings.

18.2.4 Maintenance logbooks, instrument run logs and standards prep logs are maintained and kept in the metals lab per NEL SOP #3.7.10.

19.0 Waste Management

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The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Excess reagents, samples, and method process wastes should be characterized and disposed of in applicable manner. The EPA urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any waste discharge permit and regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult the "Waste Management Manual for Laboratory Personnel," available from the American Chemical Society at the address listed in Sect. 14.3.

20.0 References

Current version of US EPA Method 6010B

NELAC Quality Systems, Chapter 5, June 5, 2003

21.0 Tables, Diagrams, Flowcharts and Validation Data

See Attached.

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TABLE 4. ICP PRECISION AND ACCURACY DATA[a]

Element	Sample No. 1				Sample No. 2				Sample No. 3			
	True Conc. (ug/L)	Mean Conc. (ug/L)	RSD[b] (%)	Accuracy[d] (%)	True Conc. (ug/L)	Mean Conc. (ug/L)	RSD[b]	Accuracy[d] (%)	True Conc. (ug/L)	Mean Conc. (ug/L)	RSD[b] (%)	Accuracy[d] (%)
Be	750	733	6.2	98	20	20	9.8	100	180	176	5.2	98
Mn	350	345	2.7	99	15	15	6.7	100	100	99	3.3	99
V	750	749	1.8	100	70	69	2.9	99	170	169	1.1	99
As	200	208	7.5	104	22	19	23	86	60	63	17	105
Cr	150	149	3.8	99	10	10	18	100	50	50	3.3	100
Cu	250	235	5.1	94	11	11	40	100	70	67	7.9	96
Fe	600	594	3.0	99	20	19	15	95	180	178	6.0	99
Al	700	696	5.6	99	60	62	33	103	160	161	13	101
Cd	50	48	12	96	2.5	2.9	16	116	14	13	16	93
Co	700	512	10	73	20	20	4.1	100	120	108	21	90
Ni	250	245	5.8	98	30	28	11	93	60	55	14	92
Pb	250	236	16	94	24	30	32	125	80	80	14	100
Zn	200	201	5.6	100	16	19	45	119	80	82	9.4	102
Se[c]	40	32	21.9	80	6	8.5	42	142	10	8.5	8.3	85

[a]Not all elements were analyzed by all laboratories.
[b]RSD = relative standard deviation.
[c]Results for Se are from two laboratories.
[d]Accuracy is expressed as the mean concentration divided by the true concentration times 100.

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TABLE 5

ICP-AES PRECISION AND ACCURACY FOR AQUEOUS SOLUTIONS[a]

Element	Mean Conc. (mg/L)	N[b]	RSD[b] (%)	Accuracy[c] (%)
Al	14.8	8	6.3	100
Sb	15.1	8	7.7	102
As	14.7	7	6.4	99
Ba	3.66	7	3.1	99
Be	3.78	8	5.8	102
Cd	3.61	8	7.0	97
Ca	15.0	8	7.4	101
Cr	3.75	8	8.2	101
Co	3.52	8	5.9	95
Cu	3.58	8	5.6	97
Fe	14.8	8	5.9	100
Pb	14.4	7	5.9	97
Mg	14.1	8	6.5	96
Mn	3.70	8	4.3	100
Mo	3.70	8	6.9	100
Ni	3.70	7	5.7	100
K	14.1	8	6.6	95
Se	15.3	8	7.5	104
Ag	3.69	6	9.1	100
Na	14.0	8	4.2	95
Tl	15.1	7	8.5	102
V	3.51	8	6.6	95
Zn	3.57	8	8.3	96

[a]these performance values are independent of sample preparation because the labs analyzed portions of the same solutions

[b]N = Number of measurements for mean and relative standard deviation (RSD).

[c]Accuracy is expressed as a percentage of the nominal value for each analyte in acidified, multi-element solutions.

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TABLE 6

ICP-AES PRECISION AND BIAS FOR SOLID WASTE DIGESTS[a]

Element	Spiked Coal Fly Ash (NIST-SRM 1633a) Mean Conc. (mg/L)	N[b]	RSD[b] (%)	Bias[c] (%AAS)	Spiked Electroplating Sludge Mean Conc. (mg/L)	N[b]	RSD[b] (%)	Bias[c] (%AAS)
Al	330	8	16	104	127	8	13	110
Sb	3.4	6	73	96	5.3	7	24	120
As	21	8	83	270	5.2	7	8.6	87
Ba	133	8	8.7	101	1.6	8	20	58
Be	4.0	8	57	460	0.9	7	9.9	110
Cd	0.97	6	5.7	101	2.9	7	9.9	90
Ca	87	6	5.6	208	954	7	7.0	97
Cr	2.1	7	36	106	154	7	7.8	93
Co	1.2	6	21	94	1.0	7	11	85
Cu	1.9	6	9.7	118	156	8	7.8	97
Fe	602	8	8.8	102	603	7	5.6	98
Pb	4.6	7	22	94	25	7	5.6	98
Mg	15	8	15	110	35	8	20	84
Mn	1.8	7	14	104	5.9	7	9.6	95
Mo	891	8	19	105	1.4	7	36	110
Ni	1.6	6	8.1	91	9.5	7	9.6	90
K	46	8	4.2	98	51	8	5.8	82
Se	6.4	5	16	73	8.7	7	13	101
Ag	1.4	3	17	140	0.75	7	19	270
Na	20	8	49	130	1380	8	9.8	95
Tl	6.7	4	22	260	5.0	7	20	180
V	1010	5	7.5	100	1.2	6	11	80
Zn	2.2	6	7.6	93	266	7	2.5	101

[a]These performance values are independent of sample preparation because the labs analyzed portions of the same digests.

[b]N = Number of measurements for mean and relative standard deviation (RSD).

[c]Bias for the ICP-AES data is expressed as a percentage of atomic absorption spectroscopy (AA) data for the same digests.

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TABLE 7 - ICP Calibration Units and Standards Concentration

Analyte	units	Cal 1	Cal 2	Cal 3	Cal 4	Cal 5	Cal 6
Ag 328.068	mg/L	0.005	0.025	0.10	0.25	0.50	
Ag 338.289	mg/L	0.005	0.025	0.10	0.25	0.50	
Al 237.313	mg/L	0.02	0.1	0.4	1.0	2.0	20
As 188.979	mg/L	0.01	0.05	0.20	0.50	1.0	
B 182.528	mg/L	0.05	0.25	1.0	2.5	5.0	
B 249.772	mg/L	0.05	0.25	1.0	2.5	5.0	
Ba 233.527	mg/L	0.01	0.05	0.20	0.50	1.0	10
Be 313.107	mg/L	0.005	0.025	0.10	0.25	0.50	
Ca 317.933	mg/L	1.0	5.0	20.0	50.0	100.0	
Cd 228.802	mg/L	0.01	0.05	0.20	0.50	1.0	
Co 230.786	mg/L	0.05	0.25	1.0	2.5	5.0	
Cr 267.716	mg/L	0.01	0.05	0.20	0.50	1.0	
Cu 324.752	mg/L	0.01	0.05	0.20	0.50	1.0	10
Fe 238.204	mg/L	0.01	0.05	0.20	0.50	1.0	20
K 766.490	mg/L	1.0	5.0	20.0	50.0	100.0	
Li 670.784	mg/L	0.020	0.1	0.4	1.0	2.0	
Mg 280.271	mg/L	1.0	5.0	20.0	50.0	100.0	
Mg 279.077	mg/L	1.0	5.0	20.0	50.0	100.0	
Mn 257.610	mg/L	0.01	0.05	0.20	0.50	1.0	10
Mo 202.031	mg/L	0.05	0.25	1.0	2.5	5.0	
Na 589.592	mg/L	1.0	5.0	20.0	50.0		
Na 330.237	mg/L	1.0	5.0	20.0	50.0	100.0	
Ni 231.604	mg/L	0.01	0.05	0.20	0.50	1.0	
P 178.221	mg/L	0.02	0.10	0.40	1.0	2.0	
Pb 220.353	mg/L	0.01	0.05	0.20	0.50	1.0	
Sb 217.582	mg/L	0.05	0.25	1.0	2.5	5.0	
Se 196.026	mg/L	0.05	0.25	1.0	2.5	5.0	
Si 288.158	mg/L	0.01	0.05	0.20	0.50	1.0	
Sn 283.998	mg/L	0.01	0.05	0.20	0.50	1.0	
Sr 460.733	mg/L	0.01	0.05	0.20	0.50	1.0	
Ti 336.121	mg/L	0.01	0.05	0.20	0.50	1.0	
Tl 190.801	mg/L	0.05	0.25	1.0	2.5	5.0	
V 292.402	mg/L	0.05	0.25	1.0	2.5	5.0	
W 207.912	mg/L	0.01	0.05	0.20	0.50	1.0	
Zn 202.548	mg/L	0.01	0.05	0.20	0.5	1.0	10

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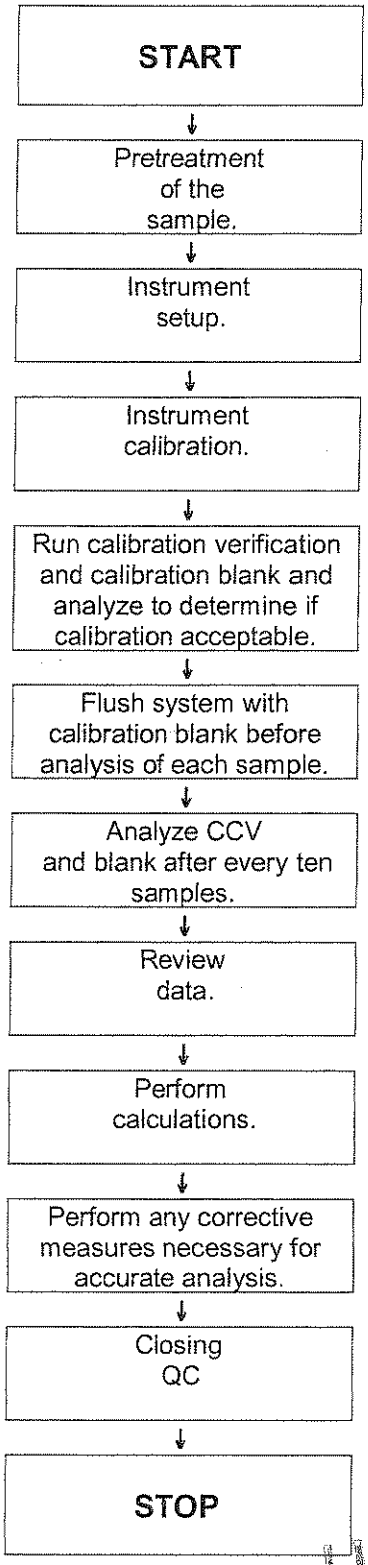
METHOD 6010B

INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROMETRY



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TITLE:	Determination of Mercury in Liquid Waste EPA 7470A
DEPARTMENTS AFFECTED:	Metals Chemistry Section

ISSUE DATE:	REVISION CODE:	SOP #:	REVISION CHANGES:
10/04/01	-	3.2.3	-
1/31/03	A	3.2.3	Update to NELAC form
5/13/04	B	3.2.3	Update Applicable Matrices
4/08/05	C	3.2.3	Annual Review & Update
1/31/06	D	3.2.3	Annual Review & Update
9/25/06	E	3.2.3	Update to include Navy Assessment Findings
4/25/07	F	3.2.3	Annual Review and Update
01/30/08	G	3.2.3	Annual Review & Update
02/28/08	H	3.2.3	Update Section 13.0

DOCUMENT APPROVAL		
Originator: Jacob Parker	Signature/Date: 3/4/08	Title Inorganic Analyst
Approved by: James Galasyn, Ph.D.	030408	Chemistry Lab Manager
Reviewed by: Andrew McCaw	030408	Metals Supervisor

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NEL	STANDARD OPERATING PROCEDURE		
Detemination of Mercury in Liquid Waste EPA 7470A			
SOP# 3.2.3	REV# H	EFFECTIVE 02/28/08	PAGE 2 of 11
WRITTEN BY: Jacob Parker	DEPARTMENTS AFFECTED: Metals Chemistry Section		

1.0 Test Method:

Method by Atomic Absorption Spectrometry, EPA Method 7470A.

2.0 Applicable Matrix or Matrices:

Wastewater, surface, groundwaters and mobility procedure extracts.

3.0 Detection Limit:

0.0002 mg/L for wastewaters, surface, groundwaters and mobility procedure extracts. Annual Method Detection Limit Studies (MDLs) are performed and all data is stored in the QA Manager's office.

4.0 Scope and Application

Method 7470A measures mercury (organic and inorganic) in mobility-procedure extracts, aqueous wastes, and groundwaters. All samples are subjected to an appropriate digestion step prior to analysis. If this digestion procedure is not sufficient to dissolve a specific matrix or type of sample, then this procedure is not acceptable for that matrix.

5.0 Summary of Method

5.1 Organic mercury compounds are oxidized and the mercury is reduced to the elemental state. The reducing agent, Tin (II) Chloride is introduced at a suitable point in the flow injection system and converts ionic mercury in the sample to a metallic mercury as it is transported through the system. The mercury vapor passes through a cell and into the light path (wavelength = 253.7nm.) Of an atomic absorption spectrometer. Absorbency (peak height) is measured as a function of mercury concentration.

5.2 Prior to analysis the liquid samples must be prepared according to the procedure outlined in this document.

5.3 The typical instrument detection limit (IDL) is 0.0002 mg/L.

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6.0 Definitions

6.1 Calibration Blank- Sample containing analytical reagents. Contains no mercury. Used as zero point when creating a calibration curve.

6.2 Flow Injection Mercury System (FIMS)- Analytical instrument used to measure the amount of mercury in a sample by atomic absorption.

6.3 Instrument Performance Check (IPC)- A solution of mercury with a concentration after or near the mid point of the calibration curve. The IPC is analyzed after the initial calibration, recalibration, and after each batch of 10 samples.

6.4 Laboratory Duplicate (LD1)- Extra aliquot of a sample which is digested separately from the original sample. Analysis of the LD1 indicates precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.

6.5 Laboratory Control Sample (LCS)- An aliquot of LCS to which a known quantity of mercury has been added in the laboratory. The LCS is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements.

6.6 Laboratory Control Sample Duplicate (LCSD)- An aliquot of LCSD to which a known quantity of mercury has been added in the laboratory. The LCSD is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements.

6.7 Matrix Spike (MS)- An aliquot of an environmental sample to which a known quantity of mercury has been added in the laboratory. The MS is analyzed exactly like a sample. Its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentration of mercury in the sample matrix must be determined in a separate aliquot and the measured value in the MS corrected for background concentrations.

6.8 Matrix Spike Duplicate (MSD)- An aliquot of an environmental sample to which a known quantity of mercury has been added in the laboratory. The MS is analyzed exactly like a sample. Its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentration of mercury in the sample matrix must be determined in a separate aliquot and the measured value in the MS corrected for background concentrations.

6.9 Laboratory Reagent Blank (LRB)- An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, solvents, and reagents that are used with other

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samples. The LRB is used to determine if mercury or other interferences are present in the laboratory environment, reagents, or apparatus.

6.10 Quality Control Sample (QCS)- A solution of mercury used to check either laboratory or instrument performance. The QCS is obtained from a source external to the laboratory and different from the source of the calibration standards.

6.11 Reporting Limit Check (RLC)- A solution of mercury having a concentration equal to the reporting limit for mercury for method 7470 (0.0002mg/L). The RLC is prepared from the same source as the calibration standards.

7.0 Interferences

7.1 Potassium permanganate is added to eliminate possible interference form sulfide. Concentrations as high as 20 mg/Kg of sulfide, as sodium sulfide do not interfere with the recovery of added inorganic mercury in reagent water.

7.2 Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/Kg have been shown to have no effect on the recovery of mercury from spiked samples.

7.3 Samples high in chlorides require additional permanganate (as much as 25 ml) because, during the oxidation step, chlorides are converted to free chlorine that also absorbs at 253 nm. All free chlorine must also be absent before samples are analyzed on the FIMS. This is accomplished by using an excess of sodium chloride hydroxylamine chloride reagent (25 ml) in the final step of the sample.

7.4 Certain organic compounds may also be absorbed at the desired wavelength. If this is suspected than the sample should be run first normally and subsequently without the reducing agent (stannous chloride). These results may then be subtracted to obtain the true mercury content of the sample.

8.0 Safety

8.1 Material Safety Data Sheets (MSDS) are available to all personnel involved in chemical analysis and handling. A written environmental health and safety plan is reviewed by all NEL staff.

8.2 Concentrated nitric and hydrochloric acids present various hazards and are moderately toxic and extremely irritating to the skin and mucus membrane. Use these reagents in a fume hood whenever possible and if eye or skin contact occurs, flush with large volumes of water. Always wear safety glasses, or a shield, for eye protection, protective clothing and observe proper procedures when mixing chemicals.

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8.3 The acidification of samples containing reactive materials may result in the release of toxic gases, such as cyanides or sulfides. Acidification of samples must be done in a fume hood.

8.4 All personnel handling environmental samples known to contain or to have been in contact with human waste should be immunized against known disease causing agents.

9.0 Equipment and Supplies

9.1 Flow Injection Mercury System:

9.1.1 Computer controlled absorption spectrometer. The laboratory has one Perkin Elmer Flow Injection Mercury System.

9.1.2 Argon gas supply- High purity grade (99.99%).

9.1.3 A variable speed peristaltic pump is used to deliver both standard and sample solutions to the mixing block of the FIMS.

9.2 Labware- Care must be taken to avoid contamination when digesting environmental samples prior to mercury analysis. Potential contamination sources include improperly cleaned laboratory apparatus and general contamination within the laboratory from dust, etc. A clean laboratory work area under a fume hood is used during the digestion process. All reusable labware (glass, quartz, polyethylene, PTFE, FEP, etc.) is sufficiently cleaned for task objectives. The cleaning procedure includes rinsing labware with hot tap water and reagent water, soaking in a 20% Nitric Acid Bath for at least 12 hours, followed by rinses with hot tap water and reagent water.

9.2.1 Glassware- Volumetric flasks and graduated cylinders (glass and/or metal free plastic). All volumetric flasks used are class A.

9.2.2 Assorted class A volumetric and calibrated adjustable pipettes.

10.0 Reagents and Standards

10.1 Only high purity reagents that conform to ACS specifications are used. Only optima grade sulfuric acid and nitric acid are used.

10.2 Reagent water: All references to water in this SOP refer to ASTM Type I grade water.

10.3 1% HNO3: Add 10 ml Optima grade Nitric Acid to 1 L of Reagent Water.

10.4 Stannous Chloride Solution: Add 5.5 g stannous chloride and 30 ml concentrated hydrochloric acid (trace metal grade or better) to 500 ml of reagent water, mix until all crystals are dissolved.

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10.5 Sodium Chloride-Hydroxylamine Chloride Solution: Dissolve 96 g sodium chloride and 96 g hydroxylamine chloride in reagent water and dilute to 800 ml.

10.6 Potassium Permanganate: Mercury free, dissolve 50g of potassium permanganate in reagent water. Dilute to 1L.

10.7 Potassium Persulfate: Dissolve 25g of potassium persulfate in 500 ml of reagent water.

10.8 Nitric Acid, concentrated: Optima grade.

10.9 Sulfuric Acid, concentrated: Optima Grade.

11.0 Sample Collection, Preservation and Storage

11.1 All samples must have been collected in a manner consistent with EPA methods.

11.2 All sample containers must be prewashed with detergents, acids, and reagent water. Plastic and glass containers are both suitable.

11.3 Aqueous samples must be acidified to a pH <2 with nitric acid. The maximum holding time for mercury is 28 days.

11.4 Nonaqueous samples are refrigerated, when possible, and analyzed as soon as possible.

12.0 Quality Control

12.1 All quality control data should be maintained and available for easy reference or inspection. The calibration correlation coefficient must be ≥ 0.995.

12.2 A calibration curve must be prepared for each run with a minimum of a calibration blank and five standards. After calibration, the calibration curve must be verified by use of at least a calibration blank and a Instrument Performance Check (IPC) (made from a reference material) at or near the mid-range. The calibration reference standard must be measured within 10% of its true value for the curve to be considered valid.

12.3 Also a Quality Control Standard QCS from a second source must be analyzed before any samples (field and QC). Value of second source for all analytes within ± 10% of the expected value.

12.4 If more than 10 samples per day are analyzed, the working standard curve must be verified by measuring satisfactorily a mid range standard (IPC) or reference standard after every 10 samples. This sample value

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must be within 10% of the true value, or the previous 10 samples need to be reanalyzed.

12.5 At least one matrix spike (MS) and one matrix spike duplicate (MSD) sample shall be included in each analytical batch. Spike recoveries must be no more than + or- 15% of the theoretical value. The relative percent difference (RPD) between spike duplicates must be no more than 20%. A laboratory control sample shall also be processed with each sample batch as well as a laboratory control sample duplicate. Calculations for % recovery and RPD can be found in Section 15.0 of this SOP.

12.6 If spike recoveries or relative % differences between spike duplicates fall outside of the acceptable range, the method of standard additions shall be used on all samples in the batch.

12.7 Where the sample matrix is so complex that viscosity, surface tension, and components cannot be accurately matched with standards, the method of standard addition (MSA) is recommended (see Section 8.7 EPA Method 7000A). Section 8.6 of EPA Method 7000A provides tests to evaluate the need for using the MSA.

13.0 Calibration and Standardization

13.1 Prepare 0.2, 0.5, 1, 2, 5 and 10 ug/L calibration standards from the 100 ug/L working standard using 100 ml class A volumetric flasks. Add 1% HNO3 and the appropriate amount of 1.0 mg/L working standard to each flask and dilute to the mark with 1% HNO3. Prepare a 4 ug/L QCS standard from the 1.0 mg/L QCS working standard in the same manner as the calibration standards.

13.2 Pour 25 ml of sample into a Falcon 50 ml centrifuge tube. Do a lab duplicate, a LRB, LCS, LCSD, and two MS's s for every 10 samples of each different matrix, i.e. wastewater, drinking water, TCLP extract. Ground waters and surface waters may be batched together and share the same set of LD1s, LRB's, LCS's, and MS's. Prepare LCS's and MS's by adding 0.1ml of the 1.0ug/L working standard to the appropriate samples.

13.3 From this point on, do all sample and standard preparation under a fume hood.

13.4 Add 1.25 ml of concentrated optima grade sulfuric acid to the samples and standards; mix well.

13.5 Add 0.625 ml of concentrated optima grade nitric acid to the samples and standards; mix well.

13.6 Add 3.75 ml of potassium permanganate to the samples and standards; mix well. Wait 15 minutes. Add an additional 3.75 ml aliquot of potassium permanganate to any samples in which the purple color does not persist

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after 15 minutes until the purple color persists for 15 minutes. Add the same additional amount to all of the samples and standards.

13.7 Add 2 ml of potassium persulfate to the samples and standards; mix well.

13.8 Heat the samples and standards for 2 hours in a 95-degree C covered water bath and let cool. If doing the above digestion procedure the day before analyzing the samples, let the samples cool overnight and do not continue the digestion procedure until the following day when your are ready to analyze the samples.

13.9 Just prior to analysis, add 1.5 ml of sodium chloride hydroxylamine chloride to the cooled samples/standards and mix until the purple color clears. Filter any samples containing visible particulates before analysis.

14.0 Procedure

14.1 Turn on the Argon gas supply and adjust the regulator to the proper pressure (approximately 40 psi).

14.2 Change the PTFE filter in the gas/liquid separator as needed. Be sure the smooth side of the filter faces down.

14.3 Before each run check the pump tubes. Replace any if needed. For further details on changing pump tubing see the FIMS **Installation, Maintenance, and System Description** handbook.

14.4 Place the reductant and carrier inlets in reagent water. On the toolbar, select the **FIAS** icon and set the pump 1 speed to 100. Measure the flow rates of the carrier and reductant for 1 minute. If the flow rates are below 9 and 5 ml/minute respectively, adjust the flows using the flow adjustment knobs until the proper flow rates are obtained.

14.5 Prepare a 3% hydrochloric acid solution for the wash and carrier solution. Prepare new tin (II) chloride solution before each run to use as the reducing agent. Place the inlets of the carrier and reagent tubes in the 3% hydrochloric acid and tin (II) chloride reagents respectively. Go to the **FIAS** screen and start Pump 1 in order to get these reagents flowing through the system.

14.6 Open the **SampInfo (Sample Information)** window to create the sample information table with the sample I.D. and its corresponding autosampler location.

14.7 Open the **MethEd (Method Editor)** window to bring up the method which will be used. We are using the method Hg 7470A for waters.

14.8 Load the autosampler with the standards and samples in appropriate locations as defined in the method and sample information table. The

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locations established in the method for Calibration Blank, Standards, and Quality Control Samples are as follows:

Standard	Autosampler Location
Calibration Blank	1
0.2ug/L Hg std.	2
0.5ug/L Hg std.	3
1.0ug/L Hg std.	4
2.0ug/L Hg std.	5
5.0ug/L Hg std.	6
10.0 ug/L Hg std.	7
QCS 4 ug/L Hg	8

14.9 Open the **Auto (Automated Analysis)** window to start the run. Make sure the sample information file and results files have been defined and that the save data and print log options are on. Save the sample information file and the results file under the same name using Hg and the day's date (i.e. hg90999). For further details on setting up an analysis see the **FIMS Setting Up and Performing Analyses** handbook.

14.10 The calibration curve must have a correlation coefficient of at least 0.995 and if it does not the run should be stopped and recalibrated. After the calibration is complete, the IPC, QCS should be run. Thereafter, the IPC should be run every 10 samples and at the end of the run. The concentrations of the Quality Control standards should be within 10% of their theoretical values. If the QC standards fail these criteria at any point during the run, the method should be set to stop, recalibrate or reslope, and rerun the QC standards and any samples that were run between the last passing QC and the failing QC.

14.11 Samples having a concentration of mercury above the calibration range must be diluted and reanalyzed.

14.12 The laboratory's reporting limit will lie within the calibration range, either at or above the lower quantitation limit. If a client requires a lower reporting limit than supported by the calibration curve, then method modification is required.

14.13 Once the analysis is complete, rinse the system with reagent water. Place the inlets of the reagent tubes into a beaker of reagent water. In the Automated Analysis Control window on the Analyze page, click on Select Location and select the Go to wash option and click on OK. In the Automated Analysis window, click on Move Probe Up/Down to raise the sampling probe. Place a beaker with reagent water in the wash location (location 0) and click on Move Probe Up/Down to lower the probe into the rinse solution. Go to the FIAS Control window and click on Valve Fill/Inject to turn the valve to the fill position. Set a value of 120 for Pump 1 and then start it. Click on Valve Fill/Inject several times while the pump is running to ensure that the inside of the FIAS-valve and the sample channel are rinsed well. Rinse the system for about 10 minutes. When done rinsing, remove the tubes from the rinse containers. Allow the

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pumps to run until all of the tubes and gas/liquid separator are dry. Stop the pumps and make sure the FIAS-valve is in the fill position. Swing all the pressure levers away from the pump tube magazines and empty the FIMS waste vessel. Turn off the Argon gas supply.

15.0 Data Analysis and Calculations

15.1 Sample data should be reported in mg/L for aqueous samples

15.2 For dissolved aqueous analytes report the data generated directly from the instrument with allowance for sample dilution. Do not report analyte concentrations below the IDL.

15.3 The QC data obtained during the analyses provide an indication of the quality of the sample data and should be provided with the sample results.

15.4 $$\% \text{ Recovery} = \frac{\text{Spike result} - \text{Sample result}}{\text{Actual Spike Value}} \times 100$$

15.5 $$\text{ABS} \left| \frac{\%\text{Recovery of Matrix Spike (MS)} - \%\text{ Recovery of Matrix Spike Dup (MSD) Result}}{\%\text{ Recovery MS} + \%\text{ Recovery MSD}/2} \right| \times 100 = \text{RPD}$$

16.0 Method Performance

Please see NEL SOP #3.2.34 for method specific performance data.

17.0 Pollution Prevention

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. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

18.0 Data Accessment, Acceptance and Corrective Action

18.1 Please refer to EPA method 245.1 (NEL SOP# 3.2.34).

18.2 Data is initially reviewed, dated and initialed by the analyst, then reviewed and validated by the supervisor. Please refer to SOP #3.5.1 for further information.

18.3 In the event that any measurement is out of control, the measurement is reported to the immediate supervisor and then to QA. Any corrective action undertaken is documented on a corrective action form (detailing

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the client information, problem, investigative findings and solution). This form is filed in the QA manager's office.

18.4 If the FIMS is producing out of control data, and the analyst has taken all appropriate steps for repair, the instrument will be taken our of service until the appropriate manufacturer can be contacted for repair. Any data generated from an out of control instrument will be flagged or if holding time permits, re-analyzed on another instrument. All out of control data is documented on corrective action forms and filed as above.

19.0 Waste Management

Any and all waste generated through any procedure in the laboratory is handled according to the Chemical Hygiene, Hazardous Waste Management and Spill Prevention Control and Contingency Plans of Northeast Laboratory Services, Inc.

20.0 References

US EPA Method 7470A, Mercury in Liquid Waste (Manual Cold-Vapor Technique), Revision 1, 1994

Methods for Chemical Analysis of Waters and Wastes, EPA-600/4-82-055, December 1982.

NELAC Quality Systems Chapter 5, June 5, 2003

21.0 Tables, Diagrams, Flowcharts and Validation Data

Not applicable.

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DOCUMENT HISTORY AND APPROVAL PAGE

TITLE:	Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectroscopy EPA Method 3010A
DEPARTMENTS AFFECTED:	Metals

ISSUE DATE:	REVISION CODE:	SOP #:	REVISION CHANGES:
4/16/02	-	3.2.5	Create an SOP in NELAC format
5/13/04	A	3.2.5	Update Heating Procedure
3/31/05	B	3.2.5	Annual Review & Update
5/04/06	C	3.2.5	Annual Review & Update
02/06/07	D	3.2.5	Annual Review & Update
01/23/08	E	3.2.5	Annual Review & Update
05/06/08	F	3.2.5	Update Sections 10.0 and 14.0

DOCUMENT APPROVAL		
Originator: Andrew McCaw	Signature/Date: Andrew McCaw 5-7-08	Title Inorganics Supervisor
Approved by: James Galasyn	J Galasyn 051208	Chemistry Lab Manager
Reviewed by: Pamela Doughty	Pamela Doughty 5/12/08	Total Quality Manager

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NEL	STANDARD OPERATING PROCEDURE		
TITLE: Acid Digestion of Aqueous Samples and Extracts for Total Metals Analysis by FLAA or ICP Spectroscopy			
SOP# 3.2.5	REV# F	EFFECTIVE 05/06/08	PAGE 2 of 8
WRITTEN BY: Andrew McCaw	DEPARTMENTS AFFECTED: Metals		

1.0 Test Method:

This standard operating procedure (SOP) was developed for EPA method 3010A, Acid Digestion of Aqueous Samples and Extracts for Total Metals Analysis by FLAA or ICP Spectroscopy.

2.0 Applicable Matrix or Matrices

This method is applicable to aqueous samples, TCLP extracts and wastes that contain suspended solids.

3.0 Detection Limit

Please see determinative method 6010B.

4.0 Scope and Application

4.1 This digestion procedure is used for the preparation of aqueous samples, TCLP extracts, and wastes that contain suspended solids for analysis, by flame atomic absorption spectroscopy (FLAA) or inductively coupled argon plasma spectroscopy (ICP). The procedure is used to determine total metals.

4.2 Samples prepared by Method 3010 may be analyzed by FLAA or ICP for the following:

Aluminum
Arsenic*
Barium
Beryllium
Cadmium
Calcium
Chromium
Cobalt
Copper
Iron
Lead
Magnesium
Manganese
Molybdenum
Nickel
Potassium
*Selenium
Sodium
Thallium
Vanadium
Zinc

*Analysis by ICP

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5.0 Summary of the Test Method

A mixture of nitric acid and the material to be analyzed is refluxed in a covered Griffin beaker. This step is repeated with additional portions of nitric acid until the digestate is light in color or until its color has stabilized. After the digestate has been brought to a low volume, it is refluxed with hydrochloric acid and brought up to volume. If sample should go to dryness, it must be discarded and the sample reprepared.

6.0 Definitions

LCS - Laboratory Control Standard – An aliquot of a laboratory reagent blank (LRB) to which a known concentration of the analyte of interest is added.

LCSD- Laboratory Control Standard Duplicate - An aliquot of a laboratory reagent blank (LRB) to which a known concentration of the analyte of interest is added. Both aliquots are prepared (and analyzed) exactly like a sample.

7.0 Interferences

Interferences are discussed in the referring analytical method.

8.0 Safety

8.1 The toxicity or carcinogenicity of the stock standards or reagents in the method have not been precisely determined or defined; however, each compound should be treated as a potential health hazard. Cautions are included for known extremely hazardous materials or procedures.

8.2 The laboratory is responsible for maintaining a current awareness of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) is made available to all personnel involved in the chemical analysis. The preparation and utilization of a formal safety plan has also been established.

9.0 Equipment and Supplies

9.1 Griffin beakers - 150-mL or equivalent.

9.2 Watch glasses - Ribbed and plain or equivalent.

9.3 Qualitative filter paper or centrifugation equipment.

9.4 Graduated cylinder or equivalent - 100mL.

9.5 Funnel or equivalent.

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9.6 Hot plate or equivalent heat source - adjustable and capable of maintaining a temperature of 90-95°C.

9.7 4 oz specimen container or equivalent.

10.0 Reagents and Standards

10.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

10.2 Reagent Water (DI water). Reagent water will be interference free. All references to water in the method refer to reagent water unless otherwise specified. Refer to SW 846, Chapter One for a definition of reagent water.

10.3 Nitric acid (concentrated), HNO_3. Acid should be analyzed to determine level of impurities. If method blank is less than the MDL, the acid can be used.

10.4 Hydrochloric acid (1:1), HCl. Prepared from water and hydrochloric acid. Hydrochloric acid should be analyzed to determine level of impurities. I method blank is less than the MDL, the acid can be used.

11.0 Sample Collection, Preservation, Shipment and Storage

11.1 All samples must have been collected using a sampling plan that addresses the considerations discussed in SW 846, Chapter Nine.

11.2 All sample containers must be pre-washed with detergents, acids, and water. Plastic and glass containers are both suitable.

11.3 Aqueous wastewaters must be acidified to a pH of < 2 with HNO_3.

12.0 Quality Control

12.1 All quality control measures described in SW 846, Chapter One should be followed.

12.2 For each analytical batch of samples processed, blanks should be carried throughout the entire sample-preparation and analytical process. These blanks will be useful in determining if samples are being contaminated. Refer to SW 846 Chapter One for the proper protocol when analyzing blanks.

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12.3 A duplicate sample should be processed with each analytical batch or every 20 samples, whichever is greater. Refer to SW 846, Chapter One for the proper protocol when analyzing replicates (duplicates).

12.4 Spiked samples (LFM) or standard reference materials (LFB) should be employed to determine accuracy. A spiked sample should be included with each batch of samples processed and whenever a new sample matrix is being analyzed. Refer to SW 846, Chapter One for the proper protocol when analyzing spikes.

12.5 The method of standard addition shall be used for the analysis of all EP extracts and delisting petitions (see Method 7000, Step 8.7). Although not required, use of the method of standard addition is recommended for any sample that is suspected of having an interference.

13.0 Calibration and Standardization

N/A

14.0 Procedure

14.1 Transfer a 50-mL aliquot of the sample to a 150-mL Griffin beaker and add 1.5 mL of concentrated HNO_3. Cover the beaker with a ribbed watch glass. Place the beaker on a 90-95°C hot plate or equivalent heat source and cautiously evaporate to a low volume (5 mL), making certain that the sample does not boil and that no portion of the bottom of the beaker is allowed to go dry. Cool the beaker and add another 1.5-mL portion of concentrated HNO_3. Cover the beaker with a nonribbed watch glass and return to the hot plate. Increase temperature of the hot plate so that a gentle reflux action occurs.

14.1.1 For a 5X digest, start with a 100 ml aliquot of sample and 3.0 ml of concentrated HNO_3 for both portions.

NOTE: If a sample is allowed to go to dryness, low recoveries will result. Should this occur, discard the sample and reprepare.

14.2 Continue heating, adding additional acid as necessary, until the digestion is complete (generally indicated when the digestate is light in color or does not change in appearance with continued refluxing). Uncover the beaker or use a ribbed watch glass, and evaporate to a low volume (3 mL), not allowing any portion of the bottom of the beaker to go dry. Cool the beaker and add a small quantity of 1:1 HCl (to be 10% of final solution volume. Cover the beaker, and reflux for an additional 15 minutes to dissolve any precipitate or residue resulting from evaporation.

14.3 Wash down the beaker walls and watch glass with water and, when necessary, filter or centrifuge the sample to remove silicates and other insoluble material that could clog the nebulizer. Filtration should be done only if there is concern that insoluble materials may clog the nebulizer. This additional step can cause sample contamination unless the filter and filtering apparatus are thoroughly cleaned. Rinse the filter and filter

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apparatus with dilute nitric acid and discard the rinsate. Pour the sample into an appropriate container and adjust the final volume to 25 ml with reagent water (final acid concentration of 10%). The sample is now ready for analysis.

14.3.1 For a 5X digestion, adjust final volume to 20 ml.

15.0 Calculations

N/A

16.0 Method performance

No data provided.

17.0 Pollution Prevention

17.1 The reagents in this SOP pose little threat to the environment when recycled and managed properly. Standards are made in batches where most of it will be used before expiration.

17.2 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the EPA recommends recycling as the next best option.

18.0 Data Assessment and Corrective Action

In the event that any measurement is out of control, the measurement is reported to the immediate supervisor and then to QA. If the microwave system, for example, is out of control, and the analyst has taken all appropriate steps for repair, the instrument will be taken out of service until the manufacturer can be contacted for repair. Any data generated from an out of control instrument will be flagged or if holding time permits, reanalyzed on another instrument or by another appropriate method. All out of control data is documented on corrective action forms and filed in the QA Manager's office.

19.0 Waste Management

The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Excess reagents, samples, and method process wastes should be characterized and disposed of in applicable manner. The EPA urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any waste discharge permit and regulations, particularly the hazardous waste identification

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rules and land disposal restrictions. For further information on waste management, please refer to Northeast Laboratory's Chemical Hygiene Plan and Spill Prevention Control and Contingency Plan, as well as the Hazardous Waste Management Plan.

20.0 References

NELAC Quality Systems Chapter 5, June 5, 2003.

Test methods for evaluating solid waste, physical/ chemical methods, 3rd edition; U.S. environmental Protection Agency, Office of Solid Waste and Emergency Response. US Government Printing Office: Washington DC, 1986; SW 846.
US EPA Method 3010A: Acid Digestion of Aqueous Samples and extracts for Total Metals for Analysis by FLAA or ICP Spectropscopy.

21.0 Tables, Diagrams, Flowcharts and Validation Data

See Attached

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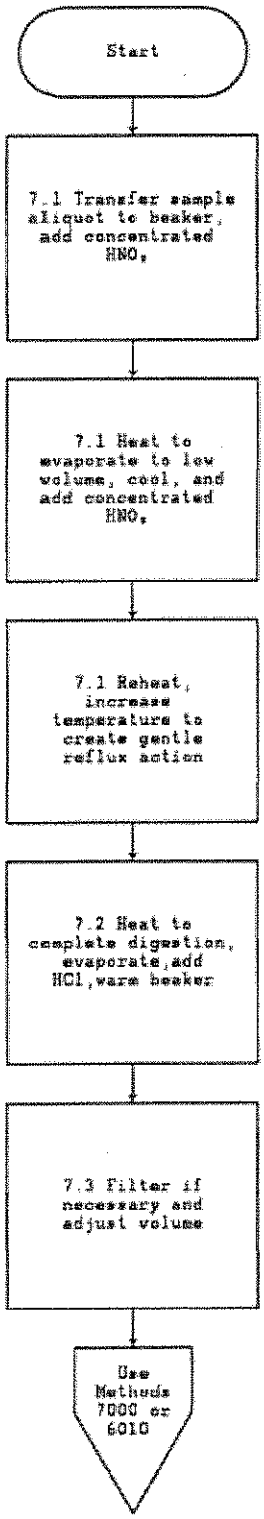
METHOD 3010A
ACID DIGESTION OF AQUEOUS SAMPLES AND EXTRACTS FOR TOTAL METALS ANALYSIS BY FLAA OR ICP SPECTROSCOPY



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DOCUMENT HISTORY AND APPROVAL PAGE

TITLE:	Toxicity Characteristic Leaching Procedure SW-846 Method 1311
DEPARTMENTS AFFECTED:	Inorganic Chemistry

ISSUE DATE:	REVISION CODE:	SOP #:	REVISION CHANGES:
06/06/02	-	3.2.8	Put into NELAC format
03/31/05	A	3.2.8	Annual Review & Update
08/08/06	B	3.2.8	Annual Review & Update
02/06/07	C	3.2.8	Annual Review & Update
02/25/08	D	3.2.8	Annual Review & Update

DOCUMENT APPROVAL		
Originator: Jacob Parker	**Signature/Date:** 2/25/08	**Title Metals Analyst**
Approved by: Andrew McCaw	2-25-08	**Metals Supervisor**
Reviewed by: Jim Galasyn	02/25/08	**Chemistry Lab Manager**

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NEL STANDARD OPERATING PROCEDURE			
TITLE: Toxicity Characteristic Leaching Procedure – SW-846 method 1311			
SOP# 3.2.8	REV# D	EFFECTIVE 02/25/08	PAGE 2 OF 29
WRITTEN BY: Jacob Parker	DEPARTMENTS AFFECTED: Inorganic Chemistry Section		

1.0 TEST METHOD

This standard operating procedure is for EPA method 1311, Toxicity Characteristic Leaching Procedure.

2.0 APPLICABLE MATRIX OR MATRICES

The TCLP is designed to determine the mobility of both organic and inorganic analytes present in liquid, solid, and multiphase wastes.

3.0 DETECTION LIMIT

The inter-laboratory detection limit for this analysis is dependent upon the individual analyte. Please see determinative methods.

4.0 SCOPE AND APPLICATION

4.1 The TCLP is designed to determine the mobility of both organic and inorganic analytes present in samples.

4.2 If a total analysis of the waste demonstrates that individual analytes are not present or are present but at such low concentrations that the appropriate regulatory levels could not possibly be exceeded, the TCLP need not be run.

4.3 This method should be used by or be under the supervision of analysts experienced in the TCLP procedure.

5.0 SUMMARY OF METHOD

5.1 Liquid samples (samples containing less than 0.5% dry solid material) are filtered though a 0.6 to 0.8 µm glass fiber filter. The resultant filtrate is then defined as the TCLP extract.

5.2 For samples containing greater than or equal to 0.5% dry solids, the liquid, if any, is separated from the solid phase and saved for later use in this method.

5.3 Limited performance-based method modifications may be acceptable provided they are fully documented and meet or exceed requirements expressed in Sect. 9.0, Quality Control.

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6.0 DEFINITIONS

6.1 ANALYSIS BATCH – The analysis batch must include an initial series of Calibration Standards, an Instrument Performance Check Standard (IPC), Quality Control Standard (QCS), Reporting Limit Check Standard (RLC), Laboratory Reagent Blank (LRB) (prior to each matrix), Laboratory Fortified Blank (LFB), and an End Calibration Check Standard. Within the Analysis Batch, for every group of ten field samples, at least one Laboratory Fortified Matrix (LFM) and either a Field Duplicate, a Laboratory Duplicate, or a duplicate of the LFM must be analyzed. In addition, when more than ten field samples are being analyzed, a Continuing Calibration Check Standard should be analyzed after the tenth field sample.

6.2 CALIBRATION BLANK (CB) – A volume of reagent water fortified with the same matrix as the calibration standards, but without the analytes, internal standards, or surrogate analytes.

6.3 CALIBRATION STANDARD (CAL) – A solution prepared from the primary standard solution or stock standard solutions. The four CAL standards are used to calibrate the instrument in respect to analyte concentrations.

6.4 INITIAL CALIBRATION STANDARDS – A series of CAL solutions used to initially establish instrument calibration and develop calibration curves for individual target compounds.

6.5 INITIAL CALIBRATION CHECK STANDARD – An individual CAL solution, analyzed prior to any sample analysis, used to verify calibration curve for sample quantification.

6.6 CONTINUING CALIBRATION CHECK STANDARDS – An individual CAL solution at method specified frequency.

6.7 END CALIBRATION CHECK STANDARD – An individual CAL solution that is analyzed after the last sample which confirms accurate quantification for all field samples within the entire ANALYSIS BATCH.

6.8 FIELD DUPLICATES – Two separate samples collected at the same time and placed under the same circumstances and treated exactly the same throughout the field and laboratory procedures. Analyses of field duplicates indicate the precision associated with sample collection, preservation and storage, as well as with laboratory procedures.

6.9 INSTRUMENT PERFORMANCE CHECK SOLUTION (IPC) – A solution of one or more method analytes used to evaluate the performance of the instrument system with respect to a defined set of criteria.

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6.10 LABORATORY DUPLICATE – Two sample aliquots, taken in the laboratory from a single sample bottle, and analyzed separately with identical procedures. Analysis of both samples indicates precision associated with the laboratory procedures, removing any associated variables attributed by sample collection, preservation, and storage procedures.

6.11 LABORATORY FORTIFIED BLANK (LFB) – An aliquot of reagent water or other blank matrices to which known quantities of the method analytes are added in the laboratory. The LFB is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

6.12 LABORATORY FORTIFIED SAMPLE MATRIX (LFM) – An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory.

6.13 LABORATORY REAGENT BLANK (LRB) – An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, solvents, and reagents that are used with other samples. The LRB is used to determine if method analytes or other interferences are present in the laboratory, reagents, or apparatus.

6.14 LABORATORY CALIBRATION RANGE (LCR) – The concentration range over which the instrument response is linear.

6.15 MATERIAL SAFETY DATA SHEET (MSDS) – Information provided by vendors concerning a chemical or reagent's toxicity, health hazards, physical properties, fire, and reactivity data including storage, waste management, spilling, and handling precautions.

6.16 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.

6.17 MINIMUM REPORTING LEVEL (MRL) – The minimum concentration that can be reported for an anion in a sample following analysis. This defined concentration can be no lower than the lowest concentration of the lowest calibration standard and can only be used if acceptable quality control criteria for this standard are met.

6.18 PERFORMANCE EVALUATION SAMPLE (PE) – A certified solution of method analytes whose concentration is unknown to the analyst. Often, an aliquot of this solution is added to a known volume of reagent water and analyzed with procedures used for samples. Results of analyses are used to determine statistically the accuracy and precision that can be expected from said methodology performed by qualified analysts.

6.19 QUALITY CONTROL SAMPLE (QCS) – A solution of method analytes of known concentrations that is used to fortify an aliquot of LRB or sample

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matrix. The QCS is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check the laboratory performance with external prepared test materials.

6.20 STOCK STANDARD SOLUTION (SSS) – A concentrated solution containing one or more method analytes prepared in the laboratory using reference materials or purchased stock solutions from reputable vendors.

7.0 INTERFERENCES

Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

8.0 SAFETY

8.1 The toxicity or carcinogenicity of the stock standards or reagents in the method have not been precisely determined or defined; however, each compound should be treated as a potential health hazard. Cautions are included for known extremely hazardous materials or procedures.

8.2 The laboratory is responsible for maintaining a current awareness of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) is made available to all personnel involved in the chemical analysis. The preparation and utilization of a formal safety plan has also been established.

9.0 EQUIPMENT AND SUPPLIES

9.1 Agitation apparatus: The apparatus must be capable of rotating the extraction vessel in an end-over-end fashion at 30 +/- 2 rpm.

9.2 Extraction vessels

9.2.1 Zero-headspace Extraction Vessel (ZHE). This device is for use only when the waste is being tested for the mobility of volatile analytes. The ZHE allows for liquid/solid separation within the device, and effectively precludes headspace. This type of vessel allows for initial liquid/solid separation, extraction, and final extract filtration without opening the vessel. The vessels shall have an internal volume of 500 – 600 mL, and be equipped to accommodate a 90-110 mm filter. The devices contain VITON O-rings which should be replaced frequently. For the ZHE to be acceptable for use, the piston within the ZHE should be able to be moved with approximately 15 psi or less. If it takes more pressure to move the piston, the O-rings in the device should be replaced. If this does not solve the problem, the ZHE is unacceptable for TCLP analyses and the manufacturer should be contacted. The ZHE should be checked for leaks after every extraction. If the device contains a built-in pressure gauge, pressure the device to

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50 psi, allow it to stand unattended for 1 hour, and recheck the pressure. If the device does not have a built-in pressure gauge, pressurize the device to 50 psi, submerge it in water, and check for the presence of air bubbles escaping from any of the fittings. If pressure is lost, check all fittings and inspect and replace O-rings, if necessary. Retest the device. If leakage problems cannot be solved, the manufacturer should be contacted. Some ZHEs use gas pressure to actuate the ZHE piston, while others use mechanical pressure. Whereas the volatiles procedure refers to pounds per square inch (psi), for the mechanically actuated piston, the pressure applied is measured in torque-inch-pounds. Refer to the manufacturer's instructions as to the proper conversion.

9.2.2 Bottle Extraction Vessel. When the waste is being evaluated using the nonvolatile extraction, a jar with sufficient capacity to hold the sample and the extraction fluid is necessary. Headspace is allowed in this vessel. The extraction bottles may be constructed from various materials depending on the analytes to be analyzed and the nature of the waste. It is recommended that borosilicate glass bottles be used instead of other types of glass, especially when inorganics are of concern. Plastic bottles, other than polytetrafluoroethylene, shall not be used if organics are to be investigated. Bottles are available from a number of laboratory suppliers. When this type of extraction vessel is used, the filtration device discussed in section 9.3 is used for initial liquid/solid separation and final extract filtration.

9.3 Filtration devices: It is recommended that all filtrations be performed in a hood.

9.3.1 Zero-Headspace Extractor Vessel (ZHE): When the waste is evaluated for volatiles, the zero-headspace extraction vessel described in section 9.2.1 is used for filtration. The device shall be capable of supporting and keeping in place the glass fiber filter and be able to withstand the pressure needed to accomplish separation (50 psi). Then it is suspected that the glass fiber filter has been ruptured, an in-line glass fiber filter may be used to filter the material within the ZHE.

9.3.2 Filter Holder: When the waste is evaluated for other than volatile analytes, any filter holder capable of supporting a glass fiber filter and able to withstand the pressure needed to accomplish separation may be used. Suitable filter holders range form simple vacuum units to relatively complex systems capable of exerting pressures of up to 50 psi or more. The type of filter holder used depends on the properties of the material to be filtered. These devices shall have a minimum internal volume of 300 ml and be equipped to accommodate a minimum filter size of 47 mm (filter holders having an internal capacity of 1.5 L or greater, and equipped to accommodate a 142 mm diameter filter, are recommended). Vacuum filtration can only be used for wastes

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with low solids content (<10%) and for highly granular, liquid-containing wastes. All other types of wastes should be filtered using positive pressure filtration.

9.3.3 Materials of Construction: Extraction vessels and filtration devices shall be made of inert materials which will not leach or absorb waste components. Glass, polytetrafluoroethylene (PTFE), or type 316 stainless steel equipment may be used when evaluating the mobility of both organic and inorganic components. Devices made of high density polyethylene (HDPE), polypropylene (PP), or polyvinyl chloride (PVC) may be used only when evaluating the mobility of metals. Borosilicate glass bottles are recommended for use over other types of glass bottles, especially when inorganics are analytes of concern.

9.4 Filters: Filters shall be made of borosilicate glass fiber, shall contain no binder materials, and shall have an effective pore size of 0.6 to 0.8 um, or equivalent. Pre-filters must not be used. When evaluating the mobility of metals, filters shall be acid-washed prior to use by rinsing with 1N nitric acid followed by three consecutive rinses with deionized distilled water (a minimum of 1 L per rinse is recommended). Glass fiber filters are fragile and should be handled with care.

9.5 pH Meters: The meter should be accurate to +/- 0.05 units at 25 °C.

9.6 ZHE Extract Collection Devices: TEDLAR bags or glass, stainless steel or PTFE gas-tight syringes are used to collect the initial liquid phase and final extract of the waste when using the ZHE device. The devices listed are recommended for use under the following conditions:

9.6.1 If a waste contains an aqueous liquid phase or if a waste does not contain a significant amount of nonaqueous liquid (i.e. <1% of total waste), the TEDLAR bag or a 600 mL syringe should be used to collect and combine the initial liquid and solid extract.

9.6.2 If a waste contains a significant amount of nonaqueous liquid in the initial liquid phase (i.e., >1% of total waste, the syringe or the TEDLAR bag may be used for both the initial solid/liquid separation and the final extract filtration. However, analysts should use one or the other, not both.

9.6.3 If the waste contains no initial liquid phase (is 100% solid) or has no significant solid phase (is 100% liquid), either the TELDAR bag or the syringe may be used. If the syringe is used, discard the first 5 mL of liquid expressed from the device. The remaining aliquots are used for analysis.

9.7 ZHE Extraction Fluid Transfer Devices: Any device capable of transferring the extraction fluid into the ZHE without changing the nature of the extraction fluid is acceptable (e.g, a positive

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displacement or peristaltic pump, a gas tight syringe, pressure filtration unit, or other ZHE device).

9.8 Laboratory Balance: Any laboratory balance accurate to within +/- 0.01 grams may be used (all weight measurements are to be within +/- 0.1 grams).

9.9 Beaker or Erlenmeyer flask, glass, 500 mL.

9.10 Watchglass, appropriate diameter to cover beaker or Erlenmeyer flask.

9.11 Magnetic Stirrer.

10.0 REAGENTS AND STANDARDS

10.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

10.2 Reagent water. Reagent water is defined as water in which an interferant is not observed at or above the method's detection limit of the analyte(s) of interest. For nonvolatile extractions, ASTM II water or equivalent meets the definition of reagent water. For volatile extractions, it is recommended that reagent water be generated by any of the following methods. Reagent waster should be monitored periodically for impurities.

10.2.1 Reagent water for volatile extractions may be generated by passing tap water through a carbon filter bed containing about 500 grams of activated carbon.

10.2.2 A water purification system (Millipore Super-Q or equivalent) may also be used to generate reagent water for volatile extractions.

10.2.3 Reagent water for volatile extractions may also be prepared by boiling for 15 minutes. Subsequently, while maintaining the water temperature at 90 +/- 5 degrees C, bubble a contaminant-free inert gas (i,e nitrogen) throughout the water for 1 hour. While it is still hot, transfer the water to a narrow mouth screw-cap bottle under zero-headspace and seal with a Teflon-lined septum and cap.

10.3 Hydrochloric acid (1N), HCl, made from ACS reagent grade.

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10.4 Nitric acid (1N), HNO_3, made from ACS reagent grade.

10.5 Sodium Hydroxide (1N), NaOH, made form ACS reagent grade.

10.6 Glacial acetic acid CH_3CH_2OOH, ACS reagent grade.

10.7 Extraction fluid.

10.7.1 Extraction fluid #1: Add 5.7 mL of glacial CH_3CH_2OOH to 500 mL of reagent water, add 64.3 mL of 1 N NaOH, and dilute to volume of 1 liter. When correctly prepared, the pH of this fluid will be 4.93 +/- 0.05.

10.7.2 Extraction fluid #2: Dilute 5.7 mL of glacial CH_3CH_2OOH with reagent water to a volume of 1 liter. When correctly prepared, the pH of this fluid will be 2.88 +/- 0.05.

NOTE: These extraction fluids should be monitored frequently for impurities. The pH should be checked prior to use to ensure that these fluids are made up accurately. If impurities are found or the pH is not within the above specifications, the fluid shall be discarded and fresh extraction fluid prepared.

10.8 Analytical standards shall be prepared according to the appropriate analytical method.

11.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

11.1 All samples shall be collected using an appropriate sampling plan.

11.2 The TCLP may place requirements on the minimal size of the filed sample, depending upon the physical state (or states) of the waste and the analytes of concern. An aliquot is needed for preliminary evaluation of which extraction fluid is to be used for the nonvolatile analyte extraction procedure. Another aliquot may be needed to actually conduct the nonvolatile extraction. If volatile organics are of concern, another aliquot may be needed. Quality control measures may require additional aliquots. Further, it is always wise to collect more sample just in case something goes wrong with the initial attempt to conduct the test.

11.3 Preservatives shall not be added to samples before extraction.

11.4 Samples may be refrigerated unless refrigeration results in irreversible physical change to the waste. If precipitation occurs, the entire sample (including the precipitate) should be extracted.

11.5 When the waste is to be evaluated for volatile analytes, care shall be taken to minimize the loss of volatiles. Samples shall be collected and stored in a manner intended to prevent the loss of volatile analytes (e.g.,

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samples should be collected in Teflon-lined septa capped vials and stored at 4 °C. Samples should be opened only immediately prior to extraction.

11.6 TCLP extracts should be prepared for analysis and analyzed as soon as possible following extraction. Extracts or portions of extracts for metallic analyte determinations must be acidified with nitric acid to a pH <2, unless precipitation occurs. Extracts should be preserved for other analytes according to the guidance given in the individual analysis methods. Extracts or portions of extracts for organic analyte determinations shall not be allowed to come into contact with the atmosphere (i.e., no headspace) to prevent loss.

12.0 QUALITY CONTROL

12.1 Every analyst using this method is required to operate under a laboratory quality assurance program (LQAP). The requirements of this program consists of an initial demonstration of laboratory performance, and subsequent analysis in each analysis batch (sect. 3.1) of a Laboratory Reagent Blank, Laboratory Fortified Blank, Instrument Performance Check standard, calibration check standards, Laboratory Fortified Sample Matrices (LFM) and either Field, Laboratory, or LFM duplicate sample analyses. The laboratory is required to maintain performance records that define the quality of the generated data. This section further defines such requirements.

12.2 INITIAL DEMONSTRATION OF PERFORMANCE

12.2.1 The initial demonstration of performance is used to characterize analyst capabilities (by the determination of four sequential Laboratory Fortified Blanks and through the analysis of an initial QCS) and instrument performance (by determination of MDLs) prior to performing or reporting analyses by this method.

12.2.2 Quality Control Sample (QCS) – At the beginning of each running sequence or batch, the QCS should be run on a weekly basis or as often as required to meet data quality, instrument performance, and verification of adequate calibration. If QCS fails within +/- 15% of assigned concentration values, corrective action must be taken before any further analysis.

12.2.3 Method Detection Limit (MDL) – Method detection limits must be established for all analytes before any analyses can be determined. These MDLs should be performed on an aqueous media and analyzed by the applicable analytical method. The determination of MDL's values are produced by running seven replicate aliquots of reagent water fortified at a concentration at three to five times higher than the estimated instrument detection limit. The values for all seven analytes must be processed within three consecutive days and fully determined within a single sequence. Baseline noise must also be utilized as a

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determinate for the final assigned MDL values. The calculations for the MDL's are defined as follows:

$$MDL = (t) \times (S)$$

Where, (t) equals the minimum concentration of an analyte that can be identified or measured with 99% confidence and a standard deviation estimate with n-1 degrees of freedom [t = 3.14 for seven replicates]. (S) equals the standard deviation of the replicate analyses.

The MDLs should be determined every 6 months, when a new analyst performs said method of analysis, a change in baseline noise occurs, or with any instrument additions or changes (i.e. new columns, eluent).

12.3 ASSESSING LABORATORY PERFORMANCE

12.3.1 Laboratory Reagent Blank (LRB) – A minimum of one blank (using the same extraction fluid as used for the samples) must be analyzed for every 20 extractions that have been conducted in an extraction vessel.

12.3.2 Laboratory Fortified Blank (LFB) – The laboratory fortified blank should be run with every analysis batch and should be prepared at concentrations equivalent to the concentrations used with the LFM. The LFB is not prepared during the TCLP leaching procedure. It is prepared and analyzed concurrently with the individual analytical procedure. The calculated recovery should be reported as percent recovery. The source of poor recovery should be identified and documented. If the source is not determined, the problem must be resolved before continuing any analyses.

12.3.2.1 Control Limits for the LFB- See individual preparative method.

12.3.2.1.1 The LFB is used to assess laboratory performance against the required control upper and lower limits described in 12.3.2.2. When sufficient internal performance data becomes available (usually a minimum of 20-30 analyses), optional control limits can be developed from the percent mean recovery (x) and the standard deviation (S) of the mean recovery. These data points can be used o establish upper and lower control limits as follows:

$$\text{UPPER CONTROL LIMIT} = x + 3S$$

$$\text{LOWER CONTROL LIMIT} = x - 3S$$

The optional control limits must be equal or better than those listed in 12.3.2.2. After each five to ten new recovery measurements, new control limits can be calculated using only the most recent 20 – 30 data points. Also, the standard deviation (S) data should be used to establish an on-going precision statement for the level of

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concentrations monitored. These data must be kept on file and be available for review.

12.3 ASSESSING ANALYTE RECOVERY AND DATA QUALITY

12.3.1 Laboratory Fortified Sample Matrix (LFM) – A matrix spike shall be performed for each waste type (e.g., wastewater treatment sludge, contaminated soil, etc) unless the result exceeds the regulatory level and the data are being used solely to demonstrate that the waste property exceeds the regulatory level. A minimum of one matrix spike must be analyzed with each analytical batch. As a minimum, follow the matrix spike addition guidance provided in each analytical method. Matrix spikes are added after filtration of the TCLP extract and before preservation. Matrix spikes should not be added prior to TCLP extraction of the samples.

12.3.1.2 The concentrations for each analyte should be corrected for concentrations measured in the unfortified samples and recorded as percent recovery using the following equation:

$$\%R = \frac{C_2 - C_1}{s} \times 100$$

where,
R = percent recovery
C_2 = fortified sample concentration
C_1 = sample concentration
S = concentration equivalent to each analyte added to sample

12.3.1.3 The laboratory control limits for percent mean recovery should be within the ranges specified by the appropriate determinative method. If the recovery falls outside of this set range, and the laboratory performance is shown to be in control, than the recovery problem can be assessed as a matrix induced bias.

12.3.2 LABORATORY and FIELD DUPLICATES – A field or laboratory duplicate must be run for every ten samples within a matrix or for a minimum of 10% of all samples within the analysis batch. The sample matrix selected for this duplicate analysis must contain measurable concentrations of all target analytes to ensure precision and quality of the data. If all samples within an analysis batch have low concentrations, than the LFM should be implemented as the laboratory duplicate.

12.3.2.1 The precision of all duplicates should be calculated as relative percent recovery (RPD) using the following equation:

$$RPD = \frac{100\,(S_1 - S_2)}{[(S_1 + S_2)/2]}$$

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where,
S_1 = the original concentration
S_2 = the concentration of the duplicate sample

12.3.2.2 The minimum acceptance level for duplicate RPD should be +/- 20%. If the RPD falls outside of these parameters, it usually indicates an instrument problem, and neither result should be reported.

12.3.3 The use of internal calibration quantitation method shall be employed for a metallic contaminant if: (1) Recovery of the contaminant from the TCLP extract is not a least 50% and the concentration does not exceed the regulatory level, and (2) the concentration of the contaminant measured in the extract is within 20% of the appropriate regulatory level.

12.3.3.1 The method of standard additions shall be employed as the internal calibration quantitation method for each metallic contaminant.

12.3.3.2 The method of standard additions requires preparing calibration standards in the sample matrix rather than reagent water or blank solution. It requires taking four identical aliquots of the solution and adding known amounts of standard to three of these aliquots. The fourth aliquot is unknown. Preferably, the first addition should be prepared so that the resulting concentration is approximately 50% of the expected concentration of the sample. The second and third additions should be prepared so that the concentrations are approximately 100% and 150% of the expected concentration of the sample. All four aliquots are maintained at the same final volume by adding reagent water or a blank solution, and may need dilution adjustment to maintain the signals in the linear range of the instrument technique. All four aliquots are analyzed.

12.3.3.2.1 Prepare a plot, or subject data to linear regression, of instrument signals or external-calibration-derived concentrations as the dependent variable (y-axis) versus concentrations of the additions of standard as the independent variable (x-axis). Solve for the intercept of the abscissa (the independent variable, x-axis) which is the concentration in the unknown.

12.3.3.2.2 Alternatively, subtract the instrumental signal or external-calibration-derived concentration of the unknown (unspiked) sample from the instrumental signals or external-calibration-derived concentrations of the standard additions. Plot or subject to linear regression of the

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corrected instrument signals or external-calibration-derived concentration as the dependent variable versus the independent variable. Derive concentrations for unknowns using the internal calibration curve as if it were an external calibration curve.

13.0 CALIBRATION AND STANDARDIZATION

See the appropriate determinative method.

14.0 PROCEDURE

14.1 Preliminary Evaluations- Perform preliminary TCLP evaluations on a minimum 100 gram aliquot of waste. This aliquot may not actually undergo TCLP extraction. These preliminary evaluations include: (1) determination of the percent solids; (2) determination of whether the waste contains insignificant solids and is, therefore, its own extract after filtration; (3) determination of whether the solid portion of the waste requires particle size reduction; and (4) determination of which of the two extraction fluids are to be used for the nonvolatile TCLP extraction of the waste.

14.1.1 Preliminary determination of percent solids: Percent solids is defined as that fraction of a waste samples (as a percentage of the total sample) from which no liquid may be forced out by an applied pressure, as described below.

14.1.1.1 If the waste will obviously yield no liquid when subjected to pressure filtration (i.e.,100% solids) proceed to section 14.1.3.

14.1.1.2 If the sample is liquid or multiphasic, liquid/solid separation to make a preliminary determination of percent solids is required. This involves the filtration device described in Section 9.3 and is outlined in Sections 14.1.1.3 through 14.1.1.9.

14.1.1.3 Pre-weigh the filter and the container that will receive the filtrate.

14.1.1.4 Assemble the filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure.

14.1.1.5 Weigh out a sub-sample of the waste (100 gram minimum) and record the weight.

14.1.1.6 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration. If used, the liquid should be decanted and filtered followed by

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filtration of the solid portion of the waste through the same filtration system.

14.1.1.7 Quantitatively transfer the waste sample to the filter holder (liquid and solid phases). Spread the waste sample evenly over the surface of the filter. If filtration of the waste at 4 °C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering. NOTE: If waste material (>1% of original sample weight) has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of the residue and subtract it from the sample weight determined in section 14.1.1.5 to determine the weight of the waste sample that will be filtered. Gradually apply vacuum or gently pressure of 1 – 10 psi until air pressurizing gas moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psi increment. When the pressurizing gas begins to move through the filter, or when liquid flow has ceased at 50 psi (i.e., filtration does not result in any additional filtrate within any 2 minute period), stop the filtration. NOTE: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

14.1.1.8 The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase. NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying vacuum or pressure filtration, as outlined in Section 14.1.1.7, this material may not filter. If this is the case, the material within the filtration device is defined as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.

14.1.1.9 Determine the weight of the liquid phase by subtracting the weight of the filtrate container from the total weight of the filtrate-filled container. Determine the weight of the solid phase of the waste sample by subtracting the weight of the liquid phase from the weight of the total waste sample, as determine din Section 14.1.1.5 or 14.1.1.7. Record the weight of the liquid and solid phases. Calculate the percent solids as follows:

$$\text{Percent solids} = \frac{\text{Weight of solid (Section 14.1.1.9)} \times 100}{\text{Total weight of waste (Section 14.1.1.5 or 14.1.1.7)}}$$

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14.1.2 If the percent solids determined in Section 14.1.1.9 is equal to or greater than 0.5%, then proceed either to Section 14.1.3 to determine whether the solid material requires particle size reduction or to section 14.1.2.1 if it is noticed that a small amount of the filtrate is entrained in wetting of the filter. If the percent solids determined in Section 14.1.1.9 is less than 0.5%, then proceed to Section 14.2.9 if the nonvolatile TCLP is to be performed and to Section 14.3 with a fresh portion of the waste if the volatile TCLP is to be performed.

14.1.2.1 Remove the solid phase and filter from the filtration apparatus.

14.1.2.2 Dry the filter and solid phase at 100 +/- 20 °C until two successive weighing yield the same value within +/- 1%. Record the final weight. NOTE: Caution should be taken to ensure that the subject solid will not flash upon heating. It is recommended that the drying oven be vented to a hood or other appropriate device.

14.1.2.2.1 Calculate the percent dry solids as follows:

$$\text{Percent dry solids} = \frac{(\text{Wt of dry waste + filter}) - \text{tared wt. of filter}}{\text{Initial wt. of waste (Section 14.1.1.5 or 14.1.1.7)}} \times 100$$

14.1.2.3 If the percent dry solids is less than 0.5%, then proceed to Section 14.2.9 if the nonvolatile TCLP is to be performed, and to Section 14.3 If the volatile TCLP is to be performed. If the percent dry solids is greater than or equal to 0.5%, and if the nonvolatile TCLP is to be performed., return tot he beginning of this Section (14.1) and, with a fresh portion of waste, determine whether particle size reduction is necessary (Section 14.1.3) and determine the appropriate extraction fluid (Section 14.1.4). If only the volatile TCLP is to be performed, see the note in Section 14.1.4.

14.1.3 Determination of whether the waste requires particle size reduction (particle size is reduced during this step): Using the solid portion of the waste, evaluate the solid for particle size. Particle size reduction is required, unless the solid has a surface area per gram of material equal to or greater than 3.1 cm^3, or is smaller than 1 cm in its narrowest dimension (i.e., is capable of passing through a 9.5 mm (0.375 inch) standard sieve). If the surface area is smaller or the particle size larger than described above, prepare the solid portion of the waste for extraction by crushing, cutting, or grinding the waste to a surface area or particle size as described above. If the solids are prepared for organic volatile extraction, special precautions must be taken (see Section 14.3.6). NOTE: Surface area criteria are meant for filamentous (e.g, paper, cloth, and similar) waste materials. Actual measurement of surface area is not required, nor is it recommended. For materials that do not obviously meet the criteria, sample specific methods would need

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to be developed and employed to measure the surface area. Such methodology is currently not available.

14.1.4 Determination of appropriate extraction fluid: If the solid content of the waste is greater than or equal to 0.5% and if the sample will be extracted for nonvolatile constituents (Section 14.2), determine the appropriate fluid (Section 10.7) for the nonvolatiles extraction as follows: NOTE: TCLP extraction for volatile constituents uses only extraction fluid #1 (Section 10.7.1). Therefore, if TCLP extraction for nonvolatiles is not required, proceed to Section 14.3.

14.1.4.1 Weigh out a small sub-sample of the solid phase of the waste, reduce the solid (if necessary) to a particle size of approximately 1 mm in diameter or less, and transfer 5.0 grams of the solid phase of the waste to a 150 mL beaker or Erlenmeyer flask.

14.1.4.2 Add 96.5 mL of reagent water to the beaker, cover with a watchglass, and stir vigorously for 5 minutes using a magnetic stirrer. Measure and record the pH. If the pH is <5.0, use extraction fluid #1. Proceed to Section 14.2.

14.1.4.3 If the pH from Section 14.1.4.2 is >5.0, add 3.5 mL 1N HCl, slurry briefly, cover with a watchglass, heat to 50 °C for 10 minutes.

14.1.4.4 Let the solution cool to room temperature and record the pH. If the pH is <5.0, use extraction fluid #1. If the pH is >5.0, use extraction fluid #2. Proceed to Section 14.2.

14.1.4.5 If the aliquot of the waste used for the preliminary evaluation (Sections 14.1.1 – 14.1.4) was determined to be 100% solid at Section 14.1.1.1, then it can be used for the Section 14.2 extraction (assuming at least 100 grams remain), and the Section 14.3 extraction (assuming at least 25 grams remain). If the aliquot was subjected to the procedure in Section 14.1.1.7, then another aliquot shall be used for the volatile extraction procedure in Section 14.3. The aliquot of the waste subjected to the procedure in Section 14.1.1.7 might be appropriate for use for Section 14.2 extraction if an adequate amount of solid (as determined by Section 14.1.1.9) was obtained. The amount of solid necessary is dependent upon whether a sufficient amount of extract will be produced to support the analyses. If an adequate amount of solid remains, proceed to Section 14.2.10 of the nonvolatile TCLP extraction.

14.2 Procedure When Volatiles are not Involved- A minimum sample size of 100 grams (solid and liquid phases) is recommended. In some cases, a larger sample size may be appropriate, depending on the solids content of the waste sample (percent solids, see Section 14.1.1), whether the initial liquid

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phase of the waste will be miscible with the aqueous extract of the solid, and whether inorganics, semivolatile organics, pesticides, and herbicides are all analytes of concern. Enough solids should be generated for the extraction such that the volume of TCLP extract will be sufficient to support all of the analyses required. If the amount of extract generated by a single TCLP extraction will not be sufficient to perform all of the analyses, more than one extraction may be performed and the extracts form each combined and aliquoted for analysis.

14.2.1 If the waste will obviously yield no liquid when subjected to pressure filtration (i.e., is 100% solid, see Section 14.1.1), weigh out a sub-sample of the waste (100 gram minimum) and proceed to Section 14.2.9.

14.2.2 If the sample is liquid or multiphasic, liquid/solid separation is required. This involves the filtration device described in Section 9.4.2 and is outline in Section 14.2.3 to 14.2.8.

14.2.3 Pre-weigh the container that will receive the filtrate.

14.2.4 Assemble the filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure. Acid wash the filter if evaluating the mobility of metals (see Section 9.4). NOTE: Acid washed filters may be used for all nonvolatile extractions even when metals are not of concern.

14.2.5 Weigh out a sub-sample of the waste (100 gram minimum) and record the weight. If the waste contains <0.5% dry solids (Section 14.1.2), the liquid portion of the waste, after filtration, is defined as the TCLP extract. Therefore, enough of the sample should be filtered so that the amount of the filtered liquid still support all of the analyses that the amount of filtered liquid will support all of the analyses required of the TCLP extract. For wastes containing >0.5% dry solids (Section 14.1.1 or 14.1.2), use the percent solids information obtained in Section 14.1.1 to determine the optimum sample size (100 gram minimum) for filtration. Enough solids should be generated by filtration to support the analyses to be performed on the TCLP extract.

14.2.6 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration. Use centrifugation only as an aid to filtration. If the waste is centrifuged, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the same filtration system.

14.2.7 Quantitatively transfer the waste sample (liquid and solid phases) to the filter holder (see Section 9.3.2). Spread the waste sample evenly over the surface of the filter. If filtration of the waste at 4 °C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering. NOTE: If waste

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material (>1% of the original sample weight) has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight in Section 14.2.5, to determine the weight of the waste sample that will be filtered. Gradually apply vacuum or gentle pressure of 1 – 10 psi, until air or pressurizing gas moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, it the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psi increment. When the pressurizing gas begins to move through the filter, or when the liquid flow has ceased at 50 psi (i.e., filtration does not result in any additional filtrate within a 2 minute period), stop the filtration. NOTE: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

14.2.8 The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase. Weigh the filtrate. The liquid phase may now be either analyzed (See Section 14.2.12) or stored at 4 °C until time of analysis. NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying vacuum or pressure filtration, as outlined in Section 14.2.7, this material may not filter. If this is the case, the material within the filtration device is defined as a solid and is carried through the extraction as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.

14.2.9 If the waste contains <0.5% dry solids (see Section 14.1.2), proceed to section 14.2.13. If the waste contains >0.5% dry solids (see Section 14.1.1 or 14.1.2), and if particle size reduction of the solid was needed in Section 14.1.3, proceed to section 14.2.10. If the waste as received passes a 9.5 mm sieve, quantitatively transfer the solid material into the extractor bottle along with the filter used to separate the initial liquid from the solid phase, and proceed to Section 14.2.11.

14.2.10 Prepare the solid portion of the waste for extraction by crushing, cutting, or grinding the waste to a surface area or particle size as described in Section 14.1.3. When the surface area or particle size has been appropriately altered, quantitatively transfer the solid material into an extractor bottle. Include the filter used to separate the initial liquid from the solid phase. NOTE: Sieving of the waste is not normally required. Surface area requirements are meant for filamentous (e.g., paper, cloth) and similar waste materials. Actual measurement of surface area is not recommended. If sieving is necessary, a Teflon coated sieve should be used to avoid contamination of the sample.

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14.2.11 Determine the amount of extraction fluid to add to the extractor vessel as follows:

$$\text{Weight of Extraction fluid} = \frac{20 \times \text{percent solids (Section 14.1.1)} \times \text{weight of waste filtered (Section 14.2.5 or 14.2.7)}}{100}$$

Slowly add this amount of appropriate fluid (see Section 14.1.4) to the extractor vessel. Close the extractor bottle tightly (it is recommended that Teflon tape be used to ensure a tight seal), secure in rotary agitation device, and rotate at 30 +/- 2 rpm for 18 +/- 2 hours. Ambient temperature (i.e. temperature of room in which extraction takes place) shall be maintained at 23 +/- 2 °C during the extraction period. NOTE: As agitation continues, pressure may build up within the extractor bottle for some types of wastes (e.g., limed or calcium carbonate containing waste may evolve gases such as carbon dioxide). To relieve excess pressure, the extractor bottle may be periodically opened (e.g., after 15 minutes, 30 minutes, and 1 hour) and vented into a hood.

14.2.12 Following the 18 +/- 2 hour extraction, separate the material in the extractor vessel into its component liquid and solid phases by filtering through a new glass fiber filter, as outlined in Section 14.2.7. For final filtration of the TCLP extract, the glass fiber filter may be changed, if necessary, to facilitate filtration. Filter(s) shall be acid-washed (see Section 9.4) if evaluating the mobility of metals.

14.2.13 Prepare the TCLP extract as follows:

14.2.13.1 If the waste contained no initial liquid phase, the filtered liquid material obtained from Section 14.2.12 is defined as the TCLP extract. Proceed to Section 14.2.14.

14.2.13.2 If compatible (e.g., multiple phases will not result on combination), combine the filtered liquid resulting form Section 14.2.12 with the initial liquid phase of the waste obtained in Section 14.2.7. This combined liquid is defined as the TCLP extract. Proceed to section 14.2.14.

14.2.13.3 If the initial liquid phase of the waste, as obtained from Section 14.2.7, is not or may not be compatible with the filtered liquid resulting from Section 14.2.12, do not combine these liquids. Analyze these liquids, collectively defined as the TCLP extract, and combine the results mathematically, as described in Section 14.2.14.

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14.2.14 Following collection of the TCLP extract, the pH of the extract should be recorded. Immediately preserve the extract for analysis. Metals aliquots must be acidified with nitric acid to pH <2. If precipitation is observed upon addition of nitric acid to a small amount of the extract, then the remaining portion of the extract for metals analyses shall not be acidified and the extract shall be analyzed as soon as possible. All other aliquots must be stored under refrigeration (4 °C) until analyzed. The TCLP extract shall be prepared and analyzed according to appropriate analytical methods. TCLP extracts to be analyzed for metals shall be acid digested except in those instances where digestion causes loss of metallic analytes. If an analysis of the undigested extract shows that the concentration of any regulated metallic analyte exceed the regulatory level, then the waste is hazardous and digestion of the extract is not necessary. However, data on undigested extracts alone cannot be used to demonstrate that the waste is not hazardous. If the individual phases are to be analyzed separately, determine the volume of the individual phases (to +/- 0.5%), conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$\text{Final Analyte Concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

Where:
V_1 = The volume of the first phase (L).
C_1 = The concentration of the analyte of concern in the first phase (mg/L).
V_2 = The volume of the second phase (L).
C_2 = The concentration of the analyte of concern in the second phase (mg/L).

14.2.15 Compare the analyte concentrations in the TCLP extract with the levels identified in the appropriate regulations. Refer to Section 12 for quality assurance requirements.

14.3 Procedure When Volatiles are Involved- Use the ZHE device to obtain TCLP extract for analysis of volatile compounds only. Extract resulting form the use of the ZHE shall not be used to evaluate the mobility of nonvolatile analytes (e.g., metals, pesticides, etc.). The ZHE device has approximately a 500 mL internal capacity. The ZHE can thus accommodate a maximum of 25 grams of solid (defined as that fraction of a sample from which no additional liquid may be forced out by an applied pressure of 50 psi), due to the need to add an amount of extraction fluid equal to 20 times the weight of the solid phase. Charge the ZHE with sample only once and do not open the device until the final extract (of the solid) has been collected. Repeated filling of the ZHE to obtain 25 grams of solid is not permitted. Do not allow the waste, the initial liquid phase, or the extract to be exposed to the atmosphere for any more time than is absolutely necessary. Any manipulation of these materials should be done when cold (4 °C) to minimize loss of volatiles.

14.3.1 Pre-weigh the (evacuated) filtrate collection container (See Section 9.6) and set aside. If using a TEDLAR bag, express all liquid from the

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ZHE device into the bag, whether for the initial or final liquid/solid separation, and take an aliquot from the liquid in the bag for analysis. The containers listed in section 9.6 are recommended for use under the conditions stated in Section 9.6.1 – 9.6.3.

14.3.2 Place the ZHE piston within the body of the ZHE (it may be helpful first to moisten the piston O-rings slightly with extraction fluid). Adjust the piston within the ZHE body to a height that will minimize the distance the piston will have to move once the ZHE is charged with sample (based upon sample size requirements determined from Section 14.3, Section 14.1.1 and/or 14.1.2). Secure the gas inlet/outlet flange (bottom flange) onto the ZHE body in accordance with the manufacturer's instructions. Secure the glass fiber filter between the support screens and set aside. Set liquid inlet/outlet flange (top flange) aside.

14.3.3 If the waste is 100% solid (see Section 14.1.1), weigh out a sub-sample (25 gram maximum) of the waste, record weight, and proceed to Section 14.3.5.

14.3.4 If the waste contains <0.5% dry solids (Section 14.1.2), the liquid portion of waste, after filtration, is defined as the TCLP extract. Filter enough of the sample so that the amount of filtered liquid will support all of the volatile analyses required. For wastes containing ≥ 0.5% dry solids (Sections 14.1.1 and/or 14.1.2), use the percent solids information obtained in Section 14.1.1 to determine the optimum sample size to charge into the ZHE. The recommended sample size is as follows:

14.3.4.1 For wastes containing <5% solids (see Section 14.1.1), weigh out a 500 gram sub-sample of waste and record the weight.

14.3.4.2 For wastes containing ≥ 5% solids (see Section 14.1.1), determine the amount of waste to charge into the ZHE as follows:

$$\text{Weight of waste to charge ZHE} = \frac{25}{\text{percent solids (Section 14.1.1)}} \times 100$$

Weigh out a sub-sample of the waste of the appropriate size and record the weight.

14.3.5 If particle size reduction of the solid portion of the waste was required in Section 14.1.3, proceed to Section 14.3.6. If particle size reduction was not required in Section 14.1.3, proceed to Section 14.3.7.

14.3.6 Prepare the waste for extraction by crushing, cutting, or grinding the solid portion of the waste to a surface area or particle size as described in Section 14.1.3. Wastes and appropriate reduction equipment should be refrigerated, if possible, to 4 °C prior to particle size reduction. The means used to effect particle size reduction must not generate heat in

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and of itself. If reduction of the solid phase of the waste is necessary, exposure of the waste to the atmosphere should be avoided to the extent possible. NOTE: Sieving of the waste is not recommended due to the possibility that volatiles may be lost. The use of an appropriately graduated ruler is recommended as an acceptable alternative. Surface area requirements are meant for filamentous (e.g., paper, cloth) and similar waste materials. Actual measurement of surface area is not recommended. When the surface area or particle size has been appropriately altered, proceed to Section 14.3.7.

14.3.7 Waste slurries need not be allowed to stand to permit the solid phase to settle. Do not centrifuge wastes prior to filtration.

14.3.8 Quantitatively transfer the entire sample (liquid and solid phases) quickly to the ZHE. Secure the filter and support screen onto the top flange of the device and secure the top flange to the ZHE body in accordance with the manufacturer's instructions. Tighten all ZHE fitting and place the device in the vertical position (gas inlet/outlet flange on the bottom). Do not attach the extract collection device to the top plate. NOTE: If waste material (>1% of original sample weight) has obviously adhered to the container used to transfer the sample to the ZHE, determine the weight of this residue and subtract it from the sample weight determined in Section 7.3.4 to determine the weight of the waste sample that will be filtered. Attach a gas line to the gas inlet/outlet valve (bottom flange) and, with the liquid inlet/outlet valve (top flange) open, begin applying gentle pressure of 1-10 psi (or more is necessary) to force all headspace slowly out of the ZHE device into a hood. At the first appearance of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue pressure. If filtration of the waste at 4 °C reduces the amount of expressed liquid over what should be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering. If the waste is 100% solid (see Section 14.1.1), slowly increase the pressure to a maximum of 50 psi to force most of the headspace out of the device and proceed to Section 14.3.12.

14.3.9 Attach the evacuated pre-weighed filtrate collection container to the liquid inlet/outlet valve and open the valve. Begin applying gentle pressure of 1 – 10 psi to force the liquid phase of the sample into the filtrate collection container. If no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psi increment. When liquid flow has ceased such that continued pressure filtration at 50 psi does not result in any additional filtrate within a 2 minute period, stop the filtration. Close the liquid inlet/outlet valve, discontinue pressure to the piston, and disconnect and weigh the filtrate collection container. NOTE: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

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14.3.10 The material in the ZHE is defined as the solid phase of the waste and the filtrate is defined as the liquid phase. NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying pressure filtration, this material will not filter. If this is the case, the material within the filtration device is defined as a solid and is carried through the TCLP extraction as a solid. If the original waste contained <0.5% dry solids (see Section 14.1.2), this filtrate is defined as the TCLP extract and is analyzed directly. Proceed to section 14.3.15.

14.3.11 The liquid phase may now be either analyzed immediately (see Sections 14.3.13 through 14.3.15) or stored at 4 °C under minimal headspace conditions until time of analysis. Determine the weight of extraction fluid #1 to add to the ZHE as follows:

$$\text{Weight of extraction fluid} = \frac{20 \times \text{percent solids (Section 14.1.1)} \times \text{weight of waste filtered (Section 14.3.4 or 14.3.8}}{100}$$

14.3.12 The following Sections detail how to add the appropriate amount of extraction fluid to the solid material within the ZHE and agitation of the ZHE vessel. Extraction fluid #1 is used in all cases.

14.3.12.1 With the ZHE in the vertical position, attach a line from the extraction fluid reservoir to the liquid inlet/outlet valve. The line used shall contain fresh extraction fluid and should be preflushed with fluid to eliminate any air pockets in the line. Release gas pressure on the ZHE piston (from the gas inlet/outlet valve), open the liquid inlet/outlet valve, and begin transferring extraction fluid (by pumping or similar means) into the ZHE. Continue pumping extraction fluid into the ZHE until the appropriate amount of fluid has been introduced into the device.

14.3.12.2 After the extraction fluid has been added, immediately close the liquid inlet/outlet valve and disconnect the extraction fluid line. Check the ZHE to ensure that all valves are in their closed positions. Manually rotate the device in an end-over-end fashion 2 or 3 times. Reposition the ZHE in the vertical position with the liquid inlet/outlet valve on top. Pressurize the ZHE to 5-10 psi (if necessary) and slowly open the liquid inlet/outlet valve to bleed out any headspace (into a hood) that may have been introduced due to the addition of extraction fluid. This bleeding shall be done quickly and shall be stopped at the first appearance of liquid from the valve. Re-pressurize the ZHE with 5-10 psi and check all ZHE fittings to ensure that they are closed.

14.3.12.3 Place the ZHE in the rotary agitation apparatus (if it is not already there) and rotate at 30 +/- 2 rpm for 18 +/- 2 hours. Ambient temperature (i.e., temperature of room in which

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extraction occurs) shall be maintained at 23 +/- 2 °C during agitation.

14.3.13 Following the 18 +/- 2 hour agitation period, check the pressure behind the ZHE piston by quickly opening and closing the gas inlet/outlet valve and noting the escape of gas. If the pressure has not been maintained (i.e., no gas release observed), the device is leaking. Check the ZHE for leaking as specified in Section 9.2.1, and perform the extraction again with a new sample of waste. If the pressure within the device has been maintained, the material in the extractor vessel is once again separated into its component liquid and solid phases. If the waste contained an initial liquid phase, the liquid may be filtered directly into the same filtrate collection container (i.e., TEDLAR bag) holding the initial liquid phase of the waste. A separate filtrate collection container must be used if combining would create multiple phases, or there is not enough volume left within the filtrate collection container. Filter through the glass fiber filter, using the ZHE device as discussed in Section 14.3.9. All extract shall be filtered and collected if the TEDLAR bag is used, if the extract is multiphasic, or if the waste contained an initial liquid phase (see Section 9.6 and 14.3.1). NOTE: An in-line glass fiber filter may be used to filter the material within the ZHE if it is suspected that the glass fiber filter has been ruptured.

14.3.14 If the original waste contained no initial liquid phase, the filtered liquid material obtained form Section 14.3.13 is defined as the TCLP extract. If the waste contained an initial liquid phase, the filtered liquid material obtained form Section 14.3.13 and the initial liquid phase (Section 14.3.9) are collectively defined as the TCLP extract.

14.3.15 Following the collection of the TCLP extract, immediately prepare the extract for analysis and store with minimal headspace at 4 °C until analyzed. Analyze the TCLP extract according to the appropriate analytical methods. If the individual phases are to be analyzed separately (i.e., are not miscible), determine the volume of the individual phases (to 0.5%), conduct the appropriate analyses, and combine the results mathematically by using a simple volume weighted average:

$$\text{Final Analyte Concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

Where:
V_1 = The volume of the first phase (L).
C_1 = The concentration of the analyte of concern in the first phase (mg/L).
V_2 = The volume of the second phase (L).
C_2 = The concentration of the analyte of concern in the second phase (mg/L).

14.3.16 Compare the analyte concentrations in the TCLP extract with the levels identified in the appropriate regulations.

15.0 DATA ANALYSIS AND CALCULATIONS

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See the appropriate determinative method.

16.0 METHODS PERFORMANCE

16.1 Two ruggedness studies have been performed to determine the effect of various perturbations on specific elements of the TCLP protocol. Ruggedness testing determines the sensitivity of small procedural variations which might be expected to occur during routine laboratory application.

16.1.1 Metals – The following conditions were used when leaching a waste for metals analysis:

Varying Conditions	
Liquid/Solid ratio	19:1 vs. 21:1
Extraction Time	16 hours vs. 18 hours
Headspace	20% vs. 60%
Buffer #2 acidity	190 meq vs. 210 meq
Acid-washed filters	Yes vs. no
Filter type	0.7 um glass fiber vs. 0.45 um vs. polycarbonate
Bottle type	Borosilicate vs. flint glass

Of the seven method variations examined, acidity of the extraction fluid had the greatest impact on the results. Four of 13 metals from an API separator sludge/electroplating waste (API/EW) mixture and two of three metals from an ammonia lime still bottom waste were extracted at higher levels by the more acidic buffer. Because of the sensitivity to pH changes, the method requires that the extraction fluids be prepared so that the final pH is within +/- 0.05 units as specified.

16.1.2 Volatile Organic Compounds – The following conditions were used when leaching a waste for VOC analysis:

Varying Conditions	
Liquid/Solid ratio	19:1 vs. 12:1
Headspace	0% vs. 5%
Buffer #1 acidity	60 meq vs. 80 meq
Method of storing extract	Syringe vs. Tedlar bag
Aliquotting	Yes vs. no
Pressure behind piston	0 psi vs. 20 psi

None of the parameters had a significant effect on the results of the ruggedness test.

16.2 Precision- Many TCLP precision (reproducibility) studies have been performed, and have shown that, in general, the precision of the TCLP is comparable to or exceeds that of the EP toxicity test and that method precision is adequate. One of the more significant contributions to poor precision appears to be related to sample homogeneity and inter-laboratory variation (due to the nature of the waste materials).

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16.2.1 Metals – The results of a multi-laboratory study indicate that a single analysis of a waste may not be adequate for waste characterization and identification requirements.

16.2.2 Semi-Volatile Organic Compounds – Single laboratory precision was excellent with greater than 90 percent of the results exhibiting an RSD less than 25 percent. Over 85 percent of all individual compounds in the multi-laboratory study fell in the RSD range of 20 – 120 percent. Both studies concluded that the TCLP provides adequate precision. It was also determined that the high acetate content of the extraction fluid did not present problems (i.e., column degradation of the gas chromatograph) for the analytical columns used.

16.2.3 Volatile Organic Compounds – Eleven laboratories participated in a collaborative study of the use of the ZHE with two waste types which were fortified with a mixture of VOCs. Precision results for VOCs tend to occur over a considerable range. However, the range and mean RSD compared very closely to the same collaborative study metals results. Blackburn and Snow concluded that at the 95% level of significance: 1) recoveries among laboratories were statistically similar, 2) recoveries did not vary significantly between the two sample types, and 3) each laboratory showed the same pattern of recovery for each of the two samples.

17.0 POLLUTION PREVENTION

17.1 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. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the EPA recommends recycling as the next best option.

17.2 Quantity of the chemicals purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

17.3 For information about pollution prevention that may be applicable to laboratories and research institutions, consult "Less is Better: Laboratory Chemical Management for Waste Reduction," available from the American Chemical Society's Department of Government Regulations and Science Policy, 1155 16th Street N.W. Washington D.C. 20036,(202)872-4477.

18.0 Data Assessment, Acceptance and Corrective Action:

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The following corrective actions should be instituted if deemed appropriate based on results obtained from the selected determinative method:

18.1 Potential sources of contamination should be investigated and eliminated and affected samples should be re-analyzed.

18.2 Poor matrix spike recoveries could be a function of matrix interference. If sample history does not indicate a matrix related problem, the affected samples should be re-extracted and re-analyzed to confirm matrix interference.

18.3 A corrective action form should be initiated by the individual that discovered the analytical problem. The corrective action form should be filled out by the appropriate personnel with documentation of the procedures to minimize reoccurrence of the problem. Finalized corrective action forms are filed with the laboratory quality assurance manager

19.0 WASTE MANAGEMENT

19.1 The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Excess reagents, samples, and method process wastes should be characterized and disposed of in applicable manner. The EPA urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any waste discharge permit and regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult the "Waste Management Manual for Laboratory Personnel," available from the American Chemical Society at the address listed in Sect. 14.3.

20.0 REFERENCES

SW-846 method 1311, Revision 00 July 1992

NELAC Quality Systems Chapter 5, June 5, 2003

21.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

N/A

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TCLP AND SPLP PREP LOGSHEET

Date:_____
Analyst:_____

SOP 3.2.8	SOP 3.2.74
Standard Method 1311	Standard Method 1312
Rev. B 2/16/06	Rev. A 2/16/06

Solution Labels or Tags			
1N HCL		Fluid 1	
Glacial Acetic Acid		Fluid 1 pH	
1N HNO3		Fluid 2	
1N NaOH		Fluid 2 pH	
H2SO4			

Parl A:Percent Solids Determination

Sample #	~%Solids = 100%	Filter Wt	Container Wt	Sample Wt	Liquid Phase	Solid Phase (wet)	%Solids
	yes = go to Part B	g	g	g	g	g	<0.5% = Treat as TCLP liquid extract
	no = Continue --->						>0.5% = Determine Extraction Fluid

Part B: Extraction Determination (For SPLP: Fluid #1=East of Mississippi/Fluid#2 West of Mississippi)

Sample #	Sample Wt	pH1 of Sample*	pH2 of Sample**	Fluid 1***	Fluid 2***	pH
	g	pH1 <5.0 use Fluid 1	pH2 <5.0 Use Fluid 1	g	g	
		pH1 >5.0**-->	pH2 >5.0 Use Fluid 2			

Part C: Agitation Record

Agitation Start Time =
Agitation End Time =
Total Agitation Time =
Room Temp Start =
Room Temp End =

TCLP Rotator Check
Of Rotations p/Min =

Percent dry Solis =
Weight of Solid Phase
* 5g sample + 96.5 ml DI water
** to pH1 Solution above 3.5 ml 1N HCL heat & Stir to 50C hold and cool
*** Weight of Fluid to use =
(20*%solids*solid phase wt)/100

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PAGE:	1 OF 11

DOCUMENT HISTORY AND APPROVAL PAGE

TITLE:	**Total Organic Carbon in Solid Matrices- EPA Method 9060 (Modified) and Lloyd Kahn (1988)**
DEPARTMENTS AFFECTED:	**Organic Chemistry Section**

ISSUE DATE:	REVISION CODE:	SOP #:	REVISION CHANGES:
01/10/08	-	3.2.83	Create an SOP for TOC In solid matrices

DOCUMENT APPROVAL		
Originator: James Galasyn, Ph.D.	**Signature/Date:** J Galasyn 011508	**Title Chemistry Lab Manager**
Approved by: Joseph Casarin	Joseph Casarin 011508	**Organics Supervisor**
Reviewed by: Pamela Doughty	Pamela Doughty 1/15/08	**Total Quality Manager**

NEL	STANDARD OPERATING PROCEDURE		
Total Organic Carbon in Solid matrices - EPA method 9060 (modified) and Lloyd Kahn (1988)			
SOP# 3.2.83	REV# -	EFFECTIVE 01/10/08	PAGE 2 OF 11
WRITTEN BY: Galasyn	DEPARTMENTS AFFECTED: Organic Chemistry Section		

1.0 Test Method:

This standard operating procedure (SOP) is for Total Organic Carbon by EPA method 9060 (modified) and Lloyd Kahn (1988).

2.0 Applicable Matrix or Matrices

This method measures the organic carbon in soils, sludge, sediments, and other solid matrices.

3.0 Detection Limit

The laboratory-reporting limit for this analysis is 0.1% TOC in all solid samples. Annual method detection limit studies are performed and all data is stored in the Quality Assurance Manager's office.

4.0 Scope and Application

4.1 This standard operating procedure describes the process of analyzing samples at Northeast Laboratory Services for Total Organic Carbon. This SOP is applicable for the above mentioned matrices.

4.2 The Hach IL500 TOC analyzer is equipped with a HSC1300 Solids module to determine the total carbon content in samples. This module is a horizontal furnace heated by electrical resistance. Samples are oxidized and the quantity of resulting carbon dioxide is measured. Oxygen is both the oxidizing agent and carrier gas for the system.

4.3 The sample is introduced manually into the HSC1300 solids module in a ceramic combustion boat.

4.4 To determine the TOC concentration, 1:1 nitric acid is added to the solid sample in the combustion boat to remove the inorganic carbon. The acid is added drop by drop until no further reaction is observed (no gas formation). The combustion boat is then left to dry at least thirty minutes in a drying oven at 105 degrees F, followed by analysis. For total carbon, addition of this nitric acid is not required.

4.5 For the Lloyd Kahn method, 1:1 phosphoric acid is used as a substitute for the nitric acid noted in step 4.4.

5.0 Summary of the Method

Organic carbon in a sample is converted to carbon dioxide by combustion at a high temperature (1200 degrees C). The amount of carbon dioxide is directly proportional to the concentration of carbonaceous material in the sample.

6.0 Definitions:

6.1 Method Blank (BLK): An aliquot of muffled sand that is treated exactly as a sample including exposure to all glassware, equipment, solvents and reagents. The BLK is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents or the instruments.

6.2 Initial Calibration Verification (ICV): Quality control sample prepared from a source separate than that of the calibration standards. The ICV must be analyzed immediately following the initial calibration. If only a single source of the standard calcium carbonate is available, a mass in the lower range of the curve or the upper range can be used as the ICV. The percent difference (%D) of the ICV must be within +/- 10% of the stated value. The LCS and LCSD QC samples, which are prepared from a second source or lot number if available, can serve as the ICV.

6.3 Laboratory Matrix Spike Matrix Spike Duplicate Sample (MS/MSD): An aliquot of an environmental sample to which known quantities of the method analytes are added in the lab. The MS is analyzed exactly like a sample and its purpose is to determine whether the sample matrix contributes bias to the analytical results. Recoveries of the MS/MSD must be within 75%-125% and the RPD within +/- 20%. MS/MSD's are analyzed at the request of the client. In the event that the client does not specifically request a MS/MSD QC pair, NEL substitutes the use of a second source LCS/LCSD pair (one set every 20 samples) to monitor the batch accuracy and precision.

6.4 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. MDL studies are conducted annually.

6.5 Duplicate (DUP): A replicate aliquot % of sample carried through the identical analytical process. The %D is measured to determine precision. The %D must be less than 20%. As is the case with MS/MSD QC samples, laboratory duplicates are analyzed at the request of the client.

6.6 Continuing Calibration Verification (CCV): Analytical standard containing all target analytes used to evaluate the analytical system in respect to a defined set of method criteria. CCV's are analyzed following the initial calibration and after every twenty samples. Recovery of the CCV must be with +/- 10% of the given value. CCV source is the same as the calibration curve.

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7.0 Interferences:

7.1 Samples not preserved correctly will cause a negative bias.

7.2 High levels of chloride or chlorine will interfere and may cause a negative bias.

8.0 Safety

8.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health risk. Please refer to the Northeast Laboratory Services Chemical Hygiene Plan as well as appropriate MSDS forms for further information.

8.2 Good laboratory practices should always be used. Protective eyewear and gloves must be worn.

9.0 Equipment and Supplies

9.1 Hach IL500 TOC Analysis System

9.2 Hach ceramic boats

9.3 Calcium carbonate, certified ACS grade, FW = 100.09, 12% C.

9.4 blank sand, baked for a minimum of 8.0 hours

10.0 Reagents and Standards

10.1 Calcium carbonate, certified ACS grade, FW = 100.09, 12% carbon. Use a different vendor of this neat powder for second source confirmation.

10.2 Sodium Persulfate Solution, 80 g/l-Prepare by dissolving 40 g of reagent grade Potassium Persulfate ($Na_2S_2O_8$) in 500 ml of DI water. Add 10 ml of 1M sulfuric acid and mix well. Bring to a final volume of 1.0 liter. Store in cool dark location.

10.3 DI Water-Draw daily deionized water from the NEL system and purge for a minimum of 15 minutes using ultra high purity nitrogen prior to use..

10.4 Nitrogen, UHP

10.5 Oxygen, UHP

10.6 1:1 nitric acid

10.7 1:1 phosphoric acid

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11.0 Preservation, Shipment and Storage

11.1 Sampling and storage of samples in glass bottles is preferable. Sampling and storage in plastic bottles such as conventional polyethylene is permissible if it is established that the containers do not contribute contaminating organics to the samples.

11.2 All solid samples must be analyzed within 28 days from the date of collection.

12.0 Quality Assurance/Quality Control

12.1 Method Blank: Analyzed immediately following initial calibration and following every CCV. Blank results must be below the reporting limit of 0.1 %C.

12.2 Continuing Calibration Verification: Analyzed after the initial calibration and every 20 samples and must be within +/- 10% of the stated value. Use a mass of calcium carbonate that is near the midpoint of the calibration curve.

12.3 Laboratory Control Sample/Laboratory Control Sample Duplicate pair: Analyzed every 20 samples; recoveries must be 80%-120% with a %RPD value less than or equal to 20%.

12.4 Matrix Spike/Matrix Spike Duplicate: Analyzed at the request of the client. Percent recoveries must be 75%-125%, and % RPD values must be below 20%.

12.5 Sample Duplicate: Analyzed every twenty samples at the request of the client.

13.0 Calibration and Standardization

13.1 The Hach IL500 TOC instrument is calibrated by analyzing a minimum of 5 standards of varying masses (typically 2.00 mg to 100 mg), with the low point near the reporting limit and the high point at the upper reporting limit of the method.

13.2 The correlation coefficient must be 0.995 or better.

13.3 The procedure for calibrating this instrument follows the manufacturer's guidelines. New initial calibration curves must be analyzed if a CCV does not meet criteria even after routine maintenance (checking gas pressures, replacing halogen trap scrubber copper and brass wool, etc).

14.0 Procedures

14.1 Open ultrahigh purity nitrogen tank, make sure pressure is at 75 psi, with a minimum tank level of 500 psi. Open the ultrahigh purity oxygen tank, making sure the pressure us at 55 psi, with a minimum tank level of 500 psi. Change the tanks when necessary.

14.2 Disconnect the red water autosampler cord from the sampler/HT port and replace with the cord from the soil module. Connect the pump out line originating

at the rear of the soil module to the pump/out fitting on the TOC instrument. Connect the soil module analyt line to the analyt fitting on the TOC instrument.

14.3 Turn the soil module on.

14.4 Go to the instrument computer and using the dropdown menus go to program files/TOCTN/Omnitoc/config. This will bring you to the autosampler option. Uncheck the autosampler box and click on "create." This will open up the OmniTOC software. Do not initialize the analyzer at this point. Go to "configuration" and check the external solid module box.

14.5 Load the soil method and then initialize the analyzer. It will take the soil module approximately one hour to reach the operating temperature of 1200 degrees C.

14.6 After initialization, follow manufacturers instructions to start a batch with the following sequence:

Blank
CCV
LCS
LCSD
Blank

14.7 If this QC passes, start a sample sequence, with continuing QC every 20 samples, as well as a closing CCV.

15.0 Calculations

15.1 $$\text{RPD \%} = \left| \frac{A-B}{(A+B)/2} \right| * 100$$

A = Sample
B = Sample Dup

15.2 $$\text{MS Recovery \%} = \left| \frac{MS - A}{MSTV} \right| * 100$$

MS = Matrix spiked sample
MSTV = Matrix spike true value
A = Sample concentration

15.4 $$\text{LCS Recovery \%:} \quad \frac{LCS}{LCSTV} * 100$$

LCSTV = LCS true value

16.0 Method Performance

The 2008 MDL study conducted at NEL on 01/14/08 yielded a MDL of 620 ppm (0.062% C) with seven replicates analyzed at a concentration of 1200 ppm (2.00 mg of calcium carbonate diluted to 200 mg with muffled sand). The 2008 reporting level for this method is 0.1%).

17.0 Pollution Prevention

Please refer to the Northeast Laboratory Services Spill Prevention Control and Contingency Plan as well as the Chemical Hygiene plan for overall laboratory pollution prevention.

18.0 Data Assessment, Acceptance Criteria and Corrective Action

18.1 In the event that any measurement is out of control and analytical data needs to be presented to the client, the measurement is reported to the immediate supervisor and then to QA. Any corrective action undertaken is documented on a corrective action form (detailing the client information, problem, investigative findings and solution). This form is filed in the QA Manager's Office.

18.2 If sample results are outside the calibration range, the sample will have to be diluted. Dilutions for this method involve the use of the muffled organic free sand previously referenced in this SOP. The diluted result should fall within the upper half of the calibration curve.

18.3 The following corrective actions should be instituted if deemed appropriate based on results obtained from the selected determinative method:

1) Potential sources of contamination should be investigated and eliminated.

2) Poor matrix spike recoveries could be a function of matrix interference. If sample history does not indicate a matrix related problem, the affected samples should be re-extracted and re-analyzed to confirm matrix interference.

As stated, results are reported by a modified 9060A and the report noted as such.

19.0 Waste Management

Any and all waste generated, through the above procedures, is handled according to the Chemical Hygiene and Hazardous Waste Management and Spill Prevention Control and Contingency Plan.

20.0 References

NELAC Quality Systems Chapter 5, June 5, 2003

SW 846 3rd Ed. Chapter 5, EPA 9060A

USEPA Methods for Chemical analysis of Water and Wastes, EPA 600/4-79-020 March 1979

Standard Methods for the Analysis of Water and Wastes, 20th Ed., 1998, 5310C

Hach-Lachat, "The Carbon Content in Solid Matrices," David Nelson and Carlos Williams

Determination of Total Organic Carbon in Sediment (Lloyd Kahn method), July 27, 1988, USEPA Region II, Environmental Services Division, Edison, NJ

21.0 Tables Diagrams, Flowcharts and Validation Data

See Attached.

Medium/Matrix	Soil/solid/sludge
Sampling SOP	NA
Analytical Parameter	Total Organic Carbon
Concentration Level	Calibration Curve Range: 0.1%-50%
Analytical Method/SOP Reference	EPA 9060/Lloyd Kahn method
Laboratory Name	NEL
No. of Sample Locations	

Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**
Method Blank	One per batch of 20 or fewer samples	TOC less than the reporting limit of 0.1%	1) Investigate source of contamination. 2) Re-prepare and reanalyze all associated samples if sample concentration ≥ QL	Analyst/ Supervisor
Reagent Blank	NA	NA	NA	NA
Storage Blank	NA	NA	NA	NA
Instrument Blank	As needed after sample with concentration exceeding the calibration curve limit	NA	NA	NA
Laboratory Duplicate	As requested by the client	RPD ≤ 20%	) Investigate source of contamination. 2) Re-prepare and reanalyze	Analyst/ Supervisor
Laboratory Matrix Spike	One per matrix or batch of 20 or fewer samples, whichever is more frequent, as requested by the client	Recovery 75% – 125%	Note on sample report	Analyst/ Supervisor
Matrix Spike Duplicate	One per matrix or batch of 20 or fewer samples, whichever is more frequent ,a s requested by the client	Recovery 75% - 125% ≤ 20 % RPD between MS and MSD	Note on sample report	Analyst/ Supervisor
LCS/LCSD	One set per batch of 20 or fewer samples	Recovery 80% - 120%	Reanalyze all affected samples.	Analyst/ Supervisor
Replicates	Every QC and field sample	CV ≤ 20%	Reanalyze fresh sample aliquot to confirm matrix interference	Analyst/ Supervisor
Initial Calibration	Before sample analysis. Minimum of 5 calibration standards	Correlation coefficient > 0.995	1) Investigate source of problem. 2) Re-prepare and or re-analyze standards as required.	Analyst/ Supervisor

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Medium/Matrix	Soil/solid/sludge
Sampling SOP	NA
Analytical Parameter	Total Organic Carbon
Concentration Level	Calibration Curve Range: 0.1%-50%
Analytical Method/SOP Reference	EPA 9060/Lloyd Kahn method
Laboratory Name	NEL
No. of Sample Locations	

Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**
Continuing Calibration	Daily before sample analysis and after every 20 samples	1. CCV +/- 10% D from initial calibration curve	Investigate source of problem. Re-prepare and or re-analyze standards as required Re-calibrate instrument	Analyst/ Supervisor

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Maintenance Schedule

Component	Maintenance Task	Interval
Analyzer	Cleaning Cleaning of the collecting trays and reagent flasks Check all exterior tube connections and threaded fasteners for tightness	Weekly Weekly and after filling Monthly
Water traps	Check gas flow	Daily
Halogen trap	Check brass wool for discoloring	Daily
	Replace exhausted wool	3 months
Peristaltic pump	Check for leaks	Daily
	Replace pump tubing	As needed
Syringe Pump	Check for leaks	weekly
	Clean the metering syringe	Every six months
Nitrogen and oxygen gas tanks	Check to ensure gas P $\geq$ 500 psi	daily

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DOCUMENT HISTORY AND APPROVAL PAGE

TITLE:	**Volatile Organic Compounds By Gas Chromatography/Mass Spectroscopy (GC/MS)-EPA Method 8260B**
DEPARTMENTS AFFECTED:	**Organics**

ISSUE DATE:	REVISION CODE:	SOP #:	REVISION CHANGES:
10/5/01	D	3.3.13	Update to be compliant with NELAC requirements
03/26/02	E	3.3.13	Edit and correct for low level 8260B analysis (1-50ppb)
06/07/02	F	3.3.13	Add Routine Maintenance to Section 18
01/02/03	G	3.3.13	Corrected typographical errors and added tables for preparation of standards
05/10/04	H	3.3.13	Edits for low-level soils, typographical errors, annual review
01/19/06	I	3.3.13	Annual Review & Update
6/19/07	J	3.3.13	Annual Review & Update
01/23/08	K	3.3.13	Annual Review & Update
06/30/08	L	3.3.13	Update PQL Table

DOCUMENT APPROVAL		
Originator: Joe Casarin for Abram Patenaude	Signature/Date: Joe Casarin 06/30/08	Title Organics Analyst
Approved by: Joe Casarin	Joe Casarin 06/30/08	Organics Supervisor
Reviewed by: Pamela Doughty	Pamela Doughty 6/30/08	Total Quality Manager

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NEL	STANDARD OPERATING PROCEDURE		
TITLE **Volatile Organic Compounds By Gas Chromatography/Mass Spectroscopy (GC/MS)-EPA Method 8260B**			
SOP# 3.3.13	REV# L	EFFECTIVE 06/30/08	PAGE 2 OF 24
WRITTEN BY: Abram Patenaude	DEPARTMENTS AFFECTED: ORGANICS		

1.0 Test Method:

This standard operating procedure (SOP) was developed to maintain high standards of analytical performance in order to generate data of known and documented quality for our clients. The test method is EPA Method 8260.

2.0 Applicable Matrix or Matrices:

This method is applicable to a variety of solid waste matrices. NEL uses this method for ground & surface waters, sludge, sediment, soils, and solid and waste oil samples.

3.0 Detection Limit:

Please see Table 1 for Standard Compound List and Reporting Limits for this method. Other analytes can be added by client request upon completion of a method detection limit study. Current MDL study results are on file in the QA Manager's office.

4.0 Scope and Application

This SOP is based on Method 8260B (USEPA Test Methods for Evaluating Solid Waste, Dec. 1996). Method 8260B describes the GC/MS procedure for the analysis of volatile organic compounds. The techniques by which compounds may be introduced into the GC/MS system are described in Method 5030 (purge-and-trap of aqueous samples) and Method 5035 (purge-and-trap of solid and waste oil samples). This method is applicable to nearly all types of samples regardless of water content, including ground and surface water, waste solvents, oily wastes, soils and sediments. The compounds amenable to this method are shown in Table 1. A gas chromatograph utilizes a temperature program to separate the purgeables before detection on a mass spectrometer.

5.0 Summary of Method

5.1 Basis Principles - The analytes are introduced into the GC/MS by purge-and-trap techniques (Method 5030 or Method 5035). Upon desorption the volatile compounds are introduced directly to the capillary column of a gas chromatograph equipped with a temperature program to separate the purgeables. The eluted analytes on the gas stream into the ion source of a mass spectrometer. The ionized molecules are focused and separated according to their mass/charge (m/z) ratio by the quadrupole analyzer. The signal is amplified by an electron multiplier and interpreted by the mass spectrometer data system to produce a total ion chromatogram with mass spectra for

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every data point on the chromatogram. The system is an HP 5890 Series II Gas Chromatograph with a 5970 Mass Spectrophotometer or equivalent.

5.2 General Method - The mass spectrometer is calibrated to recognize m/z values in the range of 35-550 amu. Reference spectra and retention times for analytes are obtained by the measurement of calibration standards under the same conditions used for samples. Analytes are quantitated based upon a minimum of a five part calibration. Hewlett-Packard Enviroquant ChemStation Ver. A.03.00 software (or equivalent) is utilized for identification and quantitation of data. The concentration of each identified component is measured by the relative response of the target compound to its respective internal standard.

5.3 The performance of the mass spectrometer is verified by the introduction of a 50ng 4-Bromofluorobenzene (BFB) tune standard. All analyses must occur within 12 hours of the passing BFB analysis time.

5.4 Before any samples can be analyzed a continuing calibration verification (CCV) standard must be analyzed to demonstrate that the instrument is stable relative to the initial calibration curve.

6.0 Definitions

6.1 INTERNAL STANDARD (IS) - Non-target analyte compounds that are similar to the target analytes but are not expected to be found in environmental media (generally, isotopically labeled target analytes are used for this purpose). Internal standards are added to every standard, quality control sample, and field sample at a known concentration prior to analysis. Internal standard responses are used as the basis for quantitation of target analytes.

6.2 SURROGATE ANALYTE (SS) - Non-target analytes compounds that are similar in composition and behavior to the target analytes but are not expected to be found in environmental media (often, isotopically labeled target analytes are used for this purpose) and are added to every standard, quality control sample, and field sample at a known concentration prior to preparation and/or analysis. Surrogate responses are used to evaluate the accuracy of the laboratory's performance of the analytical method in a specific sample matrix.

6.3 STOCK STANDARD SOLUTION - A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.

6.4 PRIMARY DILUTION STANDARD SOLUTION - A solution of several analytes prepared in the laboratory from stock standard solutions and diluted as needed to prepare calibration solutions and other needed analyte solutions.

6.5 CALIBRATION STANDARD (CAL) - A solution prepared from the primary dilution standard solution or stock standard solutions and the internal standards and surrogate analytes. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

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6.6 4-BROMOFLUOROBENZENE STANDARD (BFB)– A standard solution containing BFB in methanol and used to evaluate the performance of the MS system. The introduction and evaluation of a 50ng BFB standard by the MS system must meet the criteria in Table 2.

6.7 CONTINUING CALIBRATION VERIFICATION STANDARD (CCV) - An analytical standard containing all target analytes, surrogate and internal standard compounds that is used to evaluate the performance of the instrument system with respect to a defined set of method criteria.

6.8 METHOD BLANK (BLK, similarly known as the LABORATORY REAGENT BLANK) - An aliquot of organic free reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The BLK is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

6.9 TRIP BLANK - An aliquot of reagent water or other blank matrix that is placed in a sample container in the laboratory and treated as a sample in all respects, including shipment to the sampling site, exposure to sampling site conditions, storage, preservation, and all analytical procedures. The purpose of the trip blank is to determine if method analytes or other interferences are present in the field environment.

6.10 LABORATORY CONTROL SAMPLE (LCS) - The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. When the results of the matrix spike analysis indicates a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

6.11 MATRIX SPIKE/ MATRIX SPIKE DUPLICATE (MS/MSD) - An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS/MSD is analyzed exactly like a sample. Its purpose is used to evaluate the accuracy and precision of the laboratory performance of the analytical method in a specific sample matrix.

6.12 SYSTEM PERFORMANCE CHECK COMPOUNDS (SPCCs) - Selective analytes from the compound list that are used to check compound instability and to check for degradation caused by contaminated lines or active sites in the system. These compounds are chloromethane, 1,1-dichloroethane, bromoform, chlorobenzene and 1,1,2,2-tetrachloroethane.

6.13 CALIBRATION CHECK COMPOUNDS (CCCs) - Selective analytes from the compound list that are used to evaluate the calibration from the standpoint of the integrity of the system. High variability for these compounds may be indicative of system leaks or reactive sites on the column. These compounds are 1,1-dichloroethene, chloroform, 1,2-dichloropropane, toluene, ethylbenzene, and vinyl chloride.

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6.14 REAGENT WATER – Organic free water. NEL uses carbon filtration of water from its Milli-Q deionized water system, or equivalent.

7.0 Interferences

7.1 During analysis, major contaminant sources are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap. The use of Teflon tubing, Teflon thread sealants, or flow controllers and rubber components in the purging device should be avoided since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. Analyses of laboratory reagent blanks provide information about the presence of contaminants. Subtracting blank values from sample results is not permitted.

7.2 Interfering contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing relatively high concentrations of volatile organic compounds. A preventive technique is between-sample rinsing of the purging apparatus and sample syringes with two to three portions of reagent water. After analysis of a sample containing high concentrations of volatile organic compounds, one or more laboratory reagent blanks should be analyzed to check for cross-contamination. The blank must have a concentration less than one-half of the reporting level in order for analysis to proceed.

7.3 Special precautions must be taken to determine methylene chloride. The analytical and sample storage area should be isolated from the atmospheric sources of methylene chloride; otherwise, random background levels will result. Since methylene chloride will permeate Teflon tubing, all GC carrier gas lines and purge gas plumbing should be constructed of stainless steel or copper tubing. Laboratory worker's clothing should be cleaned frequently since clothing previously exposed to methylene chloride fumes during common extraction procedures can contribute to sample contamination. Extraction laboratory personnel should not enter the volatile analytical laboratory.

7.4 Traces of ketones, methylene chloride, and some other organic solvents can be present even in the highest purity methanol. This is another potential source of contamination, and should be assessed before standards are prepared in the methanol. Methanol is stored in a separate area from the extractions laboratory.

8.0 Safety

8.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials or procedures.

8.2 Material Safety Data Sheets (MSDS) are made available to all personnel involved in the chemical analysis. A written environmental health and safety plan is reviewed by all NEL staff.

8.3 Please note chemicals that have the potential to be highly toxic or hazardous, the appropriate MSDS must be reviewed by the employee before handling the chemical. The following method analytes have been tentatively classified as known or suspected human or mammalian carcinogens: benzene, carbon tetrachloride, 1,4-

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dichlorobenzene, 1,2-dichloroethane, hexachlorobutadiene, 1,1,2,2-tetrachloroethane, 1,1,2-trichloroethane, chloroform, 1,2-dibromoethane, tetrachloroethene, trichloroethene, and vinyl chloride. Pure standard materials and stock standard solutions of these compounds and all other target compounds will be handled in a hood.

9.0 Equipment and Supplies

9.1 **Containers**

9.1.1 Sample storage containers: 40 ml screw cap vials equipped with a Teflon faced silicone septum, certified clean, known volume of 44 ml (see also Method 5035)

9.1.2 Standard Storage Containers: 1-5 ml Mininert vials with Teflon lined screw caps.

9.2 **Syringes**

9.2.1 5 ml-50 ml gas tight hypodermic syringes with Luer-Lok or fixed needle tips.

9.2.2 Micro syringe 10 - 1000 µl.

9.3 **Instrumentation**

9.3.1 Autosampler: Archon 51 Position Autosampler or equivalent.

9.3.2 LCS 2000 purge and trap or equivalent.

9.3.3 Trap: Tekmar Purge Trap K or equivalent

9.3.4 Gas Chromatograph: Hewlett-Packard 5890 Series II or equivalent.

9.3.5 Mass Spectrometer: Hewlett-Packard 5970 MSD or equivalent.

9.3.6 Primary Column: Fused silica capillary column, Restek RTX-VMS 30m x 0.25 mm x 1.4 µm or equivalent.

9.4 **Data Acquisition and Processing**

Data System - Hewlett-Packard ChemStation software is used for data acquisition and HP Enviroquant software is used for data processing.

10.0 Reagents and Standards

10.1 **Reagents**

10.1.1 Methanol - Purge and Trap Grade, demonstrated to be free of analytes.

10.1.2 Reagent water – organic free deionized water or equivalent.

10.2 **Stock Standard Solutions** - These solutions are purchased as certified solutions or prepared from pure standard materials. Commercial standards arrive ampulized in concentrations ranging from 1-5 mg/ml. Solutions are stored per manufacturer's recommendations. Once opened, these concentrated solutions expire at the

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manufacturer's expiration date or one year from opening, whichever is the most conservative.

10.3 **Primary Dilution Standards** - Stock standard solutions are used to prepare primary dilution standard solutions that contain all the analytes of interest in methanol. The primary dilution standards are prepared at concentrations that can be easily diluted to prepare aqueous calibration solutions that will bracket the working concentration range. The primary dilution standard solutions are stored with minimal headspace and checked frequently for signs of deterioration or evaporation. Methanol solutions of gaseous standards are not stable for more than one week at >0°C.

10.4 **Calibration Standards** – Five or more concentrations for calibration in the range 5 to 200 ug/L for 8260B. For low level analysis (25 ml purge) five or more concentrations for calibration in the range 0.5 - 50 µg/L. Other concentrations and volumes may be used provided they meet method requirements.

10.5 **Calibration Verification Standard (CCV)** – Standard prepared at a concentration of 50 µg/L or 100µg/L for 8260B analysis that is used to evaluate the initial calibration curve. For low level analysis the standard is prepared at 10µg/L.

10.6 **Method Blank (BLK)** – Sample of organic free reagent water.

10.7 **Laboratory Control Sample (LCS)** -.- Standards prepared at a target concentration of 50 µg/L or 100µg/L from a second source for 8260B analysis and at 10µg/L for low level analysis.

10.8 **Matrix Spike/Matrix Spike Duplicate (MS/MSD)** - Matrix spikes and matrix spike duplicates are prepared and analyzed with each batch of 20 client specific samples of the same matrix, assuming sufficient sample is provided by the client. Matrix spikes are at a concentration of 50µg/L for 8260B analysis and 25µg/L for low level analysis. If the client does not specify a sample to be used as a MS/MSD, NEL will analyze a LCS Duplicate (LCSD) to verify precision.

10.9 **Internal Standard and Surrogates Fortification Solutions**- One fortification solution is required to prepare laboratory reagent blanks, standards and to fortify each sample. A fortification solution contains 4 internal standards; 1,4-difluorobenzene, pentafluorobenzene, chlorobenzene-d_5, and 1,4-Dichlorobenzene-d_4 and 3 surrogates; 4-Bromofluorobenzene, 1,2-Dichloroethane-d_4, Toluene-d_8 (surrogates) in methanol. The internal standards are present in all samples, blanks and standards at a concentration of 50 µg/L for 8260B and 10µg/L for low level analysis.

11.0 Sample Collection, Preservation, shipment and storage

11.1 **Sample collection** - Sample collection for aqueous samples is described in Method 5030. Sample collection for solid samples is described in Method 5035.

11.2 **Preservation** - Sample preservation for aqueous samples is described in Method 5030. Sample preservation for solid samples is described in Method 5035.

11.3 **Storage** - Samples are analyzed within fourteen days of collection. Other holding times may be selected by the client to conform to local regulatory requirements.

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Aqueous samples that have not been preserved are analyzed within 7 days of collection. Samples are stored at 4°C ± 2°C in a storage area free of organic solvent vapors and direct or intense light. The pH of each aqueous sample is monitored after analysis, with the value recorded in the injection logbook.

12.0 Quality Assurance/Quality Control

12.1 **4-Bromofluorobenzene (BFB)** - Prior to any analyses a 50ng BFB standard is introduced into the GC/MS for 8260B analysis. The BFB spectrum must meet all criteria in Table 2 before analysis of any standards or samples can follow.

12.2 **Method detection limits (MDLs)** are determined annually in accordance with the method described in 40 CFR, Part 136, Appendix B. The results of these studies are kept on file by the QA Manager.

12.3 **Method Blank** – One method blank must be run with every batch of samples after the calibration standards. An acceptable blank must not contain any volatile target analyte at concentrations greater than one-half the reporting limits. If the method blank exceeds these criteria, the analytical system may be out of control. The source of the contamination must be investigated and appropriate corrective measures must be taken and documented before further sample analysis proceeds. If one-half the reporting limit (RL) is exceeded, the laboratory shall evaluate whether reprocessing of the samples is necessary.

12.4 **Laboratory Control Sample (LCS)** - The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. When the results of the matrix spike analysis indicates a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

12.5 **Continuing Calibration Verification Standard (CCV)** - A standard sample of target compounds made from the same standard used to prepare the calibration curve. The CCV is run at 50µg/L for 8260B analysis at 10µg/L for low level analysis. Compounds with low response factors may have higher concentrations, but all are typically within the middle range of the calibration curve.

12.6 **Matrix Spike and Matrix Spike Duplicate (MS/MSD)** – The laboratory will analyze a MS/MSD pair when requested by the client. If a client MS/MSD request has not been received every twenty sample analyses, a laboratory control spike duplicate (LCSD) will be analyzed to serve as a measure of the precision of the batch. The MS/MSD will be prepared at a spike concentration of 50 µg/L, 50 µg/kg, or 25 µg/L for low level (25) purge.

12.7 **Surrogates/Internal Standards** - Three compounds are used as surrogates: 1,2-Dichloroethane-d_4, 4-Bromofluorobenzene, and Toluene-d_8.. Four compounds are used as internal standards: Pentafluorobenzene, chlorobenzene d_5, 1,4-Difluorobenzene and 1,4-Dichlorobenzene-d_4. Surrogate and internal standard concentrations in the purge vessel are 50 ug/L (Low water analysis at 10 ug/L). Table

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4 shows the statistical surrogate recovery limits for water samples. Table 5 shows the statistical surrogate recovery limits for soil samples.

12.8 **Internal Standard Area Response**

12.8.1 The relative retention time (RRT) of the sample analyte must be within ± 0.06 RRT units of the RRT of the standard analyte.

12.8.2 If the extended ion current profile (EICP) area in the CCV changes by a factor of two (-50% to +100%) from that in the midpoint lead in the most recent initial calibration curve, corrective action must be taken.

12.8.3 If the EICP area of any internal standard in a QC or field sample changes by more than a factor of two (-50% to +100%) from that in the CCV, reanalysis must occur to confirm matrix interference.

13.0 Calibration and standardization

13.1 **BFB** - The ion abundances shown in Table 2 must be met before analysis of any standards or samples may proceed.

13.2 **Initial Calibration** - The concentrations for the initial calibration are in the range of 5 to 200 ug/L. A minimum of five points are used to calibrate each analyte. For the low water analysis, the five concentrations for the initial calibration are in the range of 0.5 to 50 ug/L. A response factor (RF) is calculated for each analyte and/or isomer pair for each calibration solution using the appropriate internal standard. The calculation is performed as follows:

$$RF = \frac{(Ax)\,(Qis)}{(Ais)(Qx)}$$

Where:
Ax = integrated abundance of the quantitation ion of the analyte
Ais = integrated abundance of the quantitation ion of the internal standard
Qx = quantity of analyte purge in nanograms or concentration units
Qis = quantity of internal standard purged in ng or concentration units

For each analyte and surrogate, calculate the mean response factor from analyses of the calibration solutions. Calculate the standard deviation (SD) and relative standard deviation (RSD) from each mean (M).

$$\%RSD = 100 * \frac{SD}{M}$$

System performance check compounds (SPCCs) must meet the required minimum average response factor (RRF) shown in Table 3. The %RSD of each individual analyte must be ≤15%. If any analyte exceeds this 15% criteria, a linear or quadratic calibration curve can be used, with the minimum acceptable correlation coefficient being 0.990. In addition, individual calibration check compounds (CCC) must have an RSD ≤30%. If this criteria is not met maintenance is performed on the instrument and/or a new calibration curve is acquired. A copy of the linear/quadratic curve with the correlation coefficient should be printed and filed with the curve.

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13.3 **Continuing Calibration Verification (CCV)** – The calibration curve must be verified at the beginning of each run. The concentration of the CCV is 50 µg/L. For the low water analysis, the concentration of the CCV is 10 µg/L. The RF is calculated for each analyte and surrogate compound from the data measured in the continuing calibration check. System performance check compounds must meet the required minimum average response factor (RRF) shown in Table 3. The percent difference is calculated using the following equation:

$$\% \text{ Difference} = \frac{RFv - RF}{RF} \quad (100)$$

Where:
RFv = Response Factor from the analyses of the verification standard
RF = Mean Response Factor from the initial calibration

The percent difference for each CCV must be ≤ 20%. In addition, the internal standard retention time should not change by more than 30 seconds from the mid-point standard level of the most recent initial curve sequence. The integrated areas of the internal standards in the calibration verification standard should not change by more than a factor of two (-50% to +100%) from that in the mid-point standard level of the most recent initial calibration sequence. If the above criteria are not met, maintenance is performed on the instrument and/or a new calibration curve is acquired.

14.0 Procedure

14.1 **Sample Introduction and Purging** - See preparation and introduction Methods 5030 and 5035.

14.1.1 See Tables 8 and 9 for preparation of daily check standards and calibration curves.

14.2 **Gas Chromatography/Mass Spectrometry** - Data is acquired and stored over the nominal mass range 35-550 with a total cycle time (including scan overhead time) of two seconds or less. The cycle time is adjusted to measure five or more spectra during the elution of each GC peak. A multi-stage temperature ramp is used to separate the components of interest for this analysis. A typical GC temperature program is described below.

Initial temperature 40°C, initial time 7 min.
Ramp1: 9°C/min. to 50°C, hold for 0 min.
Ramp2: 27°C/min. to 110°C, hold for 1 min.
Ramp3: 40°C/min. to 220°C, hold for 2.5 min.

Instrument control and acquisition parameters are defined on the ChemStation software for each instrument.

14.3 At low level 8260B analysis the electron multiplier is set ~100 volts higher than that of the normal 8260B method in order to better quantitate the lower sample concentrations.

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14.4 **Identification of Analytes** - A sample is identified by comparison of its mass spectrum (after background subtraction) to a reference spectrum in the user-created data base. The GC retention time for each analyte should be with ± 0.5 minutes of the midpoint standard's retention time in the initial calibration curve.

14.4.1 In general, all ions that are present above 10% relative abundance in the mass spectrum of the standard should be present in the mass spectrum of the sample component and should agree within absolute 20%. For example, if an ion has a relative abundance of 30% in the standard spectrum, its abundance in the sample spectrum should be in the range of 10-50%. Some ions, particularly the molecular ion, are of special importance, and should be evaluated even if they are below 10% relative abundance.

14.4.2 Identification requires expert judgment when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When GC peaks obviously represent more than one sample component (i.e., broadened peak with shoulder(s) or valley between two or more maxima), appropriate analyte spectra and background spectra can be selected by examining plots of characteristic ions for tentatively identified components. When analytes coelute (i.e., only one GC peak is apparent), the identification criteria can be met but each analyte spectrum will contain extraneous ions contributed by the coeluting compound. Because purgeable organic compounds are relatively small molecules and produce comparatively simple mass spectra, this is not a significant problem for most method analytes.

14.4.3 Structural isomers that produce very similar mass spectra can be explicitly identified only if they have sufficiently different GC retention times. Acceptable resolution is achieved if the height of the valley between two peaks is less than 25% of the average height of the two peaks. Otherwise, structural isomers are identified as isomeric pairs. Two of the three isomeric xylenes are examples of structural isomers that are not resolved on the capillary column. These groups of isomers will be reported as isomeric pairs.

14.4.4 Methylene chloride, acetone, carbon disulfide, and other background components appear in variable quantities in laboratory and field reagent blanks and generally cannot be accurately measured. Subtraction of the concentration in the blank from the concentration in the sample is not performed because the concentration of the background in the blank is highly variable.

15.0 Calculations

15.1 Table 6 list target compounds associated with each of the 4 internal standards.

15.2 Concentrations of Unknowns - Method 8260B does not include reporting of tentatively identified compounds (TIC). However, reporting of up to 10 TIC's whose peak heights are ≥ 10% of the nearest internal standard may be requested or ≥ 40% of the nearest internal standard may be requested for low concentration analyses. Concentrations are calculated using the following formula using an assumed response factor (RF) of 1:

$$C(s) = \frac{C(is) * A(s) * DF}{A(is)}$$

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Where:
C(s) = Concentration of Unknown (ug/L)
C(is) = Concentration of internal standard (ug/L)
DF = Dilution Factor
A(s) = Area of Unknown
A(is) = Area of associated internal standard

Tentatively identified compounds (TIC) are estimated concentrations and are reported with a "J" qualifier.

15.3 Concentrations of Calibrated Compounds:

$$C(s) = \frac{A(s) * C(is) * DF}{A(is) * RRF}$$

Where:
C(s) = Concentration of compound (ug/L)
C(is) = Concentration of associated internal standard (ug/L)
DF = Dilution Factor.
A(is) = Area of quantitation ion for associated internal standard
A(s) = Area of quantitation ion for compound
RRF = Average Relative Response Factor from five-point initial calibration

15.4 Calculation of Recovery - Calculate the recovery of each spiked analyte in the MSA/MSB, LCS and ECV by the following equation:

$$Recovery = \%R = \frac{Cs - Cu}{Cn} \times 100$$

Where:
Cs = Measured concentration of the spiked sample aliquot
Cu = Measured concentration of the unspiked sample aliquot (use 0 for LCS and ICV)
Cn = Nominal (theoretical) concentration increase that results from spiking the sample, or the nominal concentration of the spike aliquot (for LCS and ICV)

15.5 Calculation of Precision - Precision is estimated from the relative percent difference (RPD) of the concentrations (not the recoveries) measured for matrix spike/matrix spike duplicate pairs, or for duplicate analyses of unspiked samples. The RPD is calculated according to the following equation below.

$$RPD = \frac{2*(\%Rec1 - \%Rec2) *100}{(\%Rec1+\%Rec2)}$$

Where:
%Rec1 = Calculated % Recovery of the compound in the 1st sample aliquot
%Rec2 = Calculated % Recovery of the compound in the 2nd sample aliquot

15.6 Data Reporting - Based on the mass spectra, it is appropriate to report values that are at a level at or above the MDL, but less than the reporting level (PQL). In this region, an analyte can be qualitatively detected, but not accurately quantified. Any

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concentrations calculated to be less than the reporting level are flagged with a "J" qualifier. Sample specific RL's are derived by taking into account various sample specific data, which can include the amount of the sample subject to testing, % moisture, dilution factor, interferences and the base RL's for the analysis.

15.6.1 Reporting qualifiers are as follows:

B = Analyte is found in the associated method blank as well as the sample
D = Any discrepancy between the reported results may be due to dilution of the sample
E = Compound quantitation is above the instrument's calibration range for this analysis
J = Indicates an estimated quantitation value
M = Analyte was evaluated to the instrument MDL per customer request
U = Compound was analyzed for but not detected

15.6.2 Data Package Definitions - three levels of reporting are available. The difference between these levels has nothing to do with the quality of the work being performed, only how it is presented.

- Level 1: A level 1 data package consists of sample results only
- Level 2: A level 2 data package consists of sample results and simple Excel quality control forms.
- Level 3: A level 3 data package consists of sample, CLP-like forms with Quality Control results.
- Level 4: A level 4 data package consists of a full set of CLP-like forms, and all supporting documentation.

Electronic-Diskette is available upon request.

16.0 Method performance

16.1 Laboratory accuracy and precision data were obtained for the method analytes using laboratory control spikes. The analytes were at a concentration of 5 µg/L for the procedural combination of Methods 5035 and 8260. The analytes were at a concentration of 5µg/L for the procedural combination of Methods 5030 and 8260B. For the low water analysis, the analytes were at a concentration of 1µg/L. Results were obtained using the analytical instrumentation described in section 9.

16.2 With this data, method detection limits were calculated using the formula:

MDL = S t (n-1, 1-alpha = 0.99)

Where:

t(n-1, 1-alpha = 0.99) = Student's t value for the 99% confidence level with n-1 degrees of freedom
n = number of replicates
S = the standard deviation of the replicate analysis

16.3 Precision and MDL study results are received and retained by the NEL Quality Assurance Manager.

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17.0 Pollution prevention and waste management

17.1 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. Please refer to NEL's Chemical Hygiene Plan for further information.

17.2 The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18.0 Data Assessment, Acceptance criteria and Corrective Action

18.1 Data is initially reviewed by the analyst in the lab and signed as such. Following this, the data is secondarily reviewed by the organics supervisor or his/her designee before being put into its final data package form (where the data is reviewed by the QA/QC department before being sent to the client). Please refer to NEL's Quality Assurance Plan for further information.

18.2 Data that is out of control is marked as such and slated for re-analysis. Any corrective action undertaken is documented on a corrective action form (detailing the client information, problem, investigation findings and solution). This form is kept in the Quality Assurance Manager's office. Refer to Table 7 for a list of possible corrective actions.

18.3 When sample concentrations exceed the upper limit of the calibration curve, samples shall be diluted and reanalyzed (if possible) to bring the concentrations within the range of the calibration curve.

If dilution is not possible due to a lack of sample and/or sample extract, the analyte whose concentration lies over the upper limit of the calibration curve shall be reported with an "E" qualifier.

18.4 Routine Maintenance. On a daily basis the sparger tube is rinsed and the trap and column are baked for a minimum of 10 minutes. The following maintenance is performed as needed.

- 18.4.1 Change trap when SPCC's fail, or response factors indicate a problem
- 18.4.2 Change sparger liner, septum, and clip column when contamination is suspected
- 18.4.3 Change column when peak separation is no longer adequate
- 18.4.4 Change or solvent rinse transfer lines to eliminate contamination
- 18.4.5 Clean source if BFB tune does not pass
- 18.4.6 Change rough pump oil when source is cleaned or annually at a minimum
- 18.4.7 Change compressed gas cylinders before they empty, preferably before they go under 500 psi

19.0 Corrective action and contingencies for out of control data

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Generally, any data that is out of control is considered unusable. There are, however, cases in which the laboratory supervisor will be made aware of the issue and, if the data is used, it will be thoroughly narrative noted. Refer to Table 7 for a list of possible corrective actions.

20.0 Waste Management

Please refer to the NEL Chemical Hygiene Plan and Spill Prevention Control & Contingency Plan (SPCC) for information regarding waste management.

21.0 References

Test Methods for EvaluatingSolid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996

NELAC Quality Systems, Chapter 5, June 5, 2003

22.0 Tables, Diagrams, Flowcharts and Validation Data

See Attached

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TABLE 1: PRACTICAL QUANTITATION LIMITS

COMPOUND	WATER µg/L	Low Level µg/L	Solid, METHANOL EXT., µg/kg*	Soil, Low Level µg/kg
Chloromethane	5	2	500	5
Bromomethane	10	2	1000	5
Dichlorodifluoromethane	5	2	500	5
Vinyl chloride	5	2	500	5
Chloroethane	10	10	1000	5
Methylene chloride	5	2	500	5
Acetone	10	10	1000	5
Carbon disulfide	5	2	500	5
Trichlorofluoromethane	5	2	500	5
1,1-Dichloroethene	5	1	500	5
1,1-Dichloroethane	5	1	500	5
1,2-Dichloroethene (cis or trans)	5	10	500	5
Chloroform	5	2	500	5
Methyl ethyl ketone (2-butanone)	10	10	1000	5
Vinyl acetate	15	15	1500	5
1,2-Dichloroethane	5	1	500	5
1,1,1-Trichloroethane	5	1	500	5
Carbon tetrachloride	5	5	500	5
Bromodichloromethane	5	1	500	5
1,2-Dichloropropane	5	1	500	5
Cis-1,3-Dichloropropene	5	1	500	5
Trichloroethene	5	1	500	5
Benzene	5	1	500	5
Dibromochloromethane	5	1	500	5
1,1,2-Trichloroethane	5	1	500	5
Methyl isobutyl ketone (4-methyl-2-pentanone)	5	5	500	5
2-Hexanone	5	5	500	5
Trans-1,3-Dichloropropene	5	1	500	5
Bromoform	5	1	500	5
1,1,2,2-Tetrachloroethene	5	1	500	5
Tetrachloroethene	5	1	500	5
Toluene	5	1	500	5
Chlorobenzene	5	1	500	5
Ethylbenzene	5	1	500	5
Styrene	5	1	500	5
Total Xylenes	5	1	500	5
Methyl-tert-butyl ether	5	1	500	5
Methacrylonitrile	5	1	500	5
Bromochloromethane	5	5	500	5
1-Chlorobutane	5	1	500	5
1,1-Dichloropropene	5	1	500	5
Methyl Methacrylate	5	5	500	5
Dibromomethane	5	1	500	5

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TABLE 1: PRACTICAL QUANTITATION LIMITS Continued

COMPOUND	WATER µg/L	Low Level µg/L	SOIL, METHANOL EXT., µg/KG*	SOIL, Low Level µg/L
2-Nitropropane	10	1	1000	5
Ethyl Methacrylate	5	5	500	5
1,3-Dichloropropane	5	1	500	5
1,2-Dibromoethane	5	1	500	5
1,1,12-Tetrachloroethane	5	1	500	5
Isopropylbenzene	5	1	500	5
1,2,3-Trichloropropane	5	1	500	5
n-Propylbenzene	5	1	500	5
Trans-1,4-Dichloro-2-butene	5	5	500	5
Bromobenzene	5	1	500	5
1,3,5-Trimethylbenzene	5	1	500	5
2-Chlorotoluene	5	1	500	5
4-Chlorotoluene	5	1	500	5
Tert-butylbenzene	5	1	500	5
1,2,4-Trimethylbenzene	5	1	500	5
Pentachloroethane	5	1	500	5
Sec-Butylbenzene	5	1	500	5
4-Isopropyltoluene	5	1	500	5
1,3-Dichlorobenzene	5	1	500	5
1,4 Dichlorobenzene	5	1	500	5
n-Butylbenzene	5	1	500	5
1,2-Dichlorobenzene	5	1	500	5
Hexchloroethane	5	1	500	5
1,2-Dibromo-3-chloropropane	5	1	500	5
Nitrobenzene	15	15	1500	5
1,2,4-Trichlorobenzene	5	1	500	5
Hexachlorobutadiene	5	5	500	5
Naphthalene	5	1	500	5
1,2,3-Trichlorobenzene	5	1	500	5

* NEL method routinely used for analysis of soils, sludges, and other solid materials; the low-level (heated purge) is used at the request of the client to achieve lower reporting levels.

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TABLE 2: ACCEPTABLE BFB TUNING CRITERIA

MASS	ION 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	LESS THAN 2% OF MASS 174
174	GREATER THAN 50% OF MASS 95
175	5 TO 9% OF MASS 174
176	GREATER THAN 95% BUT LESS THAN 101% OF MASS 174
177	5 TO 9% OF MASS 176

TABLE 3: CALIBRATION CRITERIA

Compound	Minimum RRF	Maximum % RSD	Maximum %D
Acetone	0.01	15	
Acrolein	0.01	15	
Acrylonitrile	0.01	15	
Allyl Chloride	0.01	15	
Benzene	0.01	15	
Bromochloromethane	0.01	15	
Bromodichloromethane	0.01	15	
Bromoform (SPCC)	0.1	15	
Bromomethane	0.01	15	
2-Butanone	0.01	15	
Carbon Disulfide	0.01	15	
Carbon Tetrachloride	0.01	15	
Chlorobenzene (SPCC)	0.30	15	
Chloroethane	0.01	15	
2-Chloroethyl Vinyl Ether	0.01	15	
Chloroform (CCC)	0.01	30	20
Chloromethane (SPCC)	0.10	15	
2-Chlorotoluene	0.01	15	
4-Chlorotoluene	0.01	15	
1,2-Dibromo-3-chloropropane	0.01	15	
Dibromochloromethane	0.01	15	
1,2-Dibromoethane	0.01	15	
Dibromomethane	0.01	15	
1,2-Dibhlorobenzene	0.01	15	
1,3-Dichlorobenzene1,4-Dichlorobenzene	0.01	15	
Cis-1,4-Dichloro-2-butene	0.01	15	
Trans-1,4 Dichloro-2-butene	0.01	15	

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Dichlorodifluoromethane	0.01	15	
1,1-Dichloroethane (SPCC)	0.1	15	
1,2-Dichloroethane	0.01	15	
1,1-Dichloroethene (CCC)	0.01	30	20
Cis-1,2-Dichloroethene	0.01	15	
Trans-1,2-Dichloroethene	0.01	15	
1,2-Dichloropropane (CCC)	0.01	30	20
1,3-Dichloropropane	0.01	15	
2,2-Dichloropropane	0.01	15	
1,1-Dichloropropene	0.01	15	
Cis-1,3-Dichloropropene	0.01	15	
Trans-1,3-Dichloropropene	0.01	15	
Ethyl Methacrylate	0.01	15	
Ethylbenzene (CCC)	0.01	30	20
Hexachlorobutadiene	0.01	15	
2-Hexanone	0.01	15	
Isopropylbenzene	0.01	15	
4-Isopropyltoluene	0.01	15	
Methacrylonitrile	0.01	15	
Methyl Methacrylate	0.01	15	
4-Methyl-2-pentanone	0.01	15	
Methyl-t-Butyl Ether	0.01	15	
Methylene Chloride	0.01	15	
Naphthalene	0.01	15	
Propionitrile	0.01	15	
n-Propylbenzene	0.01	15	
Styrene	0.01	15	
1,1,1,2-Tetrachloroethane	0.01	15	
1,1,2,2-Tetrachloroethane (SPCC)	0.3	15	
Tetrachloroethene	0.01	15	
Tetrahydrofuran	0.01	15	
Toluene (CCC)	0.01	30	20
1,2,3-Trichlorobenzene	0.01	15	
1,2,4-Trimethylbenzene	0.01	15	
1,3,5-Trimethylbenzene	0.01	15	
1,2,4-Trichlorobenzene	0.01	15	
1,1,1-Trichloroethane	0.01	15	
1,1,2-Trichloroethane	0.01	15	
Trichloroethene	0.01	15	
Trichlorofluoromethane	0.01	15	
1,2,3-Trichloropropane	0.01	15	
Vinyl Acetate	0.01	15	
Vinyl Chloride (CCC)	0.01	30	20
Xylene (m,p)	0.01	15	

(SPCC) System Performance Check Compound, (CCC) Calibration Check Compound

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TABLE 4: SURROGATE RECOVERY FOR AQUEOUS SAMPLES

Surrogate Name	Aqueous Samples %Recovery	Aqueous Samples – 25 ml Purge % Recovery
1,2 Dichloroethane-d4	76-114	70-152
Toluene-d8	88-110	82-140
4-Bromofluorobenzene	86-115	82-114

% Recovery Limits are statistical and updated annually

TABLE 5: SURROGATE RECOVERY FOR SOLID SAMPLES

Surrogate Name	MeOH Samples %Recovery	Low-level Soil Samples % Recovery
1,2 Dichloroethane-d4	83-131	61-155
Toluene-d8	70-128	61-134
4-Bromofluorobenzene	80-117	68-125

TABLE 6: INTERNAL STANDARDS AND ASSOCIATED TARGET COMPOUNDS

Pentafluorobenzene	1,4-Difluorobenzene	Chlorobenzene-d5	1,4-Dichlorobenzene-d4
Dichlorodifluoromethane Chloromethane Vinyl Chloride Bromomethane Chloroethane Trichlorofluoromethane Diethyl Ether Acetone 1,1-Dichloroethene Iodomethane Allyl Chloride Carbon Disulfide Methylene Chloride Acrylonitrile t-Butyl-Methyl-Ether trans-1,2-Dichloroethene Vinyl Acetate 1,1-Dichloroethane 2-Butanone Propionitrile 2,2-Dichloropropane cis-1,2-Dichloroethene Methacrylonitrile Chloroform	Trichloroethene 1,2-Dichloropropane Methyl Methacrylate Bromodichloromethane Dibromomethane 2-Nitropropane 2-Chloroethylvinyl Ether 4-Methyl-2-Pentanone cis-1,3-Dichloropropene Toluene-d8 Toluene Trans-1,3-Dichloropropene Ethyl Methacrylate 1,1,2-Trichloroethane 2-Hexanone 1,3-Dichloropropane Tetrachloroethene Dibromochloromethane 1,2-Dibromoethane	Chlorobenzene 1,1,1,2-Tetrachloroethane Ethylbenzene m,p-Xylene o-Xylene Styrene Bromoform Isopropylbenzene 1,1,2,2-Tetrachloroethane 4-Bromofluorobenzene 1,2,3-Trichloropropane n-Propylbenzene trans-1,4-Dichloro-2-Butene Bromobenzene 1,3,5-Trimethylbenzene 2-Chlorotoluene 4-Chlorotoluene tert-Butylbenzene 1,2,4-Trimethylbenzene Pentachloroethane sec-Butylbenzene 4-Isopropyltoluene	1,3-Dichlorobenzene 1,4-Dichlorobenzene n-Butylbenzene 1,2-Dichlorobenzene Hexachloroethane 1,2-Dibromo-3-Chloropropane 1,2,4-Trichlorobenzene Hexachlorobutadiene Naphthalene 1,2,3-Trichlorobenzene

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Bromochloromethane 1,2-Dichloroethane-d4 Tetrahydrofuran 1,1,1-Trichloroethane 1-Chlorobutane 1,1-Dichloropropene Carbon Tetrachloride 1,2-Dichloroethane Benzene			

TABLE 7: 8260 VOLATILE ORGANIC ANALYTICAL RUN SEQUENCE AND CORRECTIVE ACTION

Quality Control Criteria	Frequency	Acceptance	Corrective Action
BFB	12 Hour	Criteria Table 2	Reanalyze. Manual tune clean source.
ICAL: Minimum of five points	As Required.	Minimum RF for SPCCs, maximum 30% RSD for CCCs . All analytes ≤ 15% RSD or r^2 > 0.990.	System check, mix new standards, recalibrate, reanalyze.
CCV	Beginning of each 12-hour window, after tune, or as a part of curve.	Minimum RF for SPCCs, % Difference each CCC ≤20%	System check, recalibrate.
LCS/LCSD	Every analytical batch if MS and MSD not analyzed.	% Recovery 60-140%, 10% of compounds can fail, as long as recoveries > 10%.	Check Std., check quantitation, evaluate MS/MSD, reanalyze analytical batch if data impacted.
Method Blank	Every analytical batch.	Targets < ½ of Reporting levels.	Check for contamination, reanalyze. Batch if data impacted.
Samples	Until 12-hour window is closed.	Results < Highest Calibration Std., Surrogates meeting Table 4 and 5 criteria, int. std. Areas – 50% to +100% of CCV istd area.	Dilute and reanalyze if over curve. If surrogate/internal std. Recovery fails, reanalyze to confirm matrix interference.
Matrix Spike/Matrix Spike Duplicate	Every analytical batch if sufficient sample.	% Recoveries 60-140% % RPD 20% - Aqueous % RPD 50% - Soils	Reanalyze if analytical problem. Evaluate LCS/LCSD if poor matrix.

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TABLE 8: DAILY 8260 ANALYSES

8260 Calibrations
(Standard VOC Mix in a 50 ml Syringe)

Final Concentration ppb	ul VOC Mix 250 ug/ml	ul 8250 SURR 500 ug/ml	ul 8260 ISTD 500 ug/ml
5	1.0	0.50	5.0
10	2.0	1.0	5.0
20	4.0	2.0	5.0
50	10	5.0	5.0
100	20	10	5.0
150	30	15	5.0
200	40	20	5.0

Daily 8260 Run
(1-4 are prepared in a 50 ml Syringe & then transferred into a VOA vial for Autosampler use)

1	Tune	5 ul of 500 ug/ml 4 BFB (50 ng BFB)
2	CCV:	10 ul of 250 ug/ml VOC mix 5 ul of 500 ug/ml SURR 5 ul of 500 ug/ml ISTD (50 ppb CCV, 50 ppb ISTD & SURR)
3	LCS/LCSD	10 ul of 250 ug/ml VOC QC Mix 5 ul of 500 ug/ml SURR 5 ul of 500 ug/ml ISTD (50 ppb QC, ISTD & SURR)
4	Method Blank	5 ul of 500 ug/ml SURR 5 ul of 500 ug/ml ISTD (50 ppb ISTD & SURR)
5	Sample MS/MSD	8.8 ul 250 ug/ml VOC QC Mix 4.4 ul of 500 ug/ml SURR 4.4 ul of 500 ug/ml ISTD (50 ppb QC, ISTD & SURR)
6	Samples	4.4 ul of 500 ug/ml SURR 4.4 ul of 500 ug/ml ISTD (50 ppb ISTD & SURR)

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TABLE 9: 8260 Low Level Runs (25 ml purge)

8260 Low Level Calibrations
(Standard VOC Mix in a 50 ml Syringe)

Final Concentration ppb	UI VOC Mix 25 ug/ml	UI 8260 SURR 100 ug/ml	UI 8260 ISTD 100 ug/ml
0.5	1.0	0.25	5.0
1.0	2.0	0.50	5.0
2.0	4.0	1.0	5.0
5.0	10	2.5	5.0
10	20	5.0	5.0
20	40	10	5.0
50	100	25	5.0

Daily 8260 Low Level Run
(1-4 are prepared in a 50 ml Syringe & then transferred into a VOA vial for Autosampler use)

1	Tune	2.5 ul of 500 ug/ml 4 BFB (25 ng BFB)
2	CCV:	20 ul of 25 ug/ml VOC mix 5 ul of 100 ug/ml SURR 5 ul of 100 ug/ml ISTD (10 ppb CCV, 50 ppb ISTD & SURR)
3	LCS/LCSD	20 ul of 25 ug/ml VOC QC Mix 5 ul of 100 ug/ml SURR 5 ul of 100 ug/ml ISTD (10 ppb QC, ISTD & SURR)
4	Method Blank	5 ul of 100 ug/ml SURR 5 ul of 100 ug/ml ISTD (10 ppb ISTD & SURR)
5	Sample MS/MSD	4.4 ul 25 ug/ml VOC QC Mix 5.0 ul of 100 ug/ml SURR 5.0 ul of 100 ug/ml ISTD (25 ppb QC, 10 ppb ISTD & SURR)
6	Samples	4.4 ul of 100 ug/ml SURR 4.4 ul of 100 ug/ml ISTD (10 ppb ISTD & SURR)

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TABLE 10: Preparation of Calibration Standards

250 ug/ml VOC Std Mix:	250 ul VOC liquids (Accustandard M-502A-R-10x 2.0 mg/ml)
	250 ul VOC additions (Accustandard M-524R-B 2.0 mg/ml)
	250 ul Vinyl Acetate (Accustandard APP-9-211-20x 2.0 mg, ml)
(All compounds at 250 ug/ml except Acrylonitrile, MTBE & M,p-Xylene are at 500 ug/ml)	250 VOC Gases (Accustandard M-502-B-10x 2.0 mg/ml
	250 ul 2-CEVE (Accustandard M-601C-10x 2.0 mg/ml)
	250 ul Oxygenates (Accustandad OGAC-001 2.0 mg/ml)
	250 ul Acrolein/Acrylonitrile Mix (Ultra AMN-623 2.0 mg/ml)
	250 ul P & T Grade MeOH to bring FV to 2.0 ml

25 ug/ml VOC Std Mix:	100 ul VOC std mix from above (250 ug/ml)
(All compounds at 25 ug/ml except Acylonitrile, MTBE & M,p-Xylene are at 50 ug/ml)	900 ul P & T Grade MeOH to bring FV to 1.0 ml

250 ug/ml VOC AC Mix:	250 ul VOC mix (Ultra DWM-592 2.0 mg/ml)
	250 ul VOC mix (Ultra DWM-588 2.0 mg/ml)
(All compounds at 250 ug/ml except Acrylonitrile, MTBE & M,p-Xylene are at 500 ug/ml)	250 ul Calif Oxygenates (Restek 30465 2.0 mg/ml)
	250 ul 2-CEVE (Restek 30265 2.0 mg/ml)
	250 ul Vinyl Acetate (Restek 30216 2.0 mg/ml)
	750 ul P & T Grade MeOH to bring FV to 2.0 ml)

25 ug/ml VOC QC Mix:	100 ul VOC QC from above (500 ug/ml)
(All compounds at 25 ug/ml except Acylonitrile, MTBE & M,p-Xylene are at 50 ug/ml)	900 ul P & T Grade MeOH to bring FV to 1.0 ml

500 ug/ml 8260 ISTD	200 ul 8260 ISTD mix (Restek 30074 2.5 mg/ml)
	800 P & T Grade MeOH to bring FV to 1.0

100 ug/ml 8260 ISTD	100 ul 8260 ISTD from above (500 ug/ml)
	900 P & T Grade MeOH to bring FV to 1.0

500 ug/ml 8260 Surrogate	200 ul 8260 Surrogate Mix (Restek 3004 2.5 mg/ml)
	800 P & T Grade MeOH to bring FV to 1.0

100 ug/ml 8260 Surrogate	100 ul 8260 Surrogate from above (500 ug/ml)
	900 P & T Grade MeOH to bring FV to 1.0

500 ug/ml BFB Tuning Mix	100 ul 4-BFB (Restek 30003 5.0 mg/ml)
	900 P & T Grade MeOH to bring FV to 1.0

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REV:	I
PAGE:	1 OF 32

DOCUMENT HISTORY AND APPROVAL PAGE

TITLE:	Organochlorine Pesticides by Gas Chromatography (8081A)
DEPARTMENTS AFFECTED:	Organics

ISSUE DATE:	REVISION CODE:	SOP #:	REVISION CHANGES:
2/25/98	-	NEL Org SOP 14	Create an SOP for Pesticides
3/22/99	A	Binax /NEL ORG SOP 14	Update the SOP for Pesticides
3/18/02	B	3.3.14	Update SOP to NELAC Format
5/27/03	C	3.3.14	Client Project Plan
5/15/04	D	3.3.14	Edits & annual review
2/11/05	E	3.3.14	Annual Review & Update
10/02/06	F	3.3.14	Update to include Navy Assessment Findings
12/11/06	G	3.3.14	Update to Include Navy Assessment Findings
02/14/07	H	3.3.14	Annual Review & Update
01/23/08	I	3.3.14	Annual Review & Update

DOCUMENT APPROVAL		
Originator: James Galasyn, Ph.D.	Signature/Date: J Galasyn 012308	Title Chemistry Lab Manager
Approved by: Joe Casarin	Joe Casarin 012508	Organics Supervisor
Reviewed by: Pamela Doughty	Pamela Doughty 1/25/08	Total Quality Manager

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Northeast Laboratory Services, Inc.	Standard Operating Procedure		
TITLE: **Organochlorine Pesticides by Gas Chromatography (8081A)**			
SOP# 3.3.14	REV# I	EFFECTIVE 01/23/08	PAGE 2 of 32
WRITTEN BY: Jim Galasyn	DEPARTMENTS AFFECTED: Organics		

1.0 Test Method:

This Standard Operating Procedure (SOP) references EPA method 8081A, Organochlorine Pesticides by Gas Chromatography performed at and by Northeast Laboratory Services, Inc. (NEL).

2.0 Applicable Matrix or Matrices:

This SOP is used to determine the concentrations of various organochlorine pesticides in extracts from solid and liquid matrices.

3.0 Detection Limit:

Reporting limits for aqueous and solid samples are in the low part per billion range. Current method detection limits studies are on file in the QA/QC manager's office.

4.0 Scope and Application:

4.1 Method 8081 is used to determine the concentrations of various organochlorine pesticides in extracts from solid and liquid matrices, using fused-silica, open tubular, capillary columns with electron capture detectors (ECD). When compared to the packed columns, these columns offer improved resolution, better selectivity, increased sensitivity, and faster analysis. The compounds listed below may be determined by either a single- or dual-column analysis system.

Compound	CAS Registry No.
Aldrin	309-00-2
Alpha-BHC	319-84-6
Beta-BHC	319-85-7
Gamma-BHC (Lindane)	58-89-9
Delta-BHC	319-86-8
Chlorobenzilate	510-15-6
Alpha-Chlordane	5103-71-9
Gamma-Chlordane	5103-74-2
Technical Chlordane	57-74-9
DBCP	96-12-8
4,4'-DDD	72-54-8
4,4'-DDE	72-55-9
4,4'-DDT	50-29-3

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Diallate	2303-16-4
Dieldrin	60-57-1
Endosulfan I	959-98-8
Endosulfan II	33213-65-9
Endosulfan Sulfate	1031-07-8
Endrin	72-20-8
Endrin aldehyde	7421-93-4
Endrin ketone	53494-70-5
Heptachlor	76-44-8
Heptachlor Epoxide	1024-57-3
Hexachlorobenzene	118-74-1
Hexachlorocyclopentadiene	77-47-4
Isodrin	465-73-6
Methoxychlor	72-43-5
Toxaphene	8001-35-2

The following compounds may also be determined using this method:

Alachlor	15972-60-8
Captafol	2425-06-1
Chloroneb	2675-77-6
Chloropropylate	99516-95-7
Chlorothalonil	1897-45-6
DCPA	1861-32-1
Dichlone	117-80-6
Dicofol	115-32-2
Etridiazole	2593-15-9
Halowax-1000	58718-66-4
Halowax-1001	58718-67-5
Halowax-1013	12616-35-2
Halowax-1014	12616-36-3
Halowax-1051	2234-13-1
Halowax-1099	39450-05-0
Mirex	2385-85-5
Nitrofen	1836-75-5
PCNB	82-68-8
Permethrin	51877-74-8
Perthane	72-56-0
Propachlor	1918-16-7
Strobane	8001-50-1
Trans-Nonachlor	39765-80-5
Trifluralin	1582-09-8

4.2 The analyst must select columns, detectors and calibration procedures most appropriate for the specific analytes of interest in a study. Matrix specific performance data must be established and the stability of the analytical system and instrument calibration must be established for each analytical matrix.

4.3 Although performance data are presented for many of the target

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analytes, it is unlikely that all of them could be determined in a single analysis. The chemical and chromatographic behaviors of many of these analytes can result in co-elution of some target analytes. Several cleanup/fractionation schemes are provided in this method.

4.4 Several multicomponent mixtures (i.e. Chlordane and Toxaphene) are listed as target analytes. When samples contain more than one multicomponent analyte, a higher level of analyst expertise is required to attain acceptable levels of qualitative and quantitative analysis. The same is true of multi-component analytes that have been subjected to environmental degradation or degradation by treatment technologies. These result in "weathered" multi-component mixtures that have significant differences in peak patterns than those of standards.

4.5 Compound identification based on single-column analysis must be confirmed on a second column, or should be supported by at least one other 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. GC/MS Method 8270 is also recommended as a confirmation technique, if sensitivity permits.

4.6 This method includes a dual-column option. This option allows a hardware configuration of two analytical columns joined to a single injection port. This option allows one injection to be used for dual-column analysis. Analysts are cautioned that the dual-column option may not be appropriate when the instrument is subject to mechanical stress, many samples are to be run in a short period or when contaminated samples are analyzed.

4.7 This method is restricted to use by, or under the supervision of, analysts experienced in the use of gas chromatographs (GC) and skilled in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method.

5.0 Summary of the Method:

5.1 A measured volume or weight of sample (approximately 1 L for liquids, 2 g to 30 g for solids) is extracted using the appropriate matrix-specific sample extraction technique.

5.2 Liquid samples are extracted at neutral pH with methylene chloride using either Method 3510 (separatory funnel), Method 3520 (continuous liquid-liquid extractor), or other appropriate technique.

5.3 Solid samples are extracted with hexane-acetone (1:1) or methylene chloride-acetone (1:1) using Method 3540 (Soxhlet), Method (3541 (automated soxhlet), Method 3545 (pressurized fluid extraction), Method 3550 (ultrasonic extraction), or other appropriate technique.

5.4 A variety of cleanup steps may be applied to the extracted, depending on the nature of the matrix interferences and the target compounds.

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Suggested cleanups include alumina (Method 3610), Florisil (Method 3620), silica gel (Method 3630), gel permeation chromatography (Method 3640), and sulfur (Method 3660).

5.5 After cleanup, the extract is analyzed by injecting a 1-ul sample into a gas chromatograph with a narrow- or wide-bore fused silica capillary column and electron capture detector (GC/ECD) or an electrolytic conductivity detector (GC/ELCD).

6.0 Definitions:

6.1 SOP-Standard Operating Procedure

6.2 GC- Gas chromatograph

6.3 ECD- Electron capture detector

6.4 ELCD- Electrolytic conductivity detector

6.5 PCB- Polychlorinated Biphenyl

6.6 DCB- Decachlorobiphenyl

6.7 TCMX- Tetrachloro-m-xylene

6.8 PTFE- polytetrafluoroethylene

7.0 Interferences:

7.1 Sources of interference in this method can be grouped into four broad categories.

7.1.1 Contaminated solvents, reagents, or sample processing hardware.

7.1.2 Contaminated GC carrier gas, parts, column surfaces, or detector surfaces.

7.1.3 Compounds extracted from the sample matrix to which the detector will respond.

7.1.4 Interferences co-extracted form the samples will vary considerable from waste to waste. While general cleanup techniques are referenced or provided as part of this method, unique samples may require additional cleanup approaches to achieve desired degrees of discrimination and quantitation.

7.2 Interferences by phthalate esters introduced during sample preparation can pose a major problem in pesticide determination.

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7.2.1 These materials may be removed prior to analysis using method 3640 (Gel Permeation Cleanup) or Method 3630 (Silica Gel Cleanup).

7.2.2 Common flexible plastics contain varying amounts of phthalate esters which are easily extracted or leached from such materials during laboratory operations.

7.2.3 Cross-contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled.

7.2.4 Interferences from phthalate esters can best be minimized by avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination. Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination.

7.3 Glassware must be scrupulously cleaned. Clean all glassware as soon as possible after use by rinsing with the last solvent used. This should be followed by detergent washing with hot water, and rinses with tap water and organic-free reagent water. Drain the glassware and dry it in an oven at 130 °C for several hours, or rinse with methanol and drain. Store dry glassware in a clean environment.

7.4 The presence of elemental sulfur will result in broad peaks that interfere with the detection of early eluting organochlorine pesticides. Sulfur contamination should be expected with sediment samples. Method 3660 is suggested for removal of sulfur. Since the recovery of Endrin aldehyde (using the TBA procedure) is drastically reduced, this compound must be determined prior to sulfur cleanup.

7.5 Waxes, lipids and other high molecular weight materials can be removed by Method 3640 (gel-permeation cleanup).

7.6 Other halogenated pesticides or industrial chemicals may interfere with the analysis of pesticides. Certain co-eluting organophosporous pesticides are elimated by method 3640 (gel permeation cleanup-pesticide option). Co-eluting chlorophenols may be eliminated by using 3630(silica gel), Method 3620 (florisil) or method 3610 (alumina). Polychlorinated biphenyls (PCBs) may also interfere with the analysis of Organochlorine pesticides. The problem may be most severe for the analysis of multicomponent analytes such as Chlordane, Toxaphene and Strobane. If PCBs are known or expected to occur in samples, the analyst should consult Methods 3620 and 3630 for techniques that may be used to separate the pesticides from the PCBs.

8.0 Safety:

The laboratory is responsible for maintaining a current awareness of OSHA

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regulations regarding safe handling of chemicals. Please refer to the Northeast Laboratory Chemical Hygiene Plan and Spill Prevention Control and Contingency Plan for further information.

9.0 Equipment and Supplies:

9.1 Gas Chromatograph: an analytical system complete with gas chromatograph suitable for on-column and/or split-splitless injection and all required accessories including syringes, autosampler vials and crimp top caps, autosampler vial inserts, autosampler vial cap crimper and decrimper, analytical columns, gases, electron capture detectors (ECD) and recorder/integrator or data system.

9.2 This method describes procedure for both single-column and dual-column analyses. The single-column approach involves one analysis to determine that a compound is present, followed by a second analysis to confirm the identity of the compound.

The following column pairs are currently used at NEL. Other columns may be used provided they equal or exceed the performance described in this method, or as appropriate for the intended application.

9.2.1 Column pair 1 (two out of the following three columns)

30 m x 0.53 mm ID fused silica capillary column chemically bonded with SE-54 (RTX-5 or equivalent) 0.25 um film thickness

30 m x 0.53 mm ID fused silica capillary column chemically bonded with 35 percent phenyl methylpolysiloxane (DB608 or equivalent) 0.83 um film thickness

30m x 0.53 mm ID fused silica capillary column chemically bonded with 50 percent phenyl methylpolysiloxane (RTX-1701 or equivalent) 0.25 um film thickness

Recommended oven temperature program and operating parameters:
Injection port temperature: 225 °C
Detector A or Detector B temperature:300 °C
Oven Temperature program:
Initial Temperature: 150 °C
Initial Hold: 0.5 minutes
Ramp 1: 5.0 °C/min
Final Temperature: 275 °C
Final Hold: 0 minutes
Oven Max temperature: 300 °C
Equilibration Time: 1.00 minute
Delay Time: 0 minutes
Sampling Rate 1.2500 pts/s
Injection volume: 1.0 ul

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Injection Speed: NORM
Sample washes: 5
Sample Pumps: 5
Viscosity Delay: 0
Solvent A Washes: 5
Solvent B Washes: 5
Carrier gas: Helium (5 ml/min)

9.2.2 Column pair 2 – NEL currently uses this option.

30 m x 0.25 mm ID fused silica capillary column chemically bonded with DB-XLB or equivalent- 0.5 um film thickness

30 m x 0.32 mm ID fused silica capillary column chemically bonded with DB-35MS or equivalent- 0.25 um film thickness

Recommended oven temperature program and operating parameters:
Injection port temperature: 225 °C
Detector A and Detector B temperature: 325 °C
Oven Temperature program:
Initial Temperature: 110 °C
Initial Hold: 0.5 minutes
Ramp 1: 25.0 °C/min
Ramp 2: 15.0 °C/min to 260 °C
Ramp 3: 15.0 °C/min to 320 °C, hold for 4.00 minutes
Final Temperature: 320 °C
Final Hold: 4.00 minutes
Oven Max temperature: 325 °C
Equilibration Time: 1.00 minute
Delay Time: 3.00 minutes
Sampling Rate 1.2500 pts/s
Injection volume: 1.0 ul
Injection Speed: NORM
Sample washes: 5
Sample Pumps: 5
Carrier gas: Helium (5 ml/min)
NEL currently uses a DB-XLB column on Channel A (J & W Scientific, 30 m, 0.32 mm ID, 0.5um film thickness) and a DB-35MS column on Channel B (J & W Scientific, 30 m, 0.32 mm ID, 0.25 um film thickness)

9.3 Press tight Y-shaped glass 3-way union splitter (J&W Scientific, catalogue No. 705-0733) or a Y-shaped fused silica connector (Restek, Catalogue No. 20405), or equivalent

9.4 Volumetric flasks, 10 ml and 25 ml (or volumetric pipettes 10 ml and 25 ml) for preparation of standards.

9.5 Disposable pasteur pipets- flint glass Fisherbrand Catologue #13-678-6A (5 ¾ inch) and Fisherbrand Catalogue #13-678-6B (9 inch)

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or equivalent.

9.6 Safeskin purple nitrile exam gloves Catologue # 55082 or equivalent.

10.0 Reagents and Standards:

10.1 Reagent grade or pesticide grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

Note: Store the standard solutions (stock, composite, calibration, internal and surrogate) at 4°C in polytetrafluoroethylene (PTFE) sealed containers in the dark. All stock standard solutions must be replaced after one year or at the manufacturer's expiration date, whichever is sooner. All other standard solutions must be replaced after six months or sooner if routine QC indicates a problem.

10.2 Solvents used in the extraction and cleanup procedures (appropriate 3500 and 3600 series methods) include n-hexane, diethyl ether, methylene chloride, acetone, ethyl acetate and isooctane and must be exchanged to n-hexane or isooctane prior to analysis. Therefore, n-hexane and isooctane will be required for this procedure. Acetone or toluene may be required for the preparation of some standard solutions. All solvents should be pesticide quality or equivalent and each lot of solvent should be determined to phthalate free.

10.3 Stock standard solutions may be prepared from pure standard materials or can be purchased as certified solutions. Northeast Laboratory Services attempts to purchase standards as certified solutions whenever possible. In general, standards are purchased as composite stock standards whenever possible. Composite solutions purchased are listed in Table 1.

10.4 Calibration standards should be prepared at a minimum of five different concentrations by dilution of the composite stock standard with isooctane or hexane. The concentrations should correspond to the expected range of concentrations found in real samples and should bracket the linear range of the detector. See Table 2 for NEL calibration levels.

10.4.1 Separate calibration standards are required for each multi-component target analyte (e.g. Toxaphene and Chlordane). Analysts should evaluate the specific Toxaphene standard carefully. Some Toxaphene components, particularly the more heavily chlorinated components, are subject to dechlorination reactions. As a result, standards from different vendors may exhibit marked

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differences which could lead to possible false negative results or to large differences in quantitative results.

10.5 Surrogate standards

The performance of the method should be monitored using surrogate compounds. Surrogate standards are added to all samples, method blanks, matrix spikes and calibration standards. Decachlorobiphenyl (DCB) and tetrachloro-m-xylene (TCMX) have been found to be a useful pair of surrogates for both the single-column and dual-column configurations. Northeast Laboratory Services uses this pair of surrogates. Surrogate spiking solutions should be stored in the dark at 4 °C in PTFE-sealed containers.

11.0 Sample Collection, Preservation, and Storage:

11.1 Aqueous samples-Containers used to collect samples for the determination of semivolatile organics should be soap and water washed followed by methanol (or isopropanol) rinsing. Alternatively, containers pre-cleaned by the manufacturer should be rinsed with methylene chloride to minimize interferences. The sample containers should be of Amber glass or Teflon and have screw-caps with Teflon liners. In situations where Teflon is not available, solvent-rinsed aluminum foil may be used as a liner. However, acidic or basic samples may react with the aluminum foil, causing eventual contamination of the sample. Plastic containers or lids may NOT be used for the storage of samples due to the possibility of sample contamination from the phthalate esters and other hydrocarbons within the plastic. Sample containers should be filled with care so as to prevent any portion of the collected sample coming in contact with the sampler's gloves, thus causing contamination. Samples should not be collected or stored in the presence of exhaust fumes. If the sample comes in contact with the sampler (e.g. if an automatic sampler is used), run organic-free reagent water through the sampler and use as a field blank. A minimum of two 1-liter amber glass containers should be collected for each sample location. Additional sample should be collected if matrix spike/matrix spike duplicates are to be performed. Samples should be cooled to 4°C +/- 2 °C from the time of sampling to the time of extraction. Samples should be extracted within 7 days of collection and extracts analyzed within 40 days following extraction.

11.2 Solid samples- Containers used to collect samples for the determination of semivolatile organics should be soap and water washed followed by methanol (or isopropanol) rinsing. Alternatively, containers may be pre-cleaned by the manufacturer. The sample containers should be of glass or Teflon and have screw-caps with Teflon liners. In situations where Teflon is not available, solvent-rinsed aluminum foil may be used as a liner. However, acidic or basic samples may react with the aluminum foil, causing eventual contamination of the sample. Plastic containers or lids may NOT be used for the storage of samples due to the possibility of sample contamination from the phthalate esters and other hydrocarbons

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within the plastic. Sample containers should be filled with care so as to prevent any portion of the collected sample coming in contact with the sampler's gloves, thus causing contamination. Samples should not be collected or stored in the presence of exhaust fumes. Samples should be cooled to 4°C +/- 2 °C from the time of sampling to the time of extraction. Samples should be extracted within 14 days of collection and extracts analyzed within 40 days following extraction.

12.0 Quality Control:

12.1 The percent difference between the continuing calibration and the initial calibration should be within +/- 20 percent. Percent difference is calculated as follows:

$$\% \text{ difference} = \frac{\text{Concentration (actual)} - \text{Concentration (expected)}}{\text{Concentration (expected)}} \times 100$$

12.2 The absolute retention time for each analyte and surrogate should be centered around the retention time of the calibration verification standard at the beginning of the analytical shift. Each subsequent injection of a standard during the 12-hour analytical shift must be checked against the retention time windows. If any of these subsequent standards fall outside their absolute retention time windows, the GC system is out of control. Determine the cause of the problem and correct it. If the problem cannot be corrected, a new initial calibration must be performed.

12.3 In addition to the quantitative measure of comparison, analysts should evaluate chromatograms and instrument operation. Questions that should be asked include:

Do the peaks look normal (Gaussian)?
Is the response obtained compared to the response from previous calibrations?
Do the column fittings need tightening?
Are non-target peaks present in calibration analyses?
Are contaminants present in the blanks?
Is the injector leaking?

12.4 Recalibration of the instrument must take place when the performance changes to the point that the calibration verification acceptance criteria cannot be achieved. In addition, significant maintenance activities or hardware changes may also require recalibration. The sections below provide general guidance on the sorts of procedure that may or may not require recalibration.

12.4.1 There are various types of instrument maintenance that should not automatically require recalibration of the instrument.

Examples include changing: septa; compressed gas cylinders; syringes; moisture, hydrocarbon or oxygen traps; split seals; column

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fittings; inlets or filaments. Other procedures include breaking off or changing a guard column or cleaning the inlet. Whenever such procedures are performed, the analyst must demonstrate that the results for a calibration verification standard meet the acceptance criteria before the analysis of any samples. Otherwise, recalibration is required.

12.4.2 In contrast to section 12.2.4.1, some maintenance procedures are so likely to affect the instrument response that recalibration is automatically required, regardless of the ability to meet the calibration verification acceptance criteria. These procedures include: changing, replacing or reversing the column or changing the injection port. Whenever such procedures are performed, the analyst must perform a new initial calibration that meets the requirements. Changing or replacing the column will also require that the retention time windows be redetermined.

12.5 Initial Demonstration of Proficiency

12.5.1 Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat the following operations whenever new staff are trained or significant changes in instrumentation are made.

12.5.2 It is suggested that the quality control reference sample concentrate contain each analyte of interest. If this method is to be used for analysis of Chlordane or Toxaphene only, the QC reference sample should contain the most representative multi-component mixture.

12.5.3 Calculate the average recovery and standard deviation of the recoveries of the analytes in each of the four QC reference samples. Compare these values to those published in SW846 method 8081A. If any individual value falls outside the range for accuracy, then the system performance may be unacceptable for that analyte. When comparing the laboratory data to performance data developed from single-laboratory data, certain analytes may be outside the limits, however, the majority should be within the acceptance limits.

12.5.4 Even when the determinative method contains performance data, the development of in-house acceptance limits is strongly recommended.

12.6 Sample Quality control for preparation and analysis- The laboratory must also have procedures for documenting the effect of matrix on method performance (precision, accuracy, and detection limit). At a minimum, this includes the analysis of QC samples including a method blank, a matrix spike, a duplicate, a laboratory control sample (LCS), and the addition of surrogates to each field sample and QC sample. All target analytes are spiked into the LCS and MS/MSD.

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12.6.1 Documenting the effect of the matrix should include the extraction and analysis of a matrix spike/matrix spike duplicate pair per twenty field samples. If insufficient sample is provided by the client, a laboratory control spike/laboratory control spike duplicate pair may be used to evaluate precision.

12.6.2 In-house method performance criteria should be developed using the guidance found in Sec. 8.0 of Method 8000 for procedures for evaluating method performance.

12.6.3 A Laboratory Control Sample (LCS) is included in each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicates a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

12.6.3.1 For Air Force Center for Environmental Excellence (AFCEE) projects, control charts are used to track the performance of LCS recoveries.

All LCS analytes spiked for AFCEE batches are entered into control charts on a quarterly basis, with the charts reviewed and approved by the organics supervisor.

Any out-of-control event detected in the control charts will be documented in a laboratory corrective action report. The following conditions are considered out-of-control events:

- Any one part outside control limits (SOP Table 4).
- Any three consecutive parts outside warning limits.
- Any six consecutive parts such that each part is larger (or smaller) than its immediate predecessor.
- Any eight consecutive parts on the same side of the centerline.

These control charts will be maintained for both solid and aqueous matrices.

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12.6.3.2 For Navy samples, DoD QC limits must be met for all LCS/LCSD/MS/MSD QC samples. Limits are appended to this SOP as Tables D-14 and D-15.

12.6.4 Include a calibration standard after each group of 20 samples in the analysis sequence as a calibration check. Thus, injections of method blank extracts, matrix spike samples and other non-standards are counted in the total. Solvent blanks, injected as a check on cross-contamination, need not be counted in the total. The response for the continuing calibration should be within +/- 15% of the initial calibration (see Sec. 12.2.1). When this calibration verification standard falls out of this acceptance window, the laboratory should stop analyses and take corrective action.

12.6.5 DDT and Endrin are easily degraded in the injection port. Breakdown occurs when the injection port liner is contaminated with high boiling residue from sample injection or when the injector contains metal fittings. Check for degradation problems by injecting a standard containing only 4,4'-DDT and Endrin. Presence of 4,4'-DDE, 4,4'-DDD, Endrin ketone or Endrin aldehyde indicates breakdown. If degradation of either DDT or Endrin exceds 15%, take corrective action before proceeding with calibration.

12.6.5.1 Calculate percent breakdown as follows:

$$\% \text{ breakdown of DDT} = \frac{\text{sum of degradation peak areas (DDD + DDE)}}{\text{sum of all peak areas (DDT + DDE + DDD)}} \times 100$$

$$\% \text{ breakdown of Endrin} = \frac{\text{sum of degradation peak areas (aldehyde + ketone)}}{\text{sum of all peak areas (Endrin + aldehyde + ketone)}} \times 100$$

12.6.5.2 The breakdown of DDT and Endrin should be measured before samples are analyzed and at the beginning of each 12-hour shift. Injector maintenance and recalibration should be completed if the breakdown is greater than 15% for either compound.

12.6.6 Whenever silica gel (Method 3630) or Florisil (Method 3620) cleanups are used, the analyst must demonstrate that the fractionation scheme is reproducible. Batch to batch variation in the composition of the silica gel or Florisil or overloading the column may cause a change in the distribution patterns of the organochlorine pesticides. When compounds are found in two fractions, add the concentration found in the fractions, and correct for any additional dilution.

12.7 Surrogate recoveries: The laboratory must evaluate surrogate recovery data form individual samples versus the surrogate control limits developed by the laboratory.

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Surrogate recovery is calculated as:

$$\text{Recovery (\%)} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) added}} \times 100$$

If recovery is not within in-house surrogate recovery limits (Table 4), the following procedures are necessary.

12.7.1 Check to be sure that there are no errors in the calculations or surrogate solutions. If errors are found, recalculate the data accordingly. Examine chromatograms for interfering peaks and integrated peak areas.

12.7.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and re-analyze the extract.

12.7.3 Some samples may require dilution in order to bring or more target analytes within the calibration range or to overcome significant interferences with some analytes. This may result in the dilution of the surrogate responses to the point that the recoveries cannot be measured. If the surrogate recoveries are available from a less-diluted or undiluted aliquot of the sample or sample extract, those recoveries may be used to demonstrate that the surrogates were within the QC limits, and no further action is required. However, the results of both the diluted and undiluted (or less-diluted) analyses should be provided to the data user.

12.7.4 If no instrument problem is found, the sample should be re-extracted and re-analyzed.

12.7.5 If upon re-analysis, the recovery is again not within limits, report the data as an "estimated concentration". If the recovery is within the limits for the re-analysis, provide the re-analysis data to the data user. If the holding time for the method has expired prior to the re-analysis, provide both the original and re-analysis results to the data user and note the holding time problem.

12.8 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

13.0 Calibration and Standardization:

13.1 Calibration of an analytical instrument involves the delineation of the relationship between the response of the instrument and the amount or concentration of an analyte introduced into the instrument. The graphical depiction of this relationship is often referred to as the calibration curve. In

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order to perform quantitative measurements, this relationship must be established prior to the analysis of any samples, and thus, is termed the initial calibration. The general procedure for performing an initial calibration is described here. Table 2 lists calibration levels used for routine 8081 analysis.

13.1.1 For each analyte and surrogate of interest, prepare calibration standards at a minimum of five different concentrations by adding volumes of one or more stock standards to volumetric flask and diluting to volume with an appropriate solvent. Alternatively, solvent may be measured with the use of a volumetric pipet. A known volume of solvent is then removed from the measured volume, and stock standard solution is added (the same volume as previously removed).

13.1.2 The lowest concentration calibration standard that is analyzed during an initial calibration establishes the method quantitation limit based on the final volume of extract (or sample) described in the preparative method or employed by the laboratory.

13.1.3 The other concentrations should define the working range of the detector or correspond to the expected range of concentrations found in actual samples that are also within the working range of the detector.

13.1.4 For each analyte, at least one of the calibration standards should correspond to a sample concentration at or below that necessary to meet the data quality objectives of the project, which may include establishing compliance with a regulatory or action limit.

13.1.5 Once the standards have been prepared, the initial calibration begins by establishing chromatographic operating parameters that provide instrument performance required to meet the data quality objectives of this method.

13.1.6 Each calibration standard should be introduced into the instrument using the same technique that is used to introduce the actual samples into the gas chromatograph.

13.1.7 External calibration procedure will be used for quantitation of all samples, standards, etc. External standard calibration involves the comparison of instrument responses from the sample to the responses from the target compounds in the calibration standards. Sample peak areas (or peak heights) are compared to peak areas (or peak heights) of the standards. A calibration curve is then plotted. Linear or non-linear calibration (in situations where the analyst knows that the instrument response does not follow a linear model over a sufficiently wide working range) may be used. The curve fit should be the same in the initial calibration as for each sample quantitated against it. The correlation coefficient of the initial calibration should be 0.99 or higher for all analytes and surrogates.

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A hardcopy of the initial calibration, including the raw data, the curve fit type and the resulting correlation coefficient should be maintained by the laboratory.

For AFCEE analyses, the linear least squares regression calculation coefficient ® must be greater than or equal to 0.995 for each analyte. If a non-linear calibration method is used for AFCEE analyses, the coefficient of determination (COD) must be greater than or equal to 0.990.

14.0 Procedure:

14.1 Calibrate the GC system according to section 13 of this SOP. The same GC operating conditions used for the initial calibration must be employed for sample analyses.

14.2 Verify calibration each 12-hour shift by injecting calibration verification standards prior to conducting any sample analyses. Analysts should alternate the use of high and low concentration mixtures of single and multi-component analytes for calibration verification. A calibration standard must also be injected at intervals of not less than once every twenty samples (AFCEE every ten samples) and at the end of the analysis sequence.

14.2.1 The response for each analyte should not exceed a +/-15% difference from that calculated for the initial calibration.

14.2.2 If this criterion is exceeded for any analyte, calculate the average percent difference across all analytes. If the average of the responses for all analytes is within 15%, then the calibration has been verified. The average must include all analytes in the calibration, regardless of whether they are target analytes for a specific project, and the data user must be provided with the calibration verification data or a list of those analytes that exceeded the +/- 15% limit.

14.2.3 If the calibration does not meet the +/- 15% limits (either on the basis of each compound or the average across all compounds), check the instrument operating conditions, and if necessary, restore them to the original settings and inject another aliquot of the calibration verification standard. If the response for the analyte is still not within +/- 15%, then a new initial calibration must be prepared.

14.3 Set the center of the retention time windows to the retention time of the calibration verification standard analyzed at the beginning of the 12-hour shift.

14.4 Inject the Endrin/DDT breakdown check standard. The percent Endrin and the percent DDT breakdown should each be less than 15% as determined in section 12.6.5.

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Inject an aliquot of the concentrated sample aliquot. The injection volume should be the same as that used for the initial and continuing calibration standards. The data system will record the volume injected to the nearest 0.05 ul as well as record the resulting peak size in both area and height units.

14.5 Each sample analysis must be bracketed with an acceptable initial calibration, calibration verification standard(s) (each 12-hour analytical shift), or calibration standards interspersed within the samples. Although the analysis of a single mid-concentration standard (standard mixture or multi-component analyte) will satisfy the minimum requirements, analysts are urged to use different calibration verification standards during organochlorine pesticide analyses. The results from these bracketing standards must meet the calibration verification criteria in Sec. 12.2.1. When a calibration verification standard fails to meet the QC criteria, all samples that were injected after the last standard that last met the QC criteria must be evaluated to prevent mis-quantitations and possible false negative results, and re-injection of the sample extracts may be required. More frequent analyses of standards will minimize the number of sample extracts that would have to be re-injected if the QC limits are violated for the standard analysis. However, if the standard analyzed after a group of samples exhibits a response for an analyte that is above the acceptance limit, and the analyte was not detected in the specific samples analyzed during the analytical shift, then the extracts for those samples do not need to be re-analyzed, as the verification standard has demonstrated that the analyte would have been detected were it present. In contrast, if an analyte above QC limits was detected in a sample extract, then re-injection is necessary to ensure accurate quantitation. If an analyte was not detected in the sample and the standard response is more than 15% below the initial calibration response, then re-injection is necessary to ensure that the detector response has not deteriorated to the point that the analyte would not have been detected even though it was present.

14.6 Sample injections may continue for as long as the calibration verification standards and standards interspersed with the samples meet instrument QC requirements. It is recommended that standards be analyzed after every 10 samples (required after every 20 samples and at the end of a set) to minimize the number of samples that must be re-injected when the standards fail the QC limits. The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria are exceeded.

14.7 If the peak response is less than 2.5 times the baseline noise level, the validity of the quantitative result may be questionable. The analyst should consult with the source of the sample to determine whether further concentration of the sample is warranted.

15.0 Data Analysis and Calculations:

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15.1 Tentative identification of an analyte occurs when a peak from a sample extract falls within the absolute retention time window. Each tentative identification must be confirmed using either a second GC column of dissimilar phase or using another technique such as GC/MS. When results are confirmed using a second GC column of dissimilar phase, the analyst should check the agreement between the quantitative results on both columns once the identification has been confirmed. Calculate the relative percent difference (RPD) between the two results using the formula below.

$$RPD = \frac{|R1 - R2|}{\frac{(R1 + R2)}{2}} \times 100$$

where R1 and R2 are the results on the two columns and the vertical bars in the equation above indicate the absolute value of the difference.

15.1.1 If one result is significantly higher (e.g. >40%), check the chromatograms to see if an obviously overlapping peak is causing an erroneously high result. If no overlapping peaks are noted, examine the baseline parameters established by the instrument data system (or operator) during peak integration.

15.1.2 If no anomalies are noted, review the chromatographic conditions. If there is no evidence of chromatographic problems, report the higher result. This approach is conservative relative to protection of the environment. The data user should be advised of the disparity between the results on the two columns.

15.2 Determine the quantity of each component peak in the sample chromatogram which corresponds to the compounds used for calibration purposes according the following equations:

15.2.1 For aqueous samples

$$\text{Concentration (ug/L)} = \frac{(A)(V_t)(D)}{(V_i)(V_s)}$$

where:

A = ng of analyte detected in the sample (as determined by the initial calibration curve)

V_t = Total volume of the concentration extract (ul)

D = Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution was made, D = 1. The dilution factor is always dimensionless.

V_i = Volume of extract injected (ul). The injection volume for Samples and calibration standards must be the same.

V_s = Volume of the aqueous sample extracted in mL. If units of liters are used for this term, multiply the results by 1000

Using the units specified here for these terms will result in a

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Concentration in units of ng/ml, which is equivalent to ug/L.

15.2.2 For non-aqueous samples

$$\text{Concentration (ug/Kg)} = \frac{(A)(V_t)(D)}{(V_i)(W_s)}$$

where:

A, V_t, D, and V_i are the same as for aqueous samples and

W_s = Weight of sample extracted (g). The wet weight or dry weight may be used, depending upon the specific application of the data. If units of kilogram are used for this term, multiply the units by 1000.

Using the units specified here for these terms will result in a Concentration in units of ng/g, which is equivalent to ug/Kg.

15.2.3 If responses exceed the calibration range of the system, dilute the extract and re-analyze. Peak height measurements are recommended over peak area integration when overlapping peaks cause errors in area integration.

15.3 Quantitation of multi-component analytes. Multi-component analytes present problems in measurement. Suggestions are offered in the following section for handling Toxaphene and Chlordane.

15.3.1 Toxaphene- Toxaphene is manufactured by the chlorination of camphenes. To calculate Toxaphene from GC/ECD results:

15.3.1.1 Adjust the sample size so that the major Toxaphene peaks are 10-70% of full-scale deflection.

15.3.1.2 Inject a Toxaphene standard that is estimated to be within +/- 10 ng of the sample amount.

15.3.1.3 Quantitate Toxaphene using the total area of the Toxaphene pattern or using 4 to 6 major peaks. While Toxaphene contains a large number of compounds that will produce well resolved peaks in a GC/ECD chromatogram, it also contains many other components that are not chromatographically resolved. This unresolved complex mixture results in the "hump" in the chromatogram that is characteristic of the mixture. Although the resolved peaks are important for the identification of the mixture, the area of the unresolved complex mixture contributes a significant portion of the area of the total response. To measure total area, construct the baseline of Toxaphene in the sample chromatogram between the retention times of the first and last eluting Toxaphene components in the standard. In order to use the total area approach, the pattern in the

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sample chromatogram must be compared to that of the standard to ensure that all of the major components in the standard are present in the sample. Otherwise, the sample concentration may be significantly underestimated. Toxaphene may also be quantitated on the basis of 4 to 6 major peaks. The use of 4 to 6 major peaks may avoid difficulties when interferences with Toxaphene peaks are present in the early portion of the chromatogram from compounds such as DDT. Whichever approach is employed should be documented and available to the data user, if necessary. When Toxaphene is determined using the 4 to 6 peak approach, the analyst must take care to evaluate the relative areas of the peaks chosen in the sample and standard chromatogram. It is highly unlikely that the peaks will match exactly, but the analyst should not employ peaks from the sample chromatogram whose relative sizes or areas appear to be disproportionally larger or smaller in the sample compared to the standard. The heights or areas of the 4 to 6 peaks should be summed together and used to determine the Toxaphene concentration. Alternatively, use each peak in the standard to calculate a calibration factor for that peak, using the total mass of Toxaphene in the standard. These calibration factors are then used to calculate the concentration of each corresponding peak in the sample chromatogram and the 4 to 6 resulting concentrations are averaged to provide the final result for that sample.

15.3.2 Chlordane- Technical Chlordane is a mixture of at least 11 major components and 30 or more minor components that is used to prepare specific pesticide formulations. The exact percentage of each component in the technical material is not completely defined and is not consistent from batch to batch. The approach used for evaluating and reporting Chlordane results will often depend on the end use of the results and the analyst's skill in interpreting this multi-component pesticide residue.

15.3.2.1 Inject an amount of Technical Chlordane standard which will produce a chromatogram in which the major peaks are approximately the same size as those in the sample chromatograms.

15.3.2.2 Construct the baseline of Technical Chlordane in the standard chromatogram between the retention times of the first and last eluting Chlordane components. Use this area and the mass of Technical Chlordane in the standard to calculate a calibration factor. Construct a similar baseline in the sample

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chromatogram, measurethe area, and use the calibration factor to calculate the concentration in the sample.

15.3.2.3 The GC pattern of a Chlordane residue in a sample may differ considerably from that of the Technical Chlordane standard. In such instances, it may not be practical to relate a sample chromatogram back to the pesticide active ingredient Technical Chlordane. Therefore, depending on the objectives of the analysis, the analyst may choose to report the sum of all the identifiable Chlordane components as "Chlordane" under the CAS number 57-74-9.

15.4 GC/MS confirmation may be used in conjunction with either single-column or dual-column analysis if the concentration is sufficient for detection by GC/MS.

15.4.1 Full-scan GC/MS will normally require a concentration of approximately 10 ng/ul in the final extract for each single-component compound. Ion trap or selected ion monitoring will normally require a concentration of approximately 1 ng/ul.

15.4.2 The GC/MS must be calibrated for the specific target pesticides when it is used for quantitative analysis.

15.4.3 GC/MS may not be used for confirmation when concentrations are below 1 ng/ul in the extract.

15.4.4 GC/MS confirmation should be accomplished by analyzing the same extract that is used for GC/ECD analysis and the extract of the associated method blank.

15.4.5 The base/neutral/acid extract and the associated blank may be used for GC/MS confirmation if the surrogates and internal standards do not interfere and if it is demonstrated that the analyte is stable during acid/base partitioning. However, if the compounds are not detected in the base/neutral/acid extract, the GC/MS analysis of the pesticide extract should be performed.

15.4.6 A QC reference sample containing the compound must also be analyzed by GC/MS. The concentration of the QC reference sample must demonstrate that those pesticides identified by GC/ECD can be confirmed by GC/MS.

16.0 Method Performance:

16.1 The matrix specific method detection limits (MDLs) should be determined using the following equation:

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With this data, method detection limits were calculate using the formula (3):

$$MDL = S \ t(n-1, 1\text{-alpha} = 0.99)$$

Where:

t(n-1, 1-alpha = 0.99) =	Student's t value for the 99% confidence level with n-1 degrees of freedom
n =	number of replicates
S =	the standard deviation of the replicate analysis

16.2 The accuracy and precision obtainable with this method depends on the sample matrix, sample preparation technique, optional cleanup techniques, and the calibration procedures used. Table 3 contains criteria utilized by the laboratory for determining acceptability of surrogate recoveries, laboratory control spike recoveries, matrix spike/matrix spike duplicates and sample duplicates. The laboratory should determine statistical limits when sufficient data points become available. Per client request, the laboratory may also utilize project specific acceptance criteria.

17.0 Pollution Prevention:

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. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the EPA recommends recycling as the next best option.

18.0 Data Assessment, Acceptance and Corrective Action:

18.1 Sample data is initially reviewed by the analyst performing the analysis.

18.2 Secondary (or peer) review is required for all data before reporting to the client.

18.3 Suggested chromatographic system maintenance- When system performance does not meet the established QC requirements, corrective action is required, and may include one or more of the following.

18.3.1 Splitter connections- For dual columns which are connected using a press-fit Y-shaped glass splitter or a Y-shaped fused-silica connector, clean and deactivate the splitter port insert or replace with a cleaned and deactivated splitter. Break off the first few centimeters of the injection port side of the column. Remove the columns and solvent backflush according to the Manufacturer's instructions. If these procedures fail to eliminate the degradation

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problem, it may be necessary to deactivate the metal injector body and/or replace the columns.

18.3.2 GC injector ports can be of critical concern, especially in the analysis of DDT and Endrin. Injectors that are contaminated, chemically active, or too hot can cause the degradation ("breakdown") of the analytes. Endrin and DDT breakdown to Endrin aldehyde, Endrin ketone, DDD and DDE. When such breakdown is observed, clean and deactivate the injector port, break off at lead 30 cm of the column and remount it. Check the injector temperature and lower it to 205°C, if required. Endrin and DDT breakdown are less of a problem when ambient on-column injectors are used.

18.3.3 Metal injector body- Turn off the oven and remove the analytical columns when the oven has cooled. Remove the glass injection port insert. Lower the injection port temperature to room temperature. Inspect the injection port and remove any noticeable foreign material.

18.3.3.1 Place a beaker beneath the injector port inside the oven. Using a wash bottle, serially rinse the entire inside of the injector port with acetone and then hexane, catching the rinsate in the beaker.

18.3.3.2 Prepare a solution of deactivating agent (Sylon-CT or equivalent) following manufacturer's directions. After all metal surfaces inside the injector body have been thoroughly coated with the deactivation solution, rinse the injector body with toluene, methanol, acetone, then hexane. Reassemble the injector and replace the columns.

18.3.4 Column Rinsing- The column should be rinsed with several volumes of an appropriate solvent. Both polar and non-polar solvents are recommended. Depending on the nature of the sample residues expected, the first rinse might be water, followed by methanol and acetone. Methylene Chloride is a good final rinse and in some cases may be the only solvent required. The column should then be filled with methylene chloride and allowed to stand flooded overnight to allow materials within the stationary phase to migrate into the solvent. The column is then flushed with fresh methylene chloride, drained, and dried at room temperature with a stream of ultrapure nitrogen.

18.3.5 Autosampler syringes should be inspected daily and replaced as needed.

18.3.6 ECD detectors may become contaminated during use. Detectors may be baked overnight with a steady stream of hydrogen. If the signals do not return to useable levels, the detectors should be sent back to the manufacturer for replacement. ECD detectors (inlet,

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housing and exit) should be wiped every six months to ensure that radiation is not leaking. Records of wipe tests are maintained by the radiation safety officer.

18.3.7 Autosampler rinse and Waste vials should be inspected on a daily basis and filled or emptied as needed.

19.0 Waste Management:

Any and all waste generated, through any procedure in the laboratory, is handled according to the Chemical Hygiene, Hazardous Waste Management and Spill Prevention Control and Contingency Plans of Northeast Laboratory Services, Inc.

20.0 References:

SW-846 Method 8081A, Revision 1 December 1996

SW-846 Method 8000B, Revision 2 December 1996

SW-846 Chapter 4 Organic Analytes, Revision 3 December 1996

NELAC Quality System, Chapter 5, June 5, 2003

21.0 Tables, Diagrams, Flowcharts and Validation Data

See attached.

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Table 1 Composite Standard Solutions

Manufacturer	Catalogue #	Mix Name	Concentration	Components
Restek	32291	Organochlorine Pesticide Mix AB #1	200 ug/ml in hexane:toluene (50:50)	alpha-BHC gamma-BHC beta-BHC delta-BHC Heptachlor Aldrin Heptachlor Epoxide Alpha-Chlordane Gamma-Chlordane Endosulfan I 4,4'-DDE Dieldrin Endrin 4,4'-DDD Endosulfan II 4,4'-DDT Endrin Aldehyde Endosulfan sulfate Methoxychlor Endrin ketone
Restek	32000	Pesticide Surrogate Mix	200 ug/ml in Acetone	Tetrachloro-m-xylene Decachlorobiphenyl
Restek	32005	Toxaphene	1000 ug/ml in Hexane	Toxaphene
Restek	32021	Chlordane	1000 ug/ml in Hexane	Chlordane
Eval Mix – Supelco	4-8282	DDT/Endrin Mix	500 ug/ml in Methanol	DDT/Endrin

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Table 2. Calibration Levels (ug/ml)

Component	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Tetrachloro-m-xylene	0.005	0.01	0.025	0.05	0.075	0.100	0.125
Alpha-BHC	0.005	0.01	0.025	0.05	0.075	0.100	0.125
Gamma-BHC	0.005	0.01	0.025	0.05	0.075	0.100	0.125
Beta-BHC	0.005	0.01	0.025	0.05	0.075	0.100	0.125
Heptachlor	0.005	0.01	0.025	0.05	0.075	0.100	0.125
Delta-BHC	0.005	0.01	0.025	0.05	0.075	0.100	0.125
Aldrin	0.005	0.01	0.025	0.05	0.075	0.100	0.125
Heptachlor Epoxide	0.005	0.01	0.025	0.05	0.075	0.100	0.125
Gamma-Chlordane	0.005	0.01	0.025	0.05	0.075	0.100	0.125
Alpha-Chlordane	0.005	0.01	0.025	0.05	0.075	0.100	0.125
Endosulfan I	0.005	0.01	0.025	0.05	0.075	0.100	0.125
4,4'-DDE	0.005	0.01	0.025	0.05	0.075	0.100	0.125
Dieldrin	0.005	0.01	0.025	0.05	0.075	0.100	0.125
Endrin	0.005	0.01	0.025	0.05	0.075	0.100	0.125
4,4'-DDD	0.005	0.01	0.025	0.05	0.075	0.100	0.125
Endosulfan II	0.005	0.01	0.025	0.05	0.075	0.100	0.125
4,4'-DDT	0.005	0.01	0.025	0.05	0.075	0.100	0.125
Endrin aldehyde	0.005	0.01	0.025	0.05	0.075	0.100	0.125
Endosulfan sulfate	0.005	0.01	0.025	0.05	0.075	0.100	0.125
Methoxychlor	0.005	0.01	0.025	0.05	0.075	0.100	0.125
Endrin ketone	0.005	0.01	0.025	0.05	0.075	0.100	0.125
Decachlorobiphenyl	0.005	0.01	0.025	0.05	0.075	0.100	0.125
Technical Chlordane	0.1	0.5	1.0	2.0	3.0	4.0	
Toxaphene	0.1	0.5	1.0	2.0	3.0	4.0	

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Table 3. QC Acceptance Criteria

QC Element	Matrix	Component	Recovery Acceptance Limits (%)	RPD (%)
Surrogates	Aqueous	TCMX	29 – 157	NA
		DCB	25 – 158	NA
	Soil	TCMX	18 – 156	NA
		DCB	25 – 158	NA
LCS/LCSD	Aqueous	Gamma-BHC	56 – 123	15
		Heptachlor	40 – 131	20
		Aldrin	40 – 120	22
		Dieldrin	52 – 126	18
		Endrin	56 – 121	21
		4,4'-DDT	38 - 127	27
	Soil	Gamma-BHC	46 – 127	50
		Heptachlor	35 – 130	31
		Aldrin	34 – 132	43
		Dieldrin	31 – 134	38
		Endrin	42 – 139	45
		4,4'-DDT	23 - 134	50
MS/MSD	Aqueous	Gamma-BHC	56 – 123	15
		Heptachlor	40 – 131	20
		Aldrin	40 – 120	22
		Dieldrin	52 – 126	18
		Endrin	56 – 121	21
		4,4'-DDT	38 - 127	27
	Soil	Gamma-BHC	46 – 127	50
		Heptachlor	35 – 130	31
		Aldrin	34 – 132	43
		Dieldrin	31 – 134	38
		Endrin	42 – 139	45
		4,4'-DDT	23 - 134	50

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Table 4. QC Acceptance Criteria for AFCEE Batches

Method	Analyte	Accuracy Water (%R)	Precision Water (%RPD)	Accuracy Soil (%R)	Precision Soil (%RPD)
SW8081A	α -BHC	60-128	≤ 30	62-125	≤ 50
	β-BHC	66-126	≤ 30	62-127	≤ 50
	δ-BHC	46-136	≤ 30	57-130	≤ 50
	γ-BHC (Lindane)	30-146	≤ 30	59-123	≤ 50
	α-Chlordane	63-123	≤ 30	63-121	≤ 50
	γ-Chlordane	67-120	≤ 30	48-124	≤ 50
	4,4-DDD	50-139	≤ 30	50-139	≤ 50
	4,4-DDE	48-137	≤ 30	68-126	≤ 50
	4,4-DDT	47-138	≤ 30	46-135	≤ 50
	Aldrin	42-138	≤ 30	47-120	≤ 50
	Dieldrin	62-129	≤ 30	67-125	≤ 50
	Endosulfan I	49-120	≤ 30	41-147	≤ 50
	Endosulfan II	42-130	≤ 30	37-141	≤ 50
	Endosulfan Sulfate	54-137	≤ 30	62-135	≤ 50
	Endrin	56-134	≤ 30	61-133	≤ 50
	Endrin Aldehyde	56-137	≤ 30	37-147	≤ 50
	Heptachlor	51-128	≤ 30	51-140	≤ 50
	Heptachlor Epoxide	62-131	≤ 30	66-130	≤ 50
	Methoxychlor	56-150	≤ 30	57-143	≤ 50
	Toxaphene	41-126	≤ 30	31-136	≤ 50
	Surrogates:				
	DCB	32-135		56-132	
	TCMX	33-138		69-124	

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Table 5

Method 8081 Analytical Run Sequence and Corrective Action

QC Parameter	Frequency	Acceptance	Corrective Action
Instrument blank	At beginning of each sequence; after samples whose pesticide concentration exceeds the upper limit of the calibration curve	No pesticides greater than one-half of the reporting level	Reanalyze
Endrin/DDT breakdown	Initially prior to initial calibration curve or CCV	%DDT breakdown to DDE and/or DDD ≤ 15% %Endrin breakdown to endrin ketone and/or endrin aldehyde ≤ 15%	Injection port maintenance followed by reanalysis
Initial calibration	After serious maintenance (new columns, new ECDs) or when a CCV fails	Minimum 6 points with each pesticide correlation coefficient > 0.990.	(1) Instrument maintenance (2) reanalyze
Opening CCV (0.05 or 0.075 ppb standard)	Opening of 12 hour sequence	Each individual pesticide ≤ 15%D on each column; if any individuals fail, the average for all analytes must be ≤ 15%D	(1) Injection port maintenance (2) Reanalyze (3) new initial calibration
LCS/LCSD (second source standards)	One set per extraction batch	See Table 3	(1) if MS/MSD was performed and acceptable, narrate (2) if surrogate recoveries also low but acceptable in blank and samples, narrate (3) if LCS recovery is high, narrate (4) if LCS recovery is low, reextract batch if samples are less than twice the holding time
Surrogates	Every sample, blank, LCS/LCSD, MS/MSD	See Table 3	(1) if surrs are high, narrate (2) if surrs are low, reextract to confirm matrix interference if sample is less than twice the holding time
MS/MSD (second source standards)	One set per twenty samples at request of client; if insufficient sample, use LCS/LCSD as QC indicator	See Table 3	(1) no action if LCS/LCSD meets criteria (2) if LCS/LCSD is also low, follow LCS/LCSD corrective action
Method blank	One per twenty samples Or daily whichever is the more frequent	All pesticide concentrations less than one-half of the reporting level	(1) reextract batch if positive detects and samples also contain positive detects (2) no action if associated samples have no positive detects
Closing or intermediate CCV (0.05 or 0.075 ppb standards)	After 12 hours or every twenty samples whichever is the more frequent (DOD work requires intermediate CCVs every ten samples)	Each individual pesticide ≤ 15%D on each column; if any individuals fail, the average for all analytes must be ≤ 15%D	(1) if positive detects in bracketed samples, reanalyze affected samples (2) if no positive detects, no action as long as %D values are high
Quantitation	For any sample or QC	Reported concentrations must be less than the upper range of the initial calibration curve	Dilute sample and reanalyze

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TABLE D-14. LCS CONTROL LIMITS FOR ORGANOCHLORINE PESTICIDES SW-846 METHOD 8081 WATER MATRIX[24]

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
4,4'-DDD	88	20	25	150	10	170
4,4'-DDE	87	18	35	140	15	160
4,4'-DDT	92	15	45	140	30	155
Aldrin	83	19	25	140	10	155
alpha-BHC	94	11	60	130	50	140
alpha-Chlordane	93	10	65	125	55	135
beta-BHC	96	10	65	125	55	135
delta-BHC	91	15	45	135	30	150
Dieldrin	95	11	60	130	50	140
Endosulfan I[25]	80	10	50	110	40	120
Endosulfan II	79	17	30	130	10	150
Endosulfan sulfate	96	14	55	135	40	150
Endrin	95	13	55	135	45	145
Endrin aldehyde	96	14	55	135	40	150
Endrin ketone	102	8	75	125	70	135
gamma-BHC	82	18	25	135	10	155
gamma-Chlordane	94	11	60	125	50	135
Heptachlor	87	15	40	130	30	145
Heptachlor epoxide	96	11	60	130	50	140
Methoxychlor	103	16	55	150	40	165

[24] A number of sporadic marginal exceedances of the control limits are allowed, depending on the number of analytes spiked in the LCS. Refer to section D.2 and Table D-1 for guidance on the appropriate application of control and ME limits. LCS control limits are not available for Hexachlorobenzene and Toxaphene, although those compounds do appear on the target analyte list for method 8081 (Table C-8 in Appendix DoD-C). Sufficient data to perform statistically significant analyses were not received for those analytes during the LCS study. Additional limits for surrogate compounds can be found in section D.6.

[25] Provisional limits – outlier analyses during the LCS study resulted in LCS-CLs generated with data from fewer than four laboratories. Limits may be adjusted in the future as additional data becomes available.

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TABLE D-15. LCS CONTROL LIMITS FOR ORGANOCHLORINE PESTICIDES SW-846 METHOD 8081 SOLID MATRIX[26]

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
4,4'-DDD	81	18	30	135	10	155
4,4'-DDE	97	10	70	125	60	135
4,4'-DDT	92	16	45	140	30	155
Aldrin	93	16	45	140	30	155
alpha-BHC	93	10	60	125	50	135
alpha-Chlordane	92	10	65	120	55	130
beta-BHC	95	11	60	125	50	135
delta-BHC	94	12	55	130	45	145
Dieldrin	96	10	65	125	55	135
Endosulfan I	74	20	15	135	10	155
Endosulfan II	89	17	35	140	20	160
Endosulfan sulfate	99	12	60	135	50	145
Endrin	97	12	60	135	50	145
Endrin aldehyde	92	18	35	145	20	165
Endrin ketone	100	11	65	135	55	145
gamma-BHC	91	11	60	125	50	135
gamma-Chlordane	96	10	65	125	55	135
Heptachlor	96	15	50	140	35	155
Heptachlor epoxide	98	11	65	130	55	140
Methoxychlor	100	14	55	145	45	155

[26] A number of sporadic marginal exceedances of the control limits are allowed, depending on the number of analytes spiked in the LCS. Refer to section D.2 and Table D-1 for guidance on the appropriate application of control and ME limits. LCS control limits are not available for Hexachlorobenzene, Hexachlorocyclopentadiene, and Toxaphene, although these compounds do appear on the target analyte list for method 8081 (Table C-8 in Appendix DoD-C). Sufficient data were not received for those analytes during the LCS study to perform statistically significant analyses. Additional limits for surrogate compounds can be found in section D.6.

[27] LCS control limits are not available for Aroclors 1221, 1232, 1242, 1248, 1254, 1262, and 1268, although those compounds do appear on the target analyte list for method 8082 (Table C-9 in Appendix DoD-C). Sufficient data to perform statistically significant analyses were not received for those analytes during the LCS study. Additional limits for surrogate compounds can be found in section D.6.

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PAGE:	1 OF40

DOCUMENT HISTORY AND APPROVAL PAGE

TITLE:	SEMIVOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS) CAPILLARY COLUMN TECHNIQUE EPA METHOD 8270C
DEPARTMENTS AFFECTED:	Organics

ISSUE DATE:	REVISION CODE:	SOP #:	REVISION CHANGES:
03/22/01	3	SOP #15	Create an SOP
10/13/01	4	3.3.15	Create an SOP in NELAC Format
10/3/01	D	3.3.15	Update to Correct Revision Code
06/06/02	E	3.3.15	Add Routine Maintenance Procedure to Section 18 and Calculation of Unknowns and Target Compounds.
5/18/04	F	3.3.15	Annual Update
2/11/05	G	3.3.15	Annual Review & Update
01/13/06	H	3.3.15	Annual Review & Update
10/24/06	I	3.3.15	Update to include Navy Assessment Findings
12/19/06	J	3.3.15	Update to include Navy Assessment Findings
02/14/07	K	3.3.15	Annual Review & Update
01/23/08	L	3.3.15	Annual Review & Update
05/20/08	M	3.3.15	Update 13.9

DOCUMENT APPROVAL		
Originator: Joe Casarin	Signature/Date: Joe Casarin 060308	Title Organics Supervisor
Approved by: James Galasyn, Ph.D.	J Galasyn 052008	Chemistry Lab Manager
Reviewed by: Pamela Doughty	Pamela Doughty 6/3/08	Total Quality Manager

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NEL	STANDARD OPERATING PROCEDURE		
TITLE:	SEMIVOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS) CAPILLARY COLUMN TECHNIQUE-EPA METHOD 8270C		
SOP# 3.3.15	REV# M	EFFECTIVE 05/20/08	PAGE 2 OF 40
WRITTEN BY: Joe Casarin	DEPARTMENTS AFFECTED: ORGANICS		

1.0 Test Method:

This standard operating procedure is for EPA Method 8270C, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) Capillary Column Technique.

2.0 Applicable Matrix or Matrices

This standard operating procedure is suitable for the determination of semivolatile organic compounds in ground and surface waters as well as solid matrices.

3.0 Detection Limits

Results of annual MDL studies are on file in the NEL Quality Assurance office. The SVOCs addressed in this SOP, together with their corresponding Practical Quantitation Limits (PQLs) are listed in Table 1:

TABLE 1: PRACTICAL QUANTITATION LIMITS

COMPOUND	WATER UG/L	SOIL mg/KG
N-nitrosodimethylamine	10	0.66
Aniline	10	0.33
Bis(2-chloroethyl)ether	10	0.33
1,3-Dichlorobenzene	10	0.33
1,4-Dichlorobenzene	10	0.33
1,2-Dichlorobenzene	10	0.33
Benzyl alcohol	10	0.66
Bis(2-chloroisopropyl)ether	10	0.66
Hexachloroethane	10	0.33
N-nitrosodi-n-propylamine	10	0.33
Nitrobenzene	10	0.33
Isophorone	10	0.33
Bis(2-chloroethoxy)methane	10	0.33
1,2,4-Trichlorobenzene	10	0.33
4-Chloroaniline	10	0.66
Hexachlorobutadiene	10	0.33

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Hexachlorocyclopentadiene	10	0.33
2-Nitroaniline	50	1.6
Dimethylphthalate	10	0.33
2,6-Dinitrotoluene	10	0.33
3-Nitroaniline	50	1.6
Dibenzofuran	10	0.33
2,4-Dinitrotoluene	10	0.33
Diethylphthalate	10	0.33
4-Chlorophenylphenylether	10	0.33
4-Nitroaniline	50	1.6
N-nitrosodiphenylamine	10	0.33
Azobenzene	50	0.33
4-Bromophenylphenylether	10	0.33
Hexachlorobenzene	10	0.33
Di-n-butylphthalate	10	0.33
Benzidine	20	3.3
Benzylbutylphthalate	10	0.33
3,3-Dichlorobenzidine	20	0.66
Bis(2-ethylhexyl)phthalate	10	0.33
Di-n-octylphthalate	10	0.33
Phenol	10	0.33
2-Chlorophenol	10	0.33
2-Methylphenol (o-cresol)	10	0.33
3,4-Methylphenol (m,p-cresol)	10	0.33
2-Nitrophenol	10	0.33
2,4-Dimethylphenol	10	0.33
Benzoic acid	50	3.3
2,4-Dichlorophenol	10	0.33
4-Chloro-3-methylphenol	20	0.66
2,4,5-Trichlorophenol	10	0.33
2,4,6-Trichlorophenol	10	0.33
2,4-Dinitrophenol	50	1.6
4-Nitrophenol	50	1.6
2-Methyl-4,6-dinitrophenol	50	1.6
Pentachlorophenol	50	1.6
Naphthalene	10	0.33
2-Methylnaphthalene	10	0.33
2-Chloronaphthalene	10	0.33
Acenaphthylene	10	0.33
Acenaphthene	10	0.33
Fluorene	10	0.33
Phenanthrene	10	0.33
Anthracene	10	0.33
Fluoranthene	10	0.33
Pyrene	10	0.33
Benzo(a)anthracene	10	0.33
Chrysene	10	0.33
Benzo(b)fluoranthene	10	0.33
Benzo(k)fluoranthene	10	0.33

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Benzo(a)pyrene	10	0.33
Carbazole	10	0.33
Benzo(g,h,i)perylene	10	0.33
Dibenzo(a,h)anthracene	10	0.33
Indeno(1,2,3-cd)pyrene	10	0.33

3.1 SVOC's for PAH analysis use the base only surrogates and the compounds addressed in this SOP with their corresponding Practical Quantitation Limits are listed in Table 2:

TABLE 2: PRACTICAL QUANTITATION LIMITS

COMPOUND	WATER ug/L	SOIL mg/Kg
Naphthalene	10	0.33
2-Methylnaphthalene	10	0.33
2-Chloronaphthalene	10	0.33
Acenaphthylene	10	0.33
Acenaphthene	10	0.33
Fluorene	10	0.33
Phenanthrene	10	0.33
Anthracene	10	0.33
Fluoranthene	10	0.33
Pyrene	10	0.33
Benzo(a)anthracene	10	0.33
Chrysene	10	0.33
Benzo(b)fluoranthene	10	0.33
Benzo(k)fluoranthene	10	0.33
Benzo(a)pyrene	10	0.33
Indeno(1,2,3-cd)pyrene	10	0.33
Dibenz(a,h)anthracene	10	0.33
Benzo(g,h,i)perylene	10	0.33
Carbazole	10	0.33

3.2 SVOC's for TCLP analysis addressed in this SOP and their corresponding PQLs are listed in Table 3:

TABLE 3: TCLP PQLs

COMPOUND	MDL, ug/L
Cresols (o,m,p)	10
Pyridine	10
2,4-Dinitrotoluene	10

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Hexachlorobutadiene	10
Nitrobenzene	10
Pentachlorophenol	10
2,4,5-Trichlorophenol	10
2,4,6-Trichlorophenol	10
1,4-Dichlorobenzene	10
Hexachloroethane	10
Hexachlorobenzene	10

4.0 Scope and Application

This method is designed to meet the survey and monitoring requirements of the US EPA. The QA/QC requirements are routinely applied to water, ground water, soil and sediment analyzed for semi-volatile compounds (SVOC's) using Gas Chromatography /Mass Spectrometry (GC/MS).

5.0 Summary of Method

5.1 The samples are prepared for analysis by gas chromatography/mass spectrometry (GC/MS) using the appropriate sample preparation (refer to Method 3500) and, if necessary, sample cleanup procedures (refer to Method 3600).

5.2 The semivolatile compounds are introduced into the GC/MS by injecting the sample extract into a gas chromatograph (GC) with a narrow-bore fused silica capillary column. The GC column is temperature-programmed to separate the analytes, which are then detected by a mass spectrometer (MS) connected to the gas chromatograph.

5.3 Analytes eluted from the capillary column are introduced into the mass spectrometer via a jet separator or a direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact (or electron impact-like) spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using a five-point calibration curve.

5.4 The method includes specific calibration and quality control steps that supercede the general requirements provided in Method 8000.

6.0 Definitions

6.1 INTERNAL STANDARD (IS) - Non-target analyte compounds that are similar to the target analytes but are not expected to be found in environmental media (generally isotopically labeled target analytes are used for this purpose) and are added to every standard, quality control sample, and field sample at a known concentration prior to analysis. IS responses are used as the basis for quantitation of target analytes.

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6.2 SURROGATE ANALYTE (SS) - Non-target analytes compounds that are similar in composition and behavior to the target analytes but are not expected to be found in environmental media (often, isotopically labeled target analytes are used for this purpose) and are added to every standard, quality control sample, and field sample at a known concentration prior to preparation and/or analysis. Surrogate responses are used to evaluate the accuracy of the laboratory's performance of the analytical method in a specific sample matrix.

6.3 STOCK STANDARD SOLUTION - A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.

6.4 PRIMARY DILUTION STANDARD SOLUTION - A solution of several analytes prepared in the laboratory from stock standard solutions and diluted as needed to prepare calibration solutions and other needed analyte solutions.

6.5 CALIBRATION STANDARD (CAL) - A solution prepared from the primary dilution standard solution or stock standard solutions and the internal standards and surrogate analytes. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

6.6 CONTINUING CALIBRATION VERIFICATION (CCV) - An analytical standard containing all target analytes, surrogate and internal standard compounds that is used to evaluate the performance of the instrument system with respect to a defined set of method criteria.

6.7 METHOD BLANK (BLK, similarly known as the LABORATORY REAGENT BLANK) - An aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The BLK is used to determine if method analytes or other interferences are present in the laboratory environmental, the reagents, or the apparatus.

6.8 LABORATORY CONTROL SAMPLE (LCS) - The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. When the results of the matrix spike analysis indicates a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

6.9 LABORATORY FORTIFIED SAMPLE MATRIX/SAMPLE MATRIX DUPLICATE (MS/MSD) - An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS/MSB is analyzed exactly like a sample. Its purpose is used to evaluate the accuracy and precision of the laboratory performance of the analytical method in a specific sample matrix.

6.10 SYSTEM PERFORMANCE CHECK COMPOUNDS (SPCC) - Selective analytes from the compound list that are used to check for system instability and

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degradation caused by contaminated lines or active sites in the system. The SPCC's are N-nitroso-di-n-propylamine, hexachlorocyclopentadiene, 2,4-dinitrophenol and 4-nitrophenol.

6.11 CALIBRATION CHECK COMPOUNDS (CCCs) - Selective analytes from the compound list that are used to evaluate the calibration from the standpoint of the system stability and integrity. Variability of these compounds may indicate leaks or reactive sites on the column. The CCCs are: phenol, 1,4-dichlorobenzene, 2-nitrophenol, 2,4-dichlorophenol, hexachlorobutadiene, 4-chloro-3-methyl-phenol, 2,4,6-trichlorophenol, acenapthene, N-nitroso-diphenylamine, pentachlorophenol, fluoranthene, di-n-octylphthalate, benzo-(a)-pyrene.

7.0 Interferences

7.1 Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interference. Determine if the source of interference is in the preparation and/or cleanup of the samples and take corrective action to eliminate the problem.

7.2 All materials used in the procedure shall be demonstrated to be free from interference under the conditions of analysis by the method blank extracted with each batch of samples.

7.3 Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between injections. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross-contamination.

7.4 To further deter column contamination and carryover, the GC septum and glass sleeve (preferably a glass sleeve with wool) should be replaced after a full set of samples has run, or after particularly concentrated or dirty samples have run.

8.0 Safety

8.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined, however each reagent should be treated as a potential health hazard. Exposure to these substances should be reduced to the lowest possible levels. The MSDS sheets for the chemicals used in this procedure must be kept on file in the main chemistry laboratory and read by the analyst before the method is performed.

8.2 Analysts will be provided with a copy of the Chemistry Safety Handbook and are expected to adhere to the pertinent guidelines including but not limited to the use of chemicals in fume hoods, safety goggles, chemical protective lab aprons and proper gloves to handle specific chemicals. No eating in the lab, no mouth pipetting, not wearing protective laboratory equipment in any break areas, and washing hands after handling samples.

8.3 A daily check of fume hood velocity is to be performed and the acceptable range is 80 fpm-120 fpm.

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8.4 Analysts will know where all sinks, eyewashes and emergency showers and spill kits are located in the laboratory and who the appropriate safety personnel are to contact in an emergency.

8.5 Radioactive analyses:

8.5.1 Radiation badges are worn by all chemistry staff, attached to the chest area, at all times and should never leave the building. This ensures that any dose recorded is from just the lab and not any outside source and that the lab is within legal limits for exposure to employees. It is the responsibility of each analyst to wear the badge that is assigned to them and they will be responsible for it during the entire wear period. Upon the start of a new wear period, old badges will be collected and new ones will be assigned by the Radiation Safety Officer (RSO).

8.5.2 There are three levels at which the badges detect energies:
DDE--Deep dose equivalent, whole body (head and trunk), active blood forming organs, gonads. Applies to external whole body exposure and the annual occupational regulatory limits for dose equivalent at a tissue depth of 1 cm is 5000 mrem.
LDE--Eye dose equivalent. Applies to the external exposure to the lens of the eye and the annual occupational regulatory limits for dose equivalent at a tissue depth of 0.3 cm is 15000 mrem.
SDE-- Shallow dose equivalent. Applies to the external exposure of the skin or an extremity and the annual occupational regulatory limits for dose equivalent at a tissue depth of 0.007 cm averaged over an area of 1 cm2 is 50000 mrem.

8.5.3 All radiation exposure records are stored on file and can be viewed by employees at any time.

8.5.4 Arms and extremities need to be covered at all times that radioactive material is being used. Proper Protective Equipment (PPE) should include wearing gloves, lab coats with long sleeves and goggles. Any open wounds should be covered. Personnel exposure should be kept As Low As Reasonably Achievable (ALARA).

8.5.5 Other methods of radiation monitoring include surveys and wipe analysis. Surveys are conducted after the use of any radioactive material and wipes are done on a weekly basis. The RSO will perform these and any clean up actions will be taken to ensure that there is no contamination within the laboratory.

8.5.6 All radioactive samples are listed on the radiation cooler door and are to be stored there at all times unless procedure requires otherwise and it has been checked with the R.S.O.

8.5.7 All radioactive materials must be carried in secondary containment and no trays with holes in the bottom should be used. Upon removal from the cooler, the sign out sheet must be filled out with the following information:

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1) Date, 2) Analyst, 3) Material description, 4) Time removed from cooler, 5) Time work is finished.

8.5.8 Upon completion of work a usage log needs to be filled out to contain the following information: 1) Date, 2) Analyst, 3) Analysis, 4) Amount used. This is just for the original sample. All work areas must be surveyed by RSO before another task can be started in the same area.

8.5.9 All refuse created must be collected and returned to the storage area, including: extracted samples, gloves, disposable glassware, plastic ware, towels used for clean up, all vials containing any form of the radioactive material and any waste that is washed through the instrument during analysis. These materials need to be collected in bags and separated by client. They should be labeled as **residue or processing material** and not as waste. These bags will be given out by the RSO.

8.5.10 Any time radioactive samples are being analyzed for an extended period of time, overnight for example, a sign must be posted to warn employees of the presence of radiation.

8.5.11 For further radiation safety, emergency response and regulations refer to Radiation SOP 3.6.2.

9.0 Equipment and Supplies

9.1 All volumetric glassware will be of Type A quality and cleaned to meet EPA requirements.

9.2 Sample containers:

Soils - 4 or 8 oz. Jars equipped with PTFE-lined screw-cap lids and pre-rinsed with Methylene Chloride (MeCl2) prior to use.

Waters - 1-liter amber bottles equipped with PTFE-lined screw-cap lids and pre-rinsed with MeCl2 prior to use.

9.3 Micro vials, 1.8 ml, amber glass with screw-cap lids and Teflon-lined butyl rubber septa are used for preparing and storing working standards and dilutions.

9.4 Micro vials, 1.8 ml, amber glass with inserts and crimp tops with Teflon-lined butyl rubber septa or crimp tops are used for storing and running samples.

9.5 The GC/MS system is a temperature programmable, GC connected to a Quadrupole or Ion Trap MS via a capillary interface. An automatic injector is attached to the rear injector port. The rear injector port is equipped with a split/ splitless injector interfaced to a 30 meter, 0.25mm ID x 0.25 um, Rtx-5MS capillary column or equivalent.

The GC/MS is equipped with a multi position liquid autosampler.

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9.6 The data handling system is a computer system interfaced to the mass spectrometer and allows continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer has software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundance versus time or scan number. Software is also available that allows integrating the abundance between specified time or scan-number limits. The most recent version of the EPA/NIST Mass Spectral Library is also available. Information is archived by transfer to a server and is backed up by computer personnel.

10.0 Reagents and Standards

10.1 All reagents are pesticide grade or better and are obtained from reputable suppliers.

10.2 Reagent grade water is obtained from a Reverse Osmosis/Ion Exchange system that meets ASTM D1193077 Type II requirements for conductivity.

10.3 Standard preparation follows the procedure outlined in EPA Method 8270C, Section 5.0, and EPA Method 3500B, SW-846, Third Ed., Revision 3,1996, Sections 5.3 and 5.4 with the following exceptions and additions:

Standard stock solutions are obtained from certified chromatography suppliers and dated upon receipt. Second source standards are also obtained from certified suppliers and are used to verify the accuracy of the primary standards.

10.4 All stock standards will be kept at 4 C or as recommended by the standard manufacturer in the original sealed vials until ready to use. Transferred stocks become primary standards and will be replaced every six months or sooner if the stock integrity shows signs of degradation.

10.5 Internal standard solutions - The internal standards recommended are 1,4-dichlorobenzene-d4, naphthalene-d8, acenaphthene-d10, phenanthrene-d10, chrysene-d12, and perylene-d12. These compounds are found in a 4000 ug/ml mix supplied by Restek. Other compounds may be used as internal standards as long as the requirements in Sec. 7.3.2 of EPA method 8270C are met.

10.6 Surrogate standards - The surrogate standards used are nitrobenzene-d5, 2-fluorobiphenyl, and terphenyl-d14 for base/neutral extractable compounds. Phenol-d6, 2,4,6-Tribromophenol, and 2-Fluorophenol are the acid extractable surrogates.

10.7 Matrix spike and laboratory control standards. The matrix spike standard is made using mixes supplied by ECS. The same standard is also used as the laboratory control standard.

10.8 Acetone, hexane, Methylene chloride, isooctane, carbon disulfide, toluene and other appropriate solvents - All solvents are reagent grade or equivalent.

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10.9 All standards and dilutions are entered into the Semi-volatile Standard Logbook. The following information is entered for each standard transferred or diluted.

Date prepared
Analyst
Lot #
Supplier
Standard name/concentration(s)
Comments/dilutions
Usage
Unique identification code

All surrogates, matrix spikes and QC checks are entered into the Semi-volatile Extraction Standard Logbook. The following information is entered for each standard transferred or diluted:

Date prepared
Analyst
Lot #
Supplier
Standard name/concentration(s)
Comments/dilutions
Usage
Unique identification code

11.0 Sample Collection, Preservation, Shipment and Storage

11.1 Sample collection follows the procedure outlined in, Organic Analytes, Sec.4.1 of SW-846, Third Ed., Revision 3, 1996, with the following exception: 4-oz glass jars may be substituted for the 8-oz glass jars.

11.2 All samples shall be labeled with the sample identifier, such as site identification or sample number, and date and time sampled. Other pertinent information can be found on the chain of custody. At a minimum, the sample will be labeled with the NEL sample number.

11.3 All samples are kept at 4 C from the time they are sampled until They are analyzed. Samples should be shipped to the lab on ice or with other cooling device to maintain 4 C.

11.4 Sample holding time:

Waters – 7 days (extraction), 40 days from extraction for analysis
Soils – 14 days (extraction), 40 days from extraction for analysis

All dates are from date of sampling. Samples not extracted or analyzed within holding times are discarded and replaced, or run at the discretion of the client.

11.5 Sample extracts are stored away from light and analyzed within 40 days from extraction.

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11.6 Samples, extracts, and semi-volatile standards are stored in separate refrigerators.

11.7 All samples remaining after analysis are to be either returned to the client or are to be disposed of according to Federal regulations.

12.0 Quality Assurance/Quality Control

12.1 The analyst should demonstrate an initial ability to perform this analysis as outlined in EPA Method 8270C, and performance should be documented in the training form attached to this SOP.

12.2 Laboratory reagent blanks and method blanks, must be analyzed with each analytical batch to demonstrate that all analytical systems, reagents and glassware interference are under control.

12.3 The analyst is permitted to modify GC columns, conditions, or detectors to improve separations, turn-around time, or to lower costs, as long as the analyst can prove equivalency utilizing the QC parameters outlined in EPA Method 8000A.

12.4 The laboratory will demonstrate through the analysis of Proficiency samples that the system is in control. The frequency of these QC checks will be at least semi-annually unless problems develop.

12.5 Laboratory fortified sample matrix (matrix spike) must be performed every 20 samples if there is sufficient sample and is requested by the client.

12.6 Laboratory control samples (blank spikes) must be analyzed at a frequency equivalent to 5% of all samples analyzed or one per sample set. Recoveries should meet the acceptance criteria of EPA Method 8000A, section 8.7. If recoveries do not meet acceptance criteria, the source of the problem should be identified before continuing with the analysis. Standards used for LCS spikes are procured from a second vendor as is used for calibration.

12.7 Calculate the mean surrogate recovery and standard deviation for 25-30 samples for each matrix. The upper and lower control limits are defined as the mean +3 standard deviations. If the values fall outside of this range, then the system must be checked for the source of these errors. Possible problems are: calculation errors, degradation of standards, contamination, or instrument performance. If none of these are the problem, reanalyze the extract. If the surrogate recovery is still not within limits, then report the data as suspect and/or extract and analyze a duplicate sample.

12.8 The analyst may adopt additional QC measures to further ensure data quality.

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13.0 Calibration and Standardization

13.1 Daily Calibration

13.1.1 GC/MS tuning – This standard contains 50 PPM of decafluorotriphenylphosphine (DFTPP) and also contains 50 PPM each of 4,4-DDT, pentachlorophenol, and benzidine in order to verify injection port inertness and GC column performance. See Table 5 for preparation instructions. An acceptable DFTPP needs to be run before every 12 hour period of SVOA analysis. The acceptable criteria for a DFTPP tune are listed in Table 2.

TABLE 2: ACCEPTABLE DFTPP CRITERIA

MASS	ION ABUNDANCE CRITERIA
51	30 TO 60% OF MASS 198
68	LESS THAN 2% OF MASS 69
69	LESS THAN MASS 198
70	LESS THAN 2% OF MASS 69
127	40 TO 60% OF MASS 198
197	LESS THAN 1% OF MASS 198
198	REFERENCE PEAK
199	5 TO 9% OF MASS 198
275	10 TO 30% OF MASS 198
365	GREATER THAN 1% OF MASS 198
441	PRESENT BUT LESS THAN MASS 443
442	40 TO 100% OF MASS 198
443	17 TO 23% OF MASS 442

The acceptance criteria can be checked once the DFTPP analysis has been completed. For enviroquant, load the method DFTPP4.M into the ENVDA screen and then load the correct data file (these being labeled MD_____.D). Click on tuner in the ENVDA drop down menu. Evaluate DFTPP. Select that and then choose Autofind DFTPP. This will evaluate the tune.

13.1.2 All subsequent standards, samples, MS/MSD's and blanks associated with a DFTPP analysis must use the identical mass spectrometer instrument conditions.

13.1.3 The GC/MS tuning standard solution should also be used to assess GC column performance and injection port inertness. Degradation of DDT to DDE and DDD should not exceed 20%. (See Sec. 8.0 of Method 8081 for

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the percent breakdown calculation). Benzidine and pentachlorophenol should be present at their normal responses, and no peak tailing should be visible.

13.1.4 If degradation is excessive or poor chromatography is noted, the injection port may require cleaning or it may be necessary to break off 6-12 in. of the capillary column.

13.2 Initial Calibration - Six calibration standards are prepared in the following concentrations: 10 PPM, 20 PPM, 50 PPM, 80 PPM, 120 PPM, and 160 PPM. See Table 7 for preparation instructions. At least one of the calibration standards should correspond to sample concentration at or below that necessary to meet the data quality objectives of the project. The remaining standards should correspond to the range of concentrations found in actual samples but not exceed the working range of the GC/MS system. Each standard should contain each analyte for detection by this method. These standards must be run before samples can be analyzed.

13.2.1 All target analytes for a particular analysis must be included in the calibration standards. These target analytes may not include the entire list of analytes (Sec. 1.1) for which the method has been demonstrated. However, The laboratory shall not report a quantitative result for a target analyte that was not included in the calibration standards.

13.2.2 Once the DFTPP has run and been shown to be in criteria, the calibration curve points can be analyzed. A sequence is set up which includes the Method SVZZZZZZ.M, where ZZZZZZ is the date of analysis. The GC/MS operating conditions can be found in the method file. See section 14.5.1 for the specific operating parameters.

The calibration curve is analyzed and then quantitated and checked to see if it meets QC criteria. In order for this quantitation to occur, target compounds use specific quantitation ions and are quantitated, along with the surrogates, off of the set of internal standards. This information is listed here:

Internal Standard:	1,4-Dichlorobenzene-d4
Tests and Surrogates:	N-Nitrosodimethylamine
	Pyridine (TCLP)
	2-Fluorophenol (surrogate)
	Phenol-d6 (surrogate)
	Phenol
	Aniline (non-TCL)
	Bis(2-Chloroethyl) ether
	2-Chlorophenol
	1,3-Dichlorobenzene
	1,4-Dichlorobenzene
	1,2-Dichlorobenzene
	Benzyl alcohol
	2-Methylphenol (o-cresol)
	3,4-Methylphenol (m+p-cresol)

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	Bis (2-Chlorosopropyl) ether
	Hexachlolorethane
	N-nitroso-di-n-propylamine
Internal Standard:	Naphthalene-d8
Targets and Surrogates:	Nitrobenzene-d5 (surrogate)
	Nitrobenzene
	Isophorone
	2-Nitrophenol
	2,4-Dimethylphenol
	Bis (2-Chloroethoxy) methane
	Benzoic Acid
	2,4-Dichlorophenol
	1,2,4-Trichlorobenzene
	Nahthalene
	4-Chloroaniline
	Hexachlorobutadiene
	4-Chloro-3-methylphenol
	2-Methylnaphthalene
Internal Standard:	Acenaphthene-d10
Targets and Surrogates:	Hexachlorocyclopentadiene
	2,4,6-Trichlorophenol
	2,4,5-Trichlorophenol
	2-Fluorobiphenyl (surrogate)
	2-Chloronaphthalene
	2-Nitroaniline
	Dimethylphthalate
	Acenaphthylene
	2,6-Dinitrotoluene
	3-Nitroaniline
	Acenaphthene
	2,4-Dinitrophenol
	4-Nitrophenol
	Dibenzofuran
	2,4-Dinitrotoluene
	Diethylphthalate
	Fluorene
	4-Chlorophenylphenyl ether
	4-Nitroaniline
	Azobenzene
	2,4,6-Tribromophenol (surrogate)
Internal Standard:	Phenanthrene-d10
Targets:	4,6-Dinitro-2-methylphenol
	N-Nitrosodiphenylamine
	4-Bromophenylphenylether
	Hexachlorobenzene
	Pentachlorophenol
	Phenanthrene
	Anthracene

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Carbazole
Di-n-butylphthalate
Fluoranthene

Internal Standard:	Chrysene-d12
Targets and Surrogate:	Benzidine
	Pyrene
	Terphenyl-d14 (surrogate)
	Benzylbutylphthalate
	Benzo (a) anthracene
	3,3-Dichlorobenzidine
	Chrysene
	Bis (2-Ethylhexylphthalate)
	Di-n-octylphthalate
	Indeno (1,2,3-cd) pyrene)

Internal Standard:	Perylene-d12
Targets:	Benzo (b) fluoranthene
	Benzo (k) fluoroanthene
	Benzo (a) pyrene
	Dibenz (ah) anthracene
	Benzo (ghi) perylene

The primary and secondary ions used for each target compound and surrogate are listed in Table 4.

Once the calibration points have been quantitiated, the curve is updated using the UPDATE LEVELS option in the InitCal menu in the ENVDA menu. From this information you can obtain a printout of the curve displaying an RRF, an average RRF and percent RSD for each analyte.

The RSD for each target analyte should be less than or equal to 15%. However, the RSD for each calibration check compound (CCC) must be less than or equal to 30%. There are thirteen calibration check compounds:

Phenol
1,4-Dichlorobenzene
2-Nitrophenol
2,4-Dichlorophenol
Hexachlorobutadiene
4-Chloro-3-methylphenol
2,4,6-Trichlorophenol
Acenaphthene
N-Nitrosodiphenylamine
Pentachlorophenol
Fluoranthene
Di-n-Octylphthalate
Benzo (a) pyrene

There are also four system performance check compounds (SPCCs):

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N-nitroso-di-n-propylamine
Hexachlorocyclopentadiene
2,4-Dinitrophenol
4-Nitrophenol

These compounds must meet a minimum average RRF of 0.050.

If the RSD of a target analyte is over 15%, the curve can still be used if the linear regression coefficient is greater than or equal to 0.995 or the quadratic coefficient is greater than or equal to 0.990.

Due to software limitations, the initial calibration printout does not rotate the regression coefficients. For the analytes with RSDs over 15%, a plot containing an analyte's regression is printed out and is supplied to the client if a QC Level 4 package is requested.

A second source verification standard is then analyzed after the initial calibration. The verification standard is at a concentration of 50 ug/ml. It is prepared like the 50 ug/ml calibration curve oint except with a different standards supplier (see Tables 8 and 9).

The verification standard is then quantitated and computed to the initial calibration. Each analytes' value must be within +/- 25% of the expected value.

If the calibration curve and the second source verification standard are in criteria and there is still time within the 12-hour clock, samples may be analyzed.

13.3 Continuing Calibration – A calibration check must be performed every twelve hours after the analysis of the DFTPP. The calibration check is at a concentration of 50 ug/ml. It is prepared (Table 7) exactly like the 50 ug/ml calibration curve point.

The calibration check is then quantitated and compared to the initial calibration. The CCCs must have a percent difference of less than or equal to 20%. The SPCCs also must meet the same criteria as in the curve.

The areas for the internal standards in the calibration check must be within –50% to 100% of the 50 ug/ml calibration point of the most recent initial calibration.

If the above criteria is met then sample analysis may be performed.

13.4 Method Blank – A method blank must be extracted with each batch of samples of similar matrix and analyzed on the GC/MS along with the corresponding samples. The method blank is either extracted every 20 samples of a certain matrix or when samples are extracted by the same procedure; whichever is more frequent.

A method blank is deemed acceptable if it contains less than the Practical Quantitation Limits (PQL) of each analyte.

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If analytes in the method blank are greater than the reporting level, corrective action must be taken before samples can be analyzed. This can take the form of blank reanalysis.

13.5 Surrogates – There are six surrogates. There are three base/neutrals (Nitrobenzene-d5, 2-Fluorobiphenyl and Terphenyl-d14) and three acids (Phenol-d6, 2-Fluorophenol and 2,4,6-Tribromophenol). The recoveries for these surrogates are automatically calculated when an analysis is quantitated. They can be found on the first page of the quant report.

All of the surrogate recoveries must meet the required acceptance limits which can be found in Table 10.

When Navy samples are analyzed the surrogate recoveries must meet the acceptance criteria listed in the D tables of the DOD QSM, Version 3. They can be found in Table 12.

If surrogate recoveries are not in criteria, the sample (or blank, etc) should be re-extracted if possible. If the re-extraction is in criteria and was within hold time, only the re-extract needs to be reported. If the hold time has expired, both analyses need to be reported. If the original sample and the re-extract both are out of surrogate criteria, a matrix effect is suspected, and will be noted as such on the report of analysis. Any reported surrogates outside of QC limits will be flagged and noted.

If one needs more information regarding surrogate acceptance, the GC/MS supervisor should be contacted.

13.6 Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD) and Matrix Spike/Matrix Spike Duplicate (MS/MSD) – An LCS/LCSD needs to be extracted with each batch of samples of a similar matrix under the following circumstances, whichever is more frequent: Every 20 samples of the same matrix or for every sample batch extracted.

MS/MSDs must be extracted with each similar matrix batch of up to 20 samples. An LCS/LCSD can be substituted if there is insufficient sample to perform an MS/MSD.

Recovery limits for the LCS/LCSD and MS/MSD can be found in Attachments 1, 2 and 3, for waters, soils and Navy, respectively.

In the case of LCS/LCSDs and MS/MSDs that are extracted with Navy samples, the recoveries of the spiked analytes must meet the QC acceptance criteria specified by the DOD QSM, Version 3. They are appended to this SOP as attachments

13.7 Internal Standard Responses – Internal standard areas and compound retention times have to be checked for every blank, sample, and QC sample. In the method file the retention times are set up to detect an analyte if it is within +/- 0.06 RRT units of the compound in the midpoint standard of the initial calibration or, if applicable, that day's calibration check. Also, the internal standard areas of

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a sample need to be within –50 to 100% of the daily calibration check. If the areas are out of criteria, the sample needs to be reanalyzed. If the reanalysis is within criteria, only report the reanalysis. Otherwise report the original and the reanalysis.

MS/MSD samples do not need to be reanalyzed if they don't meet internal standard area criteria.

13.8 If any analyte concentrations exceed the upper limit of the calibration curve, the extract must be diluted and reanalyzed to bring all concentrations within the curve range.

13.9 The client may request that NEL perform a library search on any unidentified peaks present in the chromatogram, either upon submission of the samples, or at a time after the data has been analyzed and reported. All raw data files are archived for a minimum of five years and can be accessed during this time period.

The library search generates what are commonly known as tentatively-identified compounds, or "TICs." NEL uses a current NIST 75,000 compound mass spectral database as well as an Agilent computer algorithm program to generate possible mass spectral matches.

The NEL mass spectral interpretation specialist (MSIS-defined by EPA as having a minimum of two years GC/MS experience as well as pertinent course-work), compares the unknown sample spectrum to the reference spectrum. Ions with relative intensities greater than 10% of the most abundant ion in the reference spectrum should be present in the sample spectrum along with the molecular ion. The major ions in the sample spectrum should also correlate with ± 20% of their intensity compared to their intensity in the reference spectrum. The MSIS has the final decision as these determinations are subjective and must take into account many factors, including the matrix of the sample, interferences, etc.

It is NEL policy to perform library search at the request of the client with up to 20 TICs reported, each with responses greater than 10% of the closest internal standard. If there is not a good identification with CAS number reported, the MSIS attempts to give the most information possible (i.e. branched alkane, unknown chlorinated compound, phthalate ester, etc). An estimated concentration is also reported which is based upon a response factor of 1.000 with the nearest internal standard.

NEL also performs and reports a library search on the associated method blank at no additional charge.

13.10 If in any circumstances manual integrations are performed (on calibration standards, sample, etc.), they must be done according to the procedure outlined in NEL SOP 3.1.7; Standard Operating Procedure Proper Documentation Regarding Manual Integration of Chromatographic Peaks.

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Printouts are made of the peak being integrated before and after the manual integration. A reason or a reason code, reference in Section 6.0 of NEL SOP 3.1.7, must be provided explaining why the manual integration was needed. The analyst then must date and initial the after manual integration printout. All of the manual integration data is then forwarded to the organics supervisor (or designee) for his/her review and approval signature.

The manual integration chromatograms are filed with the batch QC. In the case of QC Level 4 data package, this documentation will be included with the data package given to the client if the manual integrations pertain to any of the associated data analyses in the package.

14.0 Procedure

14.1 Samples are prepared according to the procedure in EPA Method 8270C, Section 7, with the following exceptions and additions:

14.1.1 Separatory funnel liquid/liquid extraction method 3510C is used for the extraction of water samples. An alternative may be by continuous liquid/liquid extraction method 3520.

14.1.2 Ultrasonic extraction (EPA Method 3550B) is used for solid samples. The solvent used is Methylene chloride. Sodium sulfate is added to the sample until it is free flowing. An alternative at the request of the client is soxhlet extraction (EPA Method 3540C).

14.2 The addition of 1 ml of a 100-ug/ml solution of surrogate standards results in a surrogate concentration of 100 ug/sample. The B/N surrogates are nitrobenzene-d5, 2-fluorobiphenyl, and terphenyl-d14. For acid extractable compounds, the surrogates used are 2-fluorophenol, phenol-d5, and 2,4,6-tribromophenol.

14.3 For EPA Method 8270C, the matrix spike and lab control samples are spiked with all of the target compounds. All of the compounds are at a concentration of 100 ug/ml.

14.4 Appropriate volumes of 4000 ug/ml Internal Standard are added to the sample extract to give a 40 ug/L final concentration for analysis. For example, 1 ul of istd is added to 100 ul of extract for an undiluted run.

See the Prep SOP 3.3.29 for additional information.

14.5 INSTRUMENT SETUP

14.5.1 GC/MS operating conditions.

Gas Chromatograph

Parameters	Method 8270	Method DFTPP
Initial temperature	35 °C	150 °C

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Holding time	3 minutes	3 minutes
Ramp 1	25 °C/minute	20 °C/minute
Final temperature	245 °C	300 °C
Final time	0 minutes	0.0 minute
Ramp 2	6 °C/minute	
Final temperature	330 °C	
Final time	5.00 minutes	
Injector temperature	250 °C	250 °C
Carrier gas flow	1-2 ml/minute	1-2 ml/minute
Column Head pressure	10 psi	10 psi

Mass Spectrometer

Parameters	DFTPP
Mass range	35 to 500 AMU
Seconds/scan	1.0
Filament/multiplier delay	180 seconds
Peak threshold	2500 counts
Mass defect	35
Ionization mode	EI

Autosampler

Parameters	Method 8270
Syringe solvent wash	3X
Syringe sample wash	1X
Sample pumps	3X
Amount injected	1 ul
Air plug after sample	1 ul
Hold time in injector (prior to injection)	2.0 seconds
Hold time in injector (after injection)	1.5 seconds

15.0 Data Analysis and Calculations

15.1 Concentrations of Calibrated Compounds:

$$C(x) = \frac{A(s) * C(is)}{A(s) * RRF} * DF$$

Where:
C(s) = Concentration of compound (ug/L)
C(is) = Concentration of associated internal standard (ug/L)
DF = Dilution Factor.
A(is) = Area of quantitation ion for associated internal standard
A(s) = Area of quantitation ion for compound
RRF = Average Relative Response Factor from six-point initial calibration

The concentration of the sample is then corrected for extraction. The raw concentration (in ug) is multiplied by the final extract volume (ml), and then it is

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divided by the volume of water extracted (in L) or the weight of soil extracted (in kg). The resulting concentration is given in ug/L for waters and ug/kg for soils.

$$C(f) = \frac{\text{(final extract volume(ml)) X C(s)}}{\text{(Initial volume (L) or Initial weight (Kg))}}$$

15.2 Concentration of Unknowns:
Concentrations of tentatively identified compounds (TIC) are calculated using the following formula with an assumed response factor (RF) of 1 at the client's request:

$$C(x) = \frac{C(is)*A(x)}{A(is) * DF}$$

Where:
C(x) = Concentration of Unknown (ug/L)
C(is)= Concentration of Internal Standard
A(x) = Concentration of Unknown
A(is)= Area of associated internal standard
DF = Dilution factor

15.3 Relative percent difference (RPD)- percent difference between a sample and its duplicate and percent recovery (%R)-percent recovered of known amount in LCS or MS

14.1 $$RPD = \frac{100(BNA1 - BNA\ 2)}{(BNA1 + BNA2)/2}$$

$$\%R = \frac{Cs - Cu}{Cn} \times 100$$

Where:
Cs = Measured concentration of the spiked sample aliquot
Cu = Measured concentration of the unspiked sample
Cn = The nominal concentration of the spike aliquot

16.0 Method Performance

The specifications contained in this SOP are based on the requirements in US EPA Method 8270C, sec. 9.0.

17.0 Pollution Prevention

Care is used to minimize the amount of solvents used. All solvents, standards and samples are properly labeled and placed in a labeled hazardous waste container and disposed of according to regulations.

18.0 Data Assessment and Acceptance Criteria for Quality Control Measures and Corrective Action

18.1 The correlation coefficient of the calibration curve must be at least <15% of the Average response, or >0.995 linear response, or >0.990 quadratic response.

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18.2 An HPCHEM method will be generated from the calibration files and named and archived with a unique ID that includes the date the calibration files were analyzed. The analyst will review the calibration files and curve to make sure all peaks are properly integrated and identified, that no "outlier" data points or "overloaded detector" points need to be discarded, and that the curves meet the minimum required correlation coefficients.

18.3 Sample areas must fall within the range of the areas of the calibration standards. One standard must be at or below the PQL concentration. If areas exceed the calibration range, the extract must be diluted and reanalyzed.

18.4 When out-of-control data is observed, a NEL corrective action form is filled out, the source of the problem is identified and documented on the form and all necessary actions are implemented to correct the problem. This may include degradation of surrogate compounds, daily CCC, SPCC or sample blank contamination. The samples may need reanalyzing if necessary under corrected conditions. The solution to the problem is also documented on the form and submitted to management for approval.

18.5 Routine Maintenance is performed on the GC/MS instruments. The frequency is dependent on the type and quantity of samples that are run. When particularly dirty soils or sludges are run, the septum and a split glass sleeve with a carbo-frit insert should be replaced and a small amount of column on the injector side should be clipped. The ion source needs routine cleaning when usage-dependant surface deposits degrade the ion source function below user determined limits. This may be required anywhere from 2 to 6 months.

19.0 Waste Management

Please refer to the NEL Chemical Hygiene Plan and Spill Prevention Control and Prevention Plan for information regarding waste management.

20.0 References

EPA Method 8270C

Current NIST Library

NELAC Quality System, Chapter 5, June 5, 2003

21.0 Tables, Diagrams, Flowcharts and Validation Data

Characteristic Ions for this SOP are listed in Table 4:

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TABLE 4: CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

COMPOUND	Primary Ion	Secondary Ion(s)
N-nitrosodimethylamine	42	74,44
Aniline	93	66,65
Bis(2-chloroethyl)ether	93	63,95
1,3-Dichlorobenzene	146	148,111
1,4-Dichlorobenzene	146	148,111
1,2-Dichlorobenzene	146	148,111
Benzyl alcohol	108	79,77
Bis(2-chloroisopropyl)ether	45	77,121
Hexachloroethane	117	201,199
N-nitrosodi-n-propylamine	70	42,101,130
Nitrobenzene	77	123,65
Isophorone	82	95,138
Bis(2-chloroethoxy)methane	93	95,123
1,2,4-Trichlorobenzene	180	182,145
4-Chloroaniline	127	129,65,92
Hexachlorobutadiene	225	223,227
Hexachlorocyclopentadiene	237	235,272
2-Nitroaniline	65	92,138
Dimethylphthalate	163	194,164
2,6-Dinitrotoluene	165	63,89
3-Nitroaniline	138	108,92
Dibenzofuran	168	139
2,4-Dinitrotoluene	165	63,89
Diethylphthalate	149	177,150
4-Chlorophenylphenylether	204	206,141
4-Nitroaniline	138	65,108,92,80,39
N-nitrosodiphenylamine	169	168,167
Azobenzene	77	105,182
4-Bromophenylphenylether	248	250,141
Hexachlorobenzene	284	142,249
Di-n-butylphthalate	149	150,104
Benzidine	184	92,185
Benzylbutylphthalate	149	91,206
3,3-Dichlorobenzindine	252	254,126
Bis(2-ethylhexyl)phthalate	149	167,279
Di-n-octylphthalate	149	167,43
Phenol	94	65,66
2-Chlorophenol	128	64,130
2-Methylphenol (o-cresol)	107	108,77,79,90
3,4-Methylphenol (m,p-cresol)	108	107,77,90
2-Nitrophenol	139	109,65
2,4-Dimethylphenol	122	107,121
Benzoic acid	122	105,77
2,4-Dichlorophenol	162	164,98
4-Chloro-3-methylphenol	107	144,142

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2,4,5-Trichlorophenol	196	198,97,132,99
2,4,6-Trichlorophenol	196	198,200
2,4-Dinitrophenol	184	63,154
4-Nitrophenol	139	109,65
2-Methyl-4,6-dinitrophenol	198	51,105
Pentachlorophenol	266	264,268
Naphthalene	128	129,127
2-Methylnaphthalene	142	141
2-Chloronaphthalene	162	127,164
Acenaphthylene	152	151,153
Acenaphthene	154	153,152
Fluorene	166	165,167
Phenanthrene	178	179,176
Anthracene	178	176,179
Fluoranthene	202	101,203
Pyrene	202	200,203
Benzo(a)anthracene	228	229,226
Chrysene	228	226,229
Benzo(b)fluoranthene	252	253,125
Benzo(k)fluoranthene	252	253,125
Benzo(a)pyrene	252	253,125
Benzo(g,h,i)perylene	276	138,277
Dibenzo(a,h)anthracene	278	139,279
Indeno(1,2,3-cd)pyrene	276	138,227
Carbazole	167	139
Pyridine	52	51,78
2-Fluorophenol	112	64
Phenol-d6	99	42,71
Nitrobenzene-d5	82	54,128
2-Fluorobiphenyl	172	171
2,4,6-Tribromophenol	141	62,111
Terphenyl-d14	244	122,212

TABLE 5: DECAFLUOROTRIPHENYLPHOSPHINE (DFTPP) TUNING STANDARD

Supplier	Standard	Initial Conc. (ug/ml)	Volume Added (ul)	Final Conc. (ug/ml)
Restek	GC/MS Tuning Mixture	1000	50	50
Fisher Scientific	MeCl2	-	950	-

Final Volume: 1.0 ml
Final Concentration: 50 ug/ml

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TABLE 6: SVOA STOCK SOLUTION

Supplier	Standard	Initial Conc. (ug/ml)	Volume Added (ul)	Final Conc. (ug/ml)
Restek	Carbazole	1000	200	200
Restek	8270 Calibration Mix #1	2000	100	200
Restek	8270 Calibration Mix #2	2000	100	200
Restek	8270 Calibration Mix #3	2000	100	200
Restek	8270 Calibration Mix #4	2000	100	200
Restek	8270 Calibration Mix #5	2000	100	200
Restek	Acid Surrogate Mix	10000	40	400
Restek	BN Surrogate Mix	5000	80	400
Fisher Scientific	MeCl2	-	180	-

Final Volume: 1.0 ml
Final Concentration: 200 ug/ml except for the acid and BN surrogates which are at 400 ug/ml.

TABLE 7: INITIAL CALIBRATION CURVE

Final Conc. (ug/ml)	Ul Added of 200 ug/ml SVOA Stock Solution	Ul Added of 4000 ug/ml SVOA Internal Standard	Ul Added of MeCL2	Final Volume (ul)
10	5	1.0	95	101
20	10	1.0	90	101
50	25	1.0	75	101
80	40	1.0	60	101
120	60	1.0	40	101
160	80	1.0	20	101

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TABLE 8: SECOND SOURCE STOCK

SUPPLIER	STANDARD	INITIAL CONC. (ug/L)	VOLUME ADDED (ul)	FINAL CONC. (u/gml)
Accustandard	Base-Neutral Mixture	2000	100	200
Accustandard	Benzidine Mixture	2000	100	200
Accustandard	Acid Composite Mixture	2000	100	200
Accustandard	Composite #2	2000	100	200
Accustandard	Carbazole	1000	200	200
Accustandard	Surrogate Standard	4000	50	200
Fisher	MeCl2	-	350	-

Final Volume: 1.0 ml
Final Concentration: 200 ug/ml

TABLE 9: SECOND SOURCE VERIFICATION STANDARD

FINAL CONC. (ug/ml)	Ul Added of 200 ug/ml Second Source Stock	Ul added of 4000 ug/ml SVOA Internal Standard	Ul added of MeCl2	FINAL VOLUME (ul)
50	25	1.0	75	101

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TABLE 10: SURROGATE RECOVERIES

Surrogate	Soil Recovery Limits (%)	Water Recovery Limits (%)
2-Fluorophenol	25-121	21-100
Phenol-d6	24-113	10-94
Nitrobenzene-d5	23-120	35-114
2-Fluorobiphenyl	30-115	43-116
2,4,6-Tribromophenol	19-122	10-123
Terphenyl-d14	18-137	33-144

TABLE 11: METHOD 8270 ANALYTICAL RUN SEQUENCE AND CORRECTIVE ACTION

QC Parameter	Frequency	Acceptance	Corrective Action
DFTPP	Initially prior to initial calibration curve or CCV	See Table 2	Reanalyze or retune instrument
Initial Calibration	After serious maintenance (source cleaning, etc) or when the CCV fails.	SPCCs avg RF≥; 0.050, CCCs ≤ 30%D. each analyte ≤ 15%D. Refer to Section 13.2	(1) Reanalyze (2) Instrument maintenance
Second source Calibration Verification	Once after the curve, prior to sample analysis	All analytes within +/- 25% of the expected value	(1) Reanalyze (2) New initial calibration
CCV	Once per 12 hour sequence, prior to sample analysis	CCCs ≤ 20%D; SPCCs RF≥ 0.050	(1)Reanalyze (2)New initial calibration
ISs	Each analysis	Internal standard area within –50% to 100% of last CCV for each IS	Reanalysis of sample
Surrogates	Every sample, blank, LCS/LCSD, MS/MSD	See Table 8	(1) Reanalyze (2) Re-extract and analyze sample (3) If reanalysis out, report both
Method Blank	One per prep batch	No analytes > 1/2 PQL	(1) Investigate source of contamination (2) Reanalysis (3) Re-extract blank and associated samples
LCS/LCSD (second source standards)	One set per extraction batch	See Attachment 1 for waters; See Attachment 2 for soils	(1) If MS/MSD was performed and acceptable narrate. (2) If surrogate recoveries also low but acceptable in blank and samples, narrate

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			(3) If LCS recovery is high, narrate (4) If LCS recovery is low, re-extract batch
MS/MSD (second source standards)	One set per 20 samples at client request; if insufficient sample, use LCS/LCSD	See Attachment 1 for waters; See Attachment 2 for soils	(1) No action if LCS/LCSD meets criteria. (2) If LCS/LCSD is also unacceptable, re-extract the samples
Quantitation	For any sample or QC	Reported concentrations must be less than the upper range of the calibration curve	Dilute sample and reanalyze

TABLE 12: SURROGATE RECOVERIES FOR NAVY SAMPLES

SURROGATE	SOIL RECOVERY LIMITS (%)	WATER RECOVERY LIMITS (%)
2-Fluorophenol	35-105	20-100
Phenol-d6	40-100	10-94
Nitrobenzene-35	35-100	40-110
2-fluorobiphenyl	45-105	50-110
2,4,6-Tribromophenol	35-125	40-123
Terphenyl-d14	30-125	50-135

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Semivolatile Soil
LCS/LCSD Concentration Summary

Lab Name:Northeast labs

Analyst: JJC

File LCS:M7322 File LCSD:M7323

Date Extracted:08/15/06 Date Analyzed:08/17/06

Compound	Spike Added (mg/Kg)	LCS Concentration (mg/Kg)	LCS % Rec.	LCSD Concentratio (mg/Kg)	LCSD % Rec.	% RPD	QC Limits RPD	QC Limits Rec.
N-Nitrosodimethylamine	1.67	0.84	50	0.95	57	13	20	30-150
pyridine	1.67	1.28	77	1.21	72	7	20	30-150
Phenol	1.67	1.19	71	1.14	68	4	20	5-112
Aniline	1.67	1.02	61	1.02	61	0	20	30-150
Bis(2-chloroethyl)ether	1.67	1.27	76	1.24	74	3	20	12-158
2-Chlorophenol	1.67	1.18	71	1.14	68	4	20	23-134
1,3-Dichlorobenzene	1.67	1.26	75	1.24	74	1	20	D-172
1,4-Dichlorobenzene	1.67	1.24	74	1.18	71	4	20	20-124
1,2-Dichlorobenzene	1.67	1.29	77	1.24	74	4	20	32-129
2-Methylphenol (o-cresol)	1.67	1.28	77	1.25	75	3	20	30-150
3,4 Methylphenol (m+p cresol)	1.67	1.39	83	1.35	81	2	20	30-150
Bis-(2-Chloroisopropyl)ether	1.67	1.17	70	1.12	67	4	20	30-150
Benzyl Alcohol	1.67	1.20	72	1.14	68	6	20	30-150
Hexachloroethane	1.67	1.34	80	1.29	77	4	20	40-113
Nitrobenzene	1.67	1.22	73	1.17	70	4	20	35-180
Isophorone	1.67	1.21	72	1.14	68	6	20	21-196
2-Nitrophenol	1.67	1.20	72	1.18	71	1	20	29-182
2,4-Dimethyl phenol	1.67	1.19	71	1.18	71	0	20	32-119
Benzoic Acid	1.67	1.02	61	0.99	59	3	20	30-150
Bis(2-chloroethoxy)methane	1.67	1.25	75	1.23	74	1	20	33-184
N-nitroso-di-n-propylamine	1.67	1.23	74	1.14	68	8	20	D-230
2,4-Dichlorophenol	1.67	1.27	76	1.23	74	3	20	39-135
1,2,4-Trichlorobenzene	1.67	1.26	75	1.26	75	0	20	44-142
Naphthalene	1.67	1.19	71	1.13	68	4	20	21-133
4-Chloroaniline	1.67	1.21	72	1.17	70	3	20	30-150
Hexachlorobutadiene	1.67	1.41	84	1.39	83	1	20	24-116
4-Chloro-3-methyl phenol	1.67	1.18	71	1.19	71	0	20	24-116
2-Methylnaphthalene	1.67	1.28	77	1.22	73	5	20	30-150
2,4,6-Trichlorophenol	1.67	1.33	80	1.27	76	5	20	37-144
2,4,5-Trichlorophenol	1.67	1.45	87	1.31	78	11	20	30-150
2-Chloronaphthalene	1.67	1.38	83	1.25	75	10	20	30-150
2-Nitroaniline	1.67	1.11	66	1.09	65	2	20	30-150
Dimethylphthalate	1.67	1.29	77	1.24	74	4	20	D-112
2,6-Dinitrotoluene	1.67	1.12	67	1.03	62	8	20	50-158
Acenaphthylene	1.67	1.28	77	1.19	71	8	20	33-145
3-Nitroaniline	1.67	1.17	70	1.10	66	6	20	30-150
Hexachlorocyclopentadiene	1.67	1.54	92	1.56	93	1	20	30-150
Acenaphthene	1.67	1.30	78	1.19	71	9	20	47-145
2,4-Dinitrophenol	1.67	1.24	74	1.13	68	8	20	D-191
4-Nitrophenol	1.67	1.14	68	1.03	62	9	20	D-132
Dibenzofuran	1.67	1.40	84	1.29	77	9	20	30-150
2,4-Dinitrotoluene	1.67	1.22	73	1.13	68	7	20	39-139
Diethylphthalate	1.67	1.40	84	1.27	76	10	20	40-160
Fluorene	1.67	1.35	81	1.22	73	10	20	59-121
4-Chlorophenylphenyl ether	1.67	1.43	86	1.30	78	10	20	25-158
4,6-Dinitro-2-methyl phenol	1.67	1.45	87	1.39	83	5	20	D-181
4-Nitroaniline	1.67	1.22	73	1.09	65	12	20	30-150

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Semivolatile Soil
LCS/LCSD Concentration Summary

Lab Name:Northeast labs

Analyst: JJC

File LCS:M7322 File LCSD:M7323

Date Extracted:08/15/06 Date Analyzed:08/17/06

Compound	Spike Added (mg/Kg)	LCS Concentration (mg/Kg)	LCS % Rec.	LCSD Concentratio (mg/Kg)	LCSD % Rec.	% RPD	QC Limits RPD	QC Limits Rec.
N-Nitroso-di-phenyl amine	1.67	1.15	69	1.11	66	4	20	30-150
Azobenzene	1.67	1.38	83	1.26	75	10	20	30-150
4-Bromophenylphenyl ether	1.67	1.52	91	1.46	87	4	20	53-127
Hexachlorobenzene	1.67	1.43	86	1.40	84	2	20	D-152
Pentachlorophenol	1.67	1.40	84	1.36	81	4	20	14-176
Phenanthrene	1.67	1.40	84	1.29	77	9	20	54-120
Anthracene	1.67	1.49	89	1.34	80	11	20	27-133
Carbazole	1.67	1.35	81	1.27	76	6	20	30-150
Di-n-butylphthalate	1.67	1.34	80	1.26	75	6	20	1-118
Fluoranthene	1.67	1.29	77	1.19	71	8	20	26-137
Benzidine	1.67	0.45	27*	0.47	28*	4	20	30-150
Pyrene	1.67	1.37	82	1.30	78	5	20	52-115
Benzylbutylphthalate	1.67	1.14	68	1.09	65	5	20	D-152
Benzo(a)anthracene	1.67	1.31	78	1.19	71	9	20	33-143
3,3-Dichlorobenzidine	1.67	1.01	60	0.93	56	7	20	D-262
Chrysene	1.67	1.35	81	1.23	74	9	20	17-168
Bis(2-Ethylhexyl)phthalate)	1.67	1.18	71	1.13	68	4	20	36-166
Di-n-octylphthalate	1.67	1.09	65	1.01	60	8	20	4-146
Benzo (b) fluoranthene	1.67	1.15	69	0.97	58	17	20	24-159
Benzo (k) fluoranthene	1.67	1.21	72	1.16	69	4	20	11-162
Benzo (a) pyrene	1.67	1.19	71	1.05	63	12	20	17-163
Indeno(1,2,3-cd)pyrene	1.67	1.17	70	1.07	64	9	20	D-171
Dibenz (ah) anthracene	1.67	1.07	64	0.96	57	12	20	D-227
Benzo (ghi) perylene	1.67	1.06	63	0.95	57	10	20	D-219

RPD 0 out of 70 outside limits

Spike Recovery 2 out of 140 outside limits

NC = not calculated
30-150% are nominal limits; internal statistical limits will be calculated when enough data points have been collected.

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Semivolatile Water
LCS/LCSD Concentration Summary

Lab Name:Northeast labs

Analyst: JJC

File LCS:M7493 File LCSD:M7494

Date Extracted:09/11/06 Date Analyzed:09/12/06

Compound	Spike Added (ug/L)	LCS Concentration (ug/L)	LCS % Rec.	LCSD Concentratio (ug/L)	LCSD % Rec.	% RPD	QC Limits RPD	QC Limits Rec.
N-Nitrosodimethylamine	50	20	40	24	48	18	20	25-110
pyridine	50	16	32	16	32	0	20	30-150
Phenol	50	17	34	17	34	0	20	D-115
Aniline	50	31	62	32	64	3	20	30-150
Bis(2-chloroethyl)ether	50	32	64	35	70	9	20	35-110
2-Chlorophenol	50	35	70	35	70	0	20	35-105
1,3-Dichlorobenzene	50	26	52	28	56	7	20	30-100
1,4-Dichlorobenzene	50	25	50	27	54	8	20	30-100
1,2-Dichlorobenzene	50	28	56	29	58	4	20	35-100
2-Methylphenol (o-cresol)	50	39	78	39	78	0	20	40-110
3,4-Methylphenol (m+p cresol)	50	37	74	41	82	10	20	30-110
Bis (2-Chloroisopropyl)ether	50	42	84	44	88	5	20	25-130
Benzyl Alcohol	50	39	78	40	80	3	20	30-110
Hexachloroethane	50	25	50	29	58	15	20	30-95
Nitrobenzene	50	34	68	36	72	6	20	45-110
Isophorone	50	42	84	40	80	5	20	50-110
2-Nitrophenol	50	40	80	39	78	3	20	40-115
2,4-Dimethyl phenol	50	39	78	37	74	5	20	30-110
Benzoic Acid	50	11	22	12	24	9	20	D-125
Bis(2-chloroethoxy)methane	50	39	78	38	76	3	20	45-105
N-nitroso-di-n-propylamine	50	46	92	48	96	4	20	35-130
2,4-Dichlorophenol	50	40	80	39	78	3	20	50-105
1,2,4-Trichlorobenzene	50	30	60	30	60	0	20	35-105
Naphthalene	50	30	60	31	62	3	20	40-100
4-Chloroaniline	50	42	84	43	86	2	20	15-110
Hexachlorobutadiene	50	31	62	32	64	3	20	25-105
4-Chloro-3-methyl phenol	50	45	90	43	86	5	20	45-110
2-Methylnaphthalene	50	37	74	36	72	3	20	45-105
2,4,6-Trichlorophenol	50	39	78	42	84	7	20	50-115
2,4,5-Trichlorophenol	50	45	90	47	94	4	20	50-110
2-Chloronaphthalene	50	35	70	37	74	6	20	50-105
2-Nitroaniline	50	53	106	54	108	2	20	50-115
Dimethylphthalate	50	35	70	34	68	3	20	25-125
2,6-Dinitrotoluene	50	46	92	49	98	6	20	50-115
Acenaphthylene	50	37	74	39	78	5	20	50-105
3-Nitroaniline	50	48	96	49	98	2	20	20-125
Hexachlorocyclopentadiene	50	23	46	25	50	8	20	30-150
Acenaphthene	50	37	74	37	74	0	20	45-110
2,4-Dinitrophenol	50	29	58	34	68	16	20	15-140
4-Nitrophenol	50	23	46	26	52	12	20	D-125
Dibenzofuran	50	42	84	42	84	0	20	55-105
2,4-Dinitrotoluene	50	47	94	48	96	2	20	50-120
Diethylphthalate	50	43	86	44	88	2	20	40-120
Fluorene	50	41	82	43	86	5	20	50-110
4-Chlorophenylphenyl ether	50	42	84	45	90	7	20	50-110
4,6-Dinitro-2-methyl phenol	50	42	84	46	92	9	20	40-130
4-Nitroaniline	50	54	108	54	108	0	20	35-120
N-Nitroso-di-phenyl amine	50	36	72	36	72	0	20	50-110
Azobenzene	50	49	98	50	100	2	20	55-115
4-Bromophenylphenyl ether	50	48	96	47	94	2	20	50-115
Hexachlorobenzene	50	46	92	45	90	2	20	50-110

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Semivolatile Water
LCS/LCSD Concentration Summary

Lab Name:Northeast labs

Analyst: JJC

File LCS:M7493 File LCSD:M7494

Date Extracted:09/11/08 Date Analyzed:09/12/08

Compound	Spike Added (ug/L)	LCS Concentration (ug/L)	LCS % Rec.	LCSD Concentratio (ug/L)	LCSD % Rec.	% RPD	QC Limits RPD	QC Limits Rec.
Pentachlorophenol	50	43	86	44	88	2	20	40-115
Phenanthrene	50	43	86	44	88	2	20	50-115
Anthracene	50	46	92	44	88	4	20	55-110
Carbazole	50	48	96	49	98	2	20	50-115
Di-n-butylphthalate	50	48	96	49	98	2	20	55-115
Fluoranthene	50	47	94	47	94	0	20	55-115
Benzidine	50	32	64	31	62	3	20	30-150
Pyrene	50	41	82	40	80	2	20	50-130
Benzylbutylphthalate	50	46	92	45	90	2	20	45-115
Benzo(a)anthracene	50	44	88	44	88	0	20	55-110
3,3-Dichlorobenzidine	50	50	100	50	100	0	20	20-110
Chrysene	50	42	84	43	86	2	20	55-110
Bis(2-Ethylhexyl)phthalate	50	50	100	51	102	2	20	40-125
Di-n-octylphthalate	50	53	106	53	106	0	20	35-135
Benzo (b) fluoranthene	50	59	118	56	112	5	20	45-120
Benzo (k) fluoranthene	50	54	108	55	110	2	20	45-125
Benzo (a) pyrene	50	54	108	52	104	4	20	55-110
Indeno(1,2,3-cd)pyrene	50	43	86	42	84	2	20	45-125
Dibenz (ah) anthracene	50	55	110	53	106	4	20	40-125
Benzo (ghi) perylene	50	56	112	46	92	20	20	40-125

RPD: 0 out of 70 outside limits

Spike Recovery: 0 out of 140 outside limits

30-150% are nominal limits; internal statistical limits will be calculated when enough data points have been collected.
NC = Not Calculated

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Semivolatile Soil
LCS/LCSD Concentration Summary

Lab Name:Northeast labs

Analyst: JJC

File LCS:M7550 File LCSD:M7551

Date Extracted:09/21/06 Date Analyzed:09/25/06

Instrument ID: 5890M

Compound	Spike Added (mg/Kg)	LCS Concentration (mg/Kg)	LCS % Rec.	LCSD Concentratio (mg/Kg)	LCSD % Rec.	% RPD	QC Limits RPD	QC Limits Rec.
N-Nitrosodimethylamine	1.67	1.03	62	1.08	65	5	20	20-115
pyridine	1.67	1.11	66	1.06	63	5	20	30-150
Phenol	1.67	1.06	63	1.09	65	3	20	40-100
Aniline	1.67	0.98	59	1.01	60	2	20	30-150
Bis(2-chloroethyl)ether	1.67	1.19	71	1.21	72	1	20	40-105
2-Chlorophenol	1.67	1.15	69	1.14	68	1	20	45-105
1,3-Dichlorobenzene	1.67	1.16	69	1.20	72	4	20	40-100
1,4-Dichlorobenzene	1.67	1.13	68	1.15	69	1	20	35-105
1,2-Dichlorobenzene	1.67	1.19	71	1.19	71	0	20	45-95
2-Methylphenol (o-cresol)	1.67	1.28	77	1.25	75	3	20	40-105
2,4-Methylphenol (m+p cresol)	1.67	1.31	78	1.31	78	0	20	40-105
Bis-(2-Chloroisopropyl)ether	1.67	1.23	74	1.39	83	11	20	20-115
Benzyl Alcohol	1.67	1.22	73	1.25	75	3	20	20-125
Hexachloroethane	1.67	1.15	69	1.23	74	7	20	35-110
Nitrobenzene	1.67	1.14	68	1.19	71	4	20	40-115
Isophorone	1.67	1.17	70	1.24	74	6	20	45-110
2-Nitrophenol	1.67	1.12	67	1.20	72	7	20	40-110
2,4-Dimethyl phenol	1.67	1.15	69	1.15	69	0	20	30-105
Benzoic Acid	1.67	0.74	44	0.95	57	26*	20	D-110
Bis(2-chloroethoxy)methane	1.67	1.11	66	1.22	73	10	20	45-110
N-nitroso-di-n-propylamine	1.67	1.40	84	1.43	86	2	20	40-115
2,4-Dichlorophenol	1.67	1.21	72	1.26	75	4	20	45-110
1,2,4-Trichlorobenzene	1.67	1.14	68	1.26	75	10	20	45-110
Naphthalene	1.67	1.10	66	1.15	69	4	20	40-105
4-Chloroaniline	1.67	1.15	69	1.18	71	3	20	20-95
Hexachlorobutadiene	1.67	1.35	81	1.44	86	6	20	40-115
4-Chloro-3-methyl phenol	1.67	1.24	74	1.22	73	1	20	45-115
2-Methylnaphthalene	1.67	1.15	69	1.22	73	6	20	45-105
2,4,6-Trichlorophenol	1.67	1.30	78	1.23	74	5	20	45-110
2,4,5-Trichlorophenol	1.67	1.30	78	1.31	78	0	20	50-110
2-Chloronaphthalene	1.67	1.12	67	1.11	66	2	20	45-105
2-Nitroaniline	1.67	1.24	74	1.21	72	3	20	45-120
Dimethylphthalate	1.67	1.22	73	1.21	72	1	20	50-110
2,6-Dinitrotoluene	1.67	1.19	71	1.19	71	0	20	50-110
Acenaphthylene	1.67	1.16	69	1.15	69	0	20	45-105
3-Nitroaniline	1.67	1.15	69	1.12	67	3	20	25-110
Hexachlorocyclopentadiene	1.67	0.67	40	0.87	52	26*	20	30-150
Acenaphthene	1.67	1.10	66	1.11	66	0	20	45-110
2,4-Dinitrophenol	1.67	0.51	31	0.69	41	28*	20	15-130
4-Nitrophenol	1.67	1.23	74	1.29	77	4	20	15-140
Dibenzofuran	1.67	1.13	68	1.16	69	1	20	50-105
2,4-Dinitrotoluene	1.67	1.23	74	1.23	74	0	20	50-115
Diethylphthalate	1.67	1.22	73	1.24	74	1	20	50-115
Fluorene	1.67	1.27	76	1.27	76	0	20	50-110
4-Chlorophenylphenyl ether	1.67	1.25	75	1.26	75	0	20	45-110
4,6-Dinitro-2-methyl phenol	1.67	0.57	34	0.78	47	32*	20	30-135
4-Nitroaniline	1.67	1.27	76	1.29	77	1	20	35-115

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Semivolatile Soil
LCS/LCSD Concentration Summary

Lab Name:Northeast labs

Analyst: JJC

File LCS:M7550 File LCSD:M7551

Date Extracted:09/21/06 Date Analyzed:09/25/06
Instrument ID: 5890M

Compound	Spike Added (mg/Kg)	LCS Concentration (mg/Kg)	LCS % Rec.	LCSD Concentratio (mg/Kg)	LCSD % Rec.	% RPD	QC Limits RPD	QC Limits Rec.
N-Nitroso-di-phenyl amine	1.67	0.93	56	0.97	58	4	20	50-115
Azobenzene	1.67	1.19	71	1.17	70	1	20	30-150
4-Bromophenylphenyl ether	1.67	1.26	75	1.34	80	6	20	45-115
Hexachlorobenzene	1.67	1.31	78	1.36	81	4	20	45-120
Pentachlorophenol	1.67	1.28	77	1.42	85	10	20	25-120
Phenanthrene	1.67	1.20	72	1.24	74	3	20	50-110
Anthracene	1.67	1.28	77	1.30	78	1	20	55-105
Carbazole	1.67	1.24	74	1.27	76	3	20	45-115
Di-n-butylphthalate	1.67	1.24	74	1.32	79	7	20	55-110
Fluoranthene	1.67	1.36	81	1.43	86	6	20	55-115
Benzidine	1.67	0.68	41	0.78	47	14	20	30-150
Pyrene	1.67	1.21	72	1.17	70	3	20	45-125
Benzylbutylphthalate	1.67	1.22	73	1.19	71	3	20	50-125
Benzo(a)anthracene	1.67	1.32	79	1.29	77	3	20	50-110
3,3-Dichlorobenzidine	1.67	1.40	84	1.31	78	7	20	10-130
Chrysene	1.67	1.27	76	1.19	71	7	20	55-110
Bis(2-Ethylhexyl)phthalate)	1.67	1.34	80	1.30	78	3	20	45-125
Di-n-octylphthalate	1.67	1.49	89	1.45	87	2	20	40-130
Benzo (b) fluoranthene	1.67	1.34	80	1.33	80	0	20	45-115
Benzo (k) fluoranthene	1.67	1.24	74	1.36	81	9	20	45-125
Benzo (a) pyrene	1.67	1.34	80	1.39	83	4	20	50-110
Indeno(1,2,3-cd)pyrene	1.67	1.32	79	1.27	76	4	20	40-120
Dibenz (ah) anthracene	1.67	1.29	77	1.37	82	6	20	40-125
Benzo (ghi) perylene	1.67	1.31	78	1.36	81	4	20	40-125

RPD 4 out of 70 outside limits

Spike Recovery 0 out of 140 outside limits

NC = not calculated
30-150% are nominal limits; internal statistical limits will be calculated when enough data points have been collected.

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Attachment 3

TABLE D-6. LCS CONTROL LIMITS FOR SEMIVOLATILE ORGANIC COMPOUNDS SW-846 METHOD 8270 WATER MATRIX[14]

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Polynuclear Aromatics						
2-Methylnaphthalene	75.0	9.5	45	105	35	115
Acenaphthene	77.6	10.1	45	110	35	120
Acenaphthylene	78.5	9.4	50	105	40	115
Anthracene	83.0	9.7	55	110	45	120
Benz[a]anthracene	82.7	8.9	55	110	45	120
Benzo[a]pyrene	81.3	9.5	55	110	45	120
Benzo[b]fluoranthene	81.8	12.1	45	120	35	130
Benzo[k]fluoranthene	84.6	13.2	45	125	30	135

[14] A number of sporadic marginal exceedances of the control limits are allowed depending on the number of analytes spiked in the LCS. Refer to section D.2 and Table D-1 for guidance on the appropriate application of control and ME limits. LCS control limits are not available for Benzidine, 2,6-Dichlorophenol, and N-nitrosopyrrolidine, although those compounds do appear on the target analyte list for method 8270 (Table C-2 in Appendix DoD-C). Sufficient data to perform statistically significant analyses were not received for those analytes during the LCS study. Additional limits for poor performing compounds can be found in section D.5.

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TABLE D-6. LCS CONTROL LIMITS FOR SEMIVOLATILE ORGANIC COMPOUNDS SW-846 METHOD 8270 WATER MATRIX[14]

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Benzo[g,h,i]perylene	80.5	14.1	40	125	25	135
Chrysene	82.1	8.9	55	110	45	120
Dibenz[a,h]anthracene	84.7	14.1	40	125	30	140
Fluoranthene	85.2	10.4	55	115	45	125
Fluorene	80.6	10.3	50	110	40	120
Indeno[1,2,3-cd]pyrene	84.3	13.5	45	125	30	140
Naphthalene	70.8	10.5	40	100	30	115
Phenanthrene	84.0	11.0	50	115	40	130
Pyrene	88.6	13.2	50	130	35	140
Phenolic/Acidic						
2,4,5-Trichlorophenol	79.7	10.3	50	110	40	120
2,4,6-Trichlorophenol	80.7	10.7	50	115	40	125
2,4-Dichlorophenol	76.3	9.6	50	105	40	115
2,4-Dimethylphenol	68.8	13.5	30	110	15	125
2,4-Dinitrophenol	75.8	20.6	15	140	10	160
2-Chlorophenol	71.3	11.4	35	105	25	115
2-Methylphenol	73.3	11.7	40	110	25	120
2-Nitrophenol	75.8	12.4	40	115	25	125
3-Methylphenol/4-Methylphenol	71.3	13.0	30	110	20	125
4,6-Dinitro-2-methylphenol	84.9	15.0	40	130	25	145
4-Chloro-3-methylphenol	78.6	10.7	45	110	35	120
Pentachlorophenol	77.6	13.3	40	115	25	130
Basic						
3,3'-Dichlorobenzidine	65.2	15.3	20	110	10	125
4-Chloroaniline	62.2	15.6	15	110	10	125
Phthalate Esters						
Bis(2-ethylhexyl) phthalate	84.2	14.0	40	125	30	140
Butyl benzyl phthalate	81.1	11.7	45	115	35	130
Di-n-butyl phthalate	84.8	10.3	55	115	45	125
Di-n-octyl phthalate	87.4	16.6	35	135	20	155
Diethyl phthalate	79.2	12.9	40	120	30	130
Dimethyl phthalate	75.9	16.9	25	125	10	145
Nitrosoamines						
N-Nitrosodi-n-propylamine	80.9	15.7	35	130	20	145
N-Nitrosodimethylamine	67.9	14.1	25	110	10	125
N-Nitrosodiphenylamine	79.6	10.6	50	110	35	120
Chlorinated Aliphatics						
Bis(2-chloroethoxy)methane	76.2	10.2	45	105	35	115
Bis(2-chloroethyl) ether	73.3	12.3	35	110	25	120
Bis(2-chloroisopropyl) ether	78.2	17.5	25	130	10	150
Hexachlorobutadiene	65.2	12.6	25	105	15	115
Hexachloroethane	60.9	11.1	30	95	15	105

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TABLE D-6. LCS CONTROL LIMITS FOR SEMIVOLATILE ORGANIC COMPOUNDS SW-846 METHOD 8270 WATER MATRIX[14]

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Halogenated Aromatics						
1,2,4-Trichlorobenzene	71.7	11.6	35	105	25	120
1,2-Dichlorobenzene	67.3	11.4	35	100	20	115
1,3-Dichlorobenzene	64.8	10.9	30	100	20	110
1,4-Dichlorobenzene	64.8	10.9	30	100	20	110
2-Chloronaphthalene	76.5	9.3	50	105	40	115
4-Bromophenyl phenyl ether	82.9	10.2	50	115	40	125
4-Chlorophenyl phenyl ether	80.6	10.3	50	110	40	120
Hexachlorobenzene	82.3	10.0	50	110	40	120
Nitroaromatics						
2,4-Dinitrotoluene	84.3	11.2	50	120	40	130
2,6-Dinitrotoluene	82.7	11.3	50	115	35	130
2-Nitroaniline	81.8	11.2	50	115	35	125
3-Nitroaniline	72.6	17.7	20	125	10	145
4-Nitroaniline	77.2	13.7	35	120	20	130
Nitrobenzene	76.8	10.8	45	110	35	120
Neutral Aromatics						
Carbazole	82.5	11.4	50	115	35	130
Dibenzofuran	80.3	8.8	55	105	45	115
Others						
1,2-Diphenylhydrazine	84.8	9.4	55	115	45	120
Benzyl alcohol	71.0	13.8	30	110	15	125
Isophorone	81.0	10.5	50	110	40	125

TABLE D-7. LCS CONTROL LIMITS FOR SEMIVOLATILE ORGANIC COMPOUNDS SW-846 METHOD 8270 SOLID MATRIX[15]

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Polynuclear Aromatics						
2-Methylnaphthalene	77.3	10.0	45	105	35	115
Acenaphthene	77.3	10.3	45	110	35	120
Acenaphthylene	75.7	10.4	45	105	35	115
Anthracene	79.9	9.0	55	105	45	115

[15] A number of sporadic marginal exceedances of the control limits are allowed, depending on the number of analytes spike in the LCS. Refer to section D.2 and Table D-1 for guidance on the appropriate application of control and ME limits. LCS control limits are not available for Benzidine, 2,6-Dichlorophenol, 1,2-Diphenylhydrazine, and N-nitrosopyrrolidine, although those compounds do appear on the target analyte list for method 8270 (Table C-2 in Appendix DoD-C). Sufficient data to perform statistically significant analyses were not received for those analytes during the LCS study. Additional limits for poor performing compounds can be found in section D.5.

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TABLE D-7. LCS CONTROL LIMITS FOR SEMIVOLATILE ORGANIC COMPOUNDS SW-846 METHOD 8270 SOLID MATRIX[15]

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Benz[a]anthracene	81.6	9.8	50	110	40	120
Benzo[a]pyrene	80.7	10.3	50	110	40	120
Benzo[b]fluoranthene	79.7	11.4	45	115	35	125
Benzo[k]fluoranthene	83.8	12.9	45	125	30	135
Benzo[g,h,i]perylene	81.8	14.7	40	125	25	140
Chrysene	82.6	9.9	55	110	45	120
Dibenz[a,h]anthracene	82.9	13.9	40	125	25	140
Fluoranthene	83.9	10.1	55	115	45	125
Fluorene	78.3	9.8	50	110	40	115
Indeno[1,2,3-cd]pyrene	79.7	13.8	40	120	25	135
Napnthalene	73.4	11.1	40	105	30	120
Phenanthrene	80.1	10.0	50	110	40	120
Pyrene	84.4	12.8	45	125	35	135
Phenolic/Acidic						
2,4,5-Trichlorophenol	80.1	10.4	50	110	40	120
2,4,6-Trichlorophenol	76.3	11.0	45	110	30	120
2,4-Dichlorophenol	77.2	10.9	45	110	35	120
2,4-Dimethylphenol	67.3	11.9	30	105	20	115
2,4-Dinitrophenol	72.6	20.0	15	130	10	150
2-Chlorophenol	74.7	10.3	45	105	35	115
2-Methylphenol	71.7	10.6	40	105	30	115
2-Nitrophenol	76.2	11.5	40	110	30	120
3-Methylphenol/4-Methylphenol	73.9	10.9	40	105	30	120
4,6-Dinitro-2-methylphenol	83.1	18.0	30	135	10	155
4-Chloro-3-methylphenol	79.5	11.1	45	115	35	125
4-Nitrophenol	77.0	20.2	15	140	10	160
Pentachlorophenol	71.9	15.6	25	120	10	135
Phenol	69.7	10.2	40	100	30	110
Phthalate Esters						
Bis(2-etnylhexyl) phthalate	87.4	13.3	45	125	35	140
Butyl benzyl phthalate	86.4	12.3	50	125	35	135
Di-n-butyl phthalate	83.2	9.1	55	110	45	120
Di-n-octyl phthalate	86.4	15.2	40	130	25	145
Diethyl phthalate	82.2	10.6	50	115	40	125
Dimethyl phtnalate	79.6	10.2	50	110	40	120
Nitrosoamines						
N-Nitrosodi-n-propylamine	76.8	12.3	40	115	30	125
N-Nitrosodimetnylamine	66.1	15.9	20	115	10	130
N-Nitrosodiphenylamine	82.4	11.1	50	115	40	125
Chlorinated Aliphatics						
Bis(2-chloretnoxy)methane	75.5	10.9	45	110	30	120
Bis(2-chloroethyl) ether	71.1	11.2	40	105	25	115
Bis(2-chloroisopropyl) ether	68.4	15.7	20	115	10	130

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TABLE D-7. LCS CONTROL LIMITS FOR SEMIVOLATILE ORGANIC COMPOUNDS SW-846 METHOD 8270 SOLID MATRIX[15]

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Hexachlorobutadiene	78.2	12.9	40	115	25	130
Hexachloroethane	71.9	12.6	35	110	20	120
Halogenated Aromatics						
1,2,4-Trichlorobenzene	77.4	11.2	45	110	30	120
1,2-Dichlorobenzene	70.9	8.7	45	95	35	105
1,3-Dichlorobenzene	69.7	10.3	40	100	30	110
1,4-Dichlorobenzene	69.0	11.4	35	105	25	115
2-Chloronaphthalene	75.2	9.9	45	105	35	115
4-Bromophenyl phenyl ether	81.7	11.8	45	115	35	130
4-Chlorophenyl phenyl ether	79.6	10.7	45	110	35	120
Hexachlorobenzene	82.5	11.7	45	120	35	130
Nitroaromatics						
2,4-Dinitrotoluene	82.0	11.4	50	115	35	130
2,6-Dinitrotoluene	80.2	10.7	50	110	35	125
2-Nitroaniline	81.0	12.2	45	120	30	130
3-Nitroaniline	68.8	13.8	25	110	15	125
4-Nitroaniline	73.6	13.1	35	115	20	125
Nitrobenzene	77.2	11.9	40	115	30	125
Neutral Aromatics						
Carbazole	80.4	12.3	45	115	30	130
Dibenzofuran	77.1	8.8	50	105	40	110
Others						
Benzyl alcohol	70.9	17.4	20	125	10	140
Isophorone	77.0	11.4	45	110	30	125

[6] LCS control limits were generated using non-parametric statistics (see section D.1 for further explanation). LCS control limits are not available for MCPP, although the compound does appear on the target analyte list for method 8151 (Table C-5 in Appendix DoD-C). Sufficient data to perform statistically significant analyses were not received for the analyte during the LCS study.

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DOCUMENT HISTORY AND APPROVAL PAGE

TITLE:	**Separatory Funnel Liquid-Liquid Extraction Method 3510C- Acid/Base Neutral Semivolatile Organics**
DEPARTMENTS AFFECTED:	**Organics**

ISSUE DATE:	REVISION CODE:	SOP #:	REVISION CHANGES:
10/3/01	-	3.3.29	Create a Prep SOP for Semivolatile Organics
5/17/04	A	3.3.29	Update to include internal annual method review and Navy assessment
01/16/06	B	3.3.29	Annual Review & Update
09/19/06	C	3.3.29	Update to include Navy Assessment Findings
10/20/06	D	3.3.29	Update for Additional Assessment Findings
12/17/07	E	3.3.29	Annual Review & Update
01/23/08	F	3.3.29	Annual Review & Update

DOCUMENT APPROVAL		
Originator: James Galasyn, Ph.D	Signature/Date: J Galasyn 012308	Title Chemistry Lab Manager
Approved by: Kelsey Anthony	Kelsey Anthony 01/24/08	Chemistry Analyst
Reviewed by: Pamela Doughty	Pamela Doughty 1/23/08	Total Quality Manager

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NEL STANDARD OPERATING PROCEDURE			
TITLE: **Separatory Funnel Liquid-Liquid Extraction Method 3510C- Acid/Base Neutral Semivolatile Organics**			
SOP# 3.3.29	REV# F	EFFECTIVE 01/23/08	PAGE 1OF 15
WRITTEN BY: James Galasyn	DEPARTMENTS AFFECTED: ORGANICS		

1.0 Purpose:.

This standard operating procedure (SOP) is for the separatory liquid-liquid extraction of acid/base/neutral semivolatile organics (SW-846 Method 3510C).

2.0 Applicable Matrix or Matrices:

This procedure is designed to isolate acid/base/neutral semivolatile organic compounds from aqueous samples.

3. 0 Summary of Method:

3.1 Please refer to the determinative methods (NEL SOPs 3.3.15 and 3.3.39) for details regarding detection limits.

3.2 The laboratory's reporting limit shall lie within the calibration range, at or above the lower calibration limit. If the client requires a reporting limit below the lower limit of the calibration curve, then method modification is required.

4.0 Scope and Application:

4.1 This method describes a procedure for isolating organic compounds (water-insoluble and slightly water-soluble) from aqueous samples. The method also describes concentration techniques suitable for preparing the extract for analysis by Gas Chromatography/Mass Spectrometry (GC/MS).

4.2 This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method.

5.0 Summary of Method:

5.1 A measured volume of sample, usually 1 liter, is serially extracted (at a specified pH) with methylene chloride using a separatory funnel.

5.2 The extract is dried and concentrated (if necessary).

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6.0 Definitions:

6.1 ABN: Acid/Base/Neutral compound.

6.2 SVOA: Semivolatile Organic Compound.

6.3 Method Detection Limit (MDL): Minimum concentration at which an analyte can be measured and reported with 99% confidence that the analyte concentration is greater than zero. MDL is determined from analysis of a sample in a given matrix containing the analyte. The MDL is determined using the Student t Test following EPA Appendix B to Part 136, CFR 40 Ch. 1 (7-1-94).

6.4 Method Blank: An aliquot of an analyte free water that is spiked with surrogates and extracted daily with each batch of up to 20 samples. Method blanks should not contain analytes of interest above one-half the reporting limit.

6.5 Surrogate: A surrogate is a compound that is chemically similar to the analyte group but not expected to occur in an environmental sample. Surrogates should be added to all samples.

6.6 Laboratory Control Sample (LCS): The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the sample weight or volume. The LCS is spiked with the same analytes as the matrix spike sample. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. LCS should be extracted daily with each extraction batch of up to 20 samples. Results should be compared to those in the determinative method.

6.7 Laboratory Control Spike Duplicate (LCSD): An aliquot of clean matrix spiked with components of interest as in the LCS. The LCSD is used to evaluate both precision and accuracy. LCSD should be extracted daily with each batch of up to 20 samples. Results should be compared to the limits in the determinative method.

6.8 Matrix Spike (MS): A sample aliquot which a known amount of target analyte is added. Matrix spike analyses should be performed every 20 samples. If there is insufficient sample volume to perform the matrix spike, LCS/LCSD samples should be used to access precision and accuracy. Results should be compared to the limits in the determinative method.

6.9 Matrix Spike Duplicate (MSD): A sample aliquot spiked with components of interest as in the MS. The MSD is used to evaluate both precision and accuracy. If there is sufficient sample volume, the MSD should be extracted every 20 samples. Results should be compared to those in the determinative method.

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6.10 Duplicate: An aliquot of actual sample extracted at the same time as the native sample. The duplicate is used to assess the precision of analytical results in a given matrix and are expressed as relative percent difference (RPD). In the absence of MS/MSD analysis, duplicates should be performed at the rate of 1 per 20 samples.

7.0 Interferences:

7.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be necessary.

7.1.1 Please refer to NEL SOP 3.7.1 for specifics regarding washing of glassware and preservation of sample bottles.

7.2 Interferences coextracted from the samples will vary considerably from source to source. If the analysis of an extracted sample is prevented due to interferences, further cleanup of the sample extract may be necessary.

7.3 Phthalate esters contaminate many types of products commonly found in the laboratory. Plastics, in particular, must be avoided because phthalates are commonly used as plasticizers and are easily extracted from plastic materials. Serious phthalate contamination may result at any time if consistent quality control is not practiced.

7.4 The decomposition of some analytes has been demonstrated under basic extraction conditions. Phthalate esters may exchange and phenols may react to form tannates. These reactions increase with increasing pH, and are decreased by the shorter reaction times available in Method 3510. Method 3510 is preferred over method 3520 for the analysis of these classes of compounds.

8.0 Safety

8.1 Analysts should read and understand the laboratory chemical hygiene plan before handling any reagents specified in this standard operating procedure.

8.2 Analysts should wear the proper protective attire when handling the chemicals referenced in this standard operating procedure. Proper protective attire includes labcoats, chemically resistant gloves and aprons, safety glasses and goggles. Open-toed shoes should not be worn in the laboratory.

8.3 Sample extraction should be performed in a hood with the hood sash pulled down to ensure that analyst exposure to solvents is minimized. Analysts should read the applicable MSDS sheets and be aware of hazards, odor thresholds and cleanup procedures required in case of an accidental spill.

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8.4 Methylene chloride creates excessive pressure very rapidly. Therefore, initial venting should be done immediately after the separatory funnel has been sealed and shaken once. The separatory funnel should be vented into a hood to avoid exposure of the analyst to solvent vapors.

8.5 Syringes are used to spike surrogates, matrix spiking solutions and internal standards into samples or sample extracts. Care should be taken to ensure that the analyst is not mistakenly injected with a syringe.

9.0 Equipment and Supplies

9.1 Separatory funnel- 2-liter, with polytetrafluoroethylene (PFTE) stopcock.

9.2 Drying column- 20 mm ID Pyrex chromatographic column or glass funnels with glass wool at the bottom. Prewash the glass wool pad with 50 ml of methylene chloride prior to packing the column/funnel with adsorbent.

9.3 Kuderna-Danish (K-D) apparatus.

9.3.1 Concentrator tube- 10 ml, graduated (Kontes K-5700250-1025 or equivalent). A ground glass stopper is used to prevent evaporation of extracts.

9.3.2 Evaporation flask- 500 ml (Kontes K-570001-500 or equivalent). Attach to concentrator tube with springs, clamps, or equivalent.

9.3.3 Snyder column- Three-ball macro (Kontes K-503000-0121 or equivalent)

9.3.4 Snyder column- Two-ball micro (Kontes K-569001-0219 or equivalent)

9.3.5 Springs- ½ inch (Kontes K-662750 or equivalent)

Alternately, use of N-EVAP system may be used to concentrate the extracts.

9.4 Boiling chips- Solvent extracted, approximately 10/40 mesh (silicon carbide or equivalent)

9.5 Water bath- Heated, with concentric ring cover, capable of temperature control (+/_ 5°C). The bath should be used in a hood.

9.6 Vials- 2 ml, glass with PTFE-lined screw caps or crimp tops.

9.7 pH indicator paper- pH range including the desired extraction pH. (Fisher alkacid test, cat. No. A-979, range pH 2-10)

9.8 Erlenmeyer flask- 250 ml.

9.9 Syringe- 1ml

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9.10 Graduated cylinder- 1 liter.

9.11 Turbovap concentrator device including evaporation tubes.

10.0 Reagents and Standards

10.1 Reagent grade chemicals should be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. Reagents should be stored in glass to prevent the leaching of contaminants from plastic containers. Extraction solvent is methylene Chloride- Pesticide grade. The lot of methylene chloride should be checked for contaminants prior to use by extracting a method blank.

10.2 Organic-free reagent water

10.3 Sodium hydroxide solution (10N), NaOH. Dissolve 40 g NaOH in organic-free reagent water and dilute to 100 ml. Other concentrations of hydroxide solutions may be used to adjust sample pH, provided that the volume added does not appreciably change (e.g. <1%) the total sample volume. 75% KOH solution is another alternative. Dissolve 75g KOH in organic-free reagent water and dilute to 100 ml.

10.4 Sodium sulfate (granular, anhydrous), Na_2SO_4. Purify by heating to 550 °C for 4 hours in a shallow tray, or by precleaning the sodium sulfate with methylene chloride. If the sodium sulfate is precleaned with methylene chloride, a method blank must be analyzed, demonstrating that there is no interference from the sodium sulfate.

10.5 Sulfuric acid solution (1:1 v/v), H_2SO_4. Slowly add 50 ml of H_2SO_4 to 50 ml of organic-free reagent water. Other concentration of acid solutions may be used to adjust sample pH, provided that the volume added does not appreciably change (e.g.<1%) the total sample volume.

10.6 Stock standards for spiking solutions- Stock solutions may be prepared form the pure standard materials or purchased as certified solutions. The stock solutions for the calibration standards are acceptable (dilutions must be made in a water miscible solvent) except for the control check sample stock concentrate which must be prepared independently to serve as a check on the accuracy of the calibration solution.

10.6.1 Prepare the stock standard solutions by accurately weighing about 0.0100 g of pure compound. Dissolve the compound in a water miscible solvent (i.e. methanol, acetone, 2-propanol, etc) and dilute to volume in a 10-ml volumetric flask. If compound purity is

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96 percent or greater, the weight can be used without correction to calculate the concentration of the stock standard solution. Commercially prepared stock standard solutions can be used at the concentration if they are certified by the manufacturer or by an independent source.

10.6.2 Stock standard solutions should be stored in PTFE sealed containers at 4 °C or below. Refer to the determinative method for holding times of the stock solutions.

10.7 Surrogate standards- A surrogate (i.e., a compound that is chemically similar to the analyte group but is not expected to occur in an environmental sample) should be added to each sample, blank, laboratory control spike (LCS), and matrix spike sample just prior to extraction or processing. The recovery of the surrogate standard is used to monitor for unusual matrix effects, gross sample processing errors, etc. Surrogate recovery is evaluated for acceptance by determining whether the measured concentration falls within the acceptance limits. Surrogates used in this procedure are listed in Table 1.

10.7.1 Prepare a surrogate spiking concentrate by mixing stock standards prepared above and diluting with a water miscible solvent. Commercially prepared spiking solutions are acceptable. The concentration should be such that a 1-ml aliquot into 1000 ml of a sample provides a concentration of 10 times the quantitation limit or near the mid-point of the calibration curve. For matrices other than water, 1 ml of surrogate standard is still the normal spiking volume. However, if gel permeation chromatography will be used for sample cleanup, 2 ml should be added to the sample.

10.8 Matrix spike standards- The recommended matrix spike standard is listed in Table 2. This standard encompasses all of the reported analytes. Prepare a matrix spike concentrate by mixing stock standards prepared above and diluting with a water miscible solvent. Commercially-prepared spiking solutions are acceptable. The matrix spike standards should be independent of the calibration standard.

10.9 Laboratory control spike standard- Use the matrix spike standard prepared in Sec. 10.8 as the spike standard for the laboratory control sample (LCS). The LCS should be spiked at the same concentration as the matrix spike.

11.0 Sample collection, preservation, shipment and storage

Containers used to collect samples for the determination of semivolatile organics should be soap and water washed followed by methanol (or isopropanol) rinsing. Alternatively, containers pre-cleaned by the manufacturer should be rinsed with methylene chloride to minimize interferences. The sample containers should be of Amber glass or Teflon and have screw-caps with Teflon liners. In situations where Teflon is not available, solvent-rinsed aluminum foil may be used as a

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liner. However, acidic or basic samples may react with the aluminum foil, causing eventual contamination of the sample. Plastic containers or lids may NOT be used for the storage of samples due to the possibility of sample contamination from the phthalate esters and other hydrocarbons within the plastic. Sample containers should be filled with care so as to prevent any portion of the collected sample coming in contact with the sampler's gloves, thus causing contamination. Samples should not be collected or stored in the presence of exhaust fumes. If the sample cones in contact with the sampler (e.g. if an automatic sampler is used), run organic-free reagent water through the sampler and use as a field blank. A minimum of two 1-liter amber glass containers should be collected for each sample location. Additional sample should be collected if matrix spike/matrix spike duplicates are to be performed. Samples should be cooled to 4 °C +/- 2 °C from the time of sampling to the time of extraction. Samples should be extracted within 7 days and extracts analyzed within 40 days following extraction.

12.0 Quality Control

12.1 Any reagent blanks, matrix spikes, or replicate samples should be subjected to exactly the sample analytical procedures as those used on actual samples.

12.2 Each laboratory using this method should maintain a formal quality assurance program. Each extraction batch of 20 or less samples should contain: a method blank, either a matrix spike/matrix spike duplicate or a matrix spike and duplicate samples and a laboratory control sample.

12.3 Initial demonstration of Proficiency- Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean reference matrix. This will include a combination of the sample extraction method and the determinative method. The laboratory should also repeat the following operations whenever new staff are trained or significant changes in instrumentation are made.

12.3.1 The reference samples are prepared from a spiking solution containing each analyte of interest. The reference sample concentrate (spiking solution) may be prepared from pure standard materials, or purchased as certified solutions. If prepared by the laboratory, the reference sample concentrate should be made using stock standards prepared independently from those used for calibration.

12.3.2 The procedure for preparation of the reference sample concentrate is dependent of the method being evaluated. The concentration of target analytes in the reference sample may be adjusted to more accurately reflect the concentrations that will be analyzed by the laboratory.

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12.3.3 To evaluate the performance of the total analytical process, the reference samples must be handled in exactly the same manner as actual samples. Use a clean matrix for spiking purposes (e.g.. organic-free reagent water for the water matrix).

12.4 Sample Quality Control for Preparation and Analysis

12.4.1 Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair per analytical batch.

12.4.2 A Laboratory Control Sample (LCS) should be included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the sample weight or volume. The LCS is spiked with the same analytes at the same concentration as the matrix spike sample. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

12.4.3 Analyze these QC samples (the LCS and the matrix spikes or the optional duplicates) following the procedure in the selected determinative method. Calculate and evaluate the QC data.

12.4.4 Blanks- Use of method blanks and other blanks are necessary to track contamination of samples during the sampling and analysis processes. Method blanks should not contain analytes of interest at levels greater than one-half the reporting limit.

12.4.5 Surrogates- A surrogate is a compound that is chemically similar to the analyte group but not expected to occur in an environmental sample. Surrogates should be added to all samples.

Refer to the determinative method for surrogate recovery limits.

13.0 Calibration and Standardization

Refer to the determinative method.

14.0 Procedure

14.1 Rinse all glassware and glass wool with approximately 60 ml methylene chloride. Discard. Using a 1-liter graduated cylinder, measure 1 liter (nominal) of sample. Alternately, if the entire contents of the sample bottle are to be extracted, mark the level of the sample on the outside of the bottle. If high analyte concentrations are anticipated, a smaller sample volume may be taken and diluted to 1-L with organic-free reagent water, or the samples may be collected in smaller sample bottles and the whole sample used.

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14.2 Spike 1.0 ml of the surrogate spiking solution into each sample and mix well.

14.2.1 For the sample in each batch selected for use as a matrix spike sample, add 500 ul of the acid/ base/neutral matrix spiking standard.

14.2.2 If method 3640, Gel-Permeation Cleanup, is to be employed, add twice the volume of the surrogate spiking solution and the matrix spiking standard, since half of the extract is not recovered from the GPC apparatus. (Alternately, use 1.0 ml of the spiking solutions and concentrate the final extract to half the normal volume, e.g. 0.5 ml instead of 1.0 ml).

14.3 Check the pH of the sample with wide-range pH paper (Fisher alkacid test, cat. A-979, range pH 2-10) and adjust the pH, if necessary, to pH > 11 using 75% KOH. The pH is checked by stirring the sample with a clean glass stirring rod. After stirring, drops from the rod are placed directly in the pH paper. The glass rod must be rinsed with deionized water in between uses. Lesser strengths of acid or base solution may be employed, provided that they do not result in a significant change (<1%) in the volume of sample extracted. Extraction pH sequence may be reversed to better separate acid and neutral waste components. Excessive pH adjustments may result in the loss of some analytes.

14.4 Quantitatively transfer the sample from the graduated cylinder or sample bottle to the separatory funnel. Use 60 ml of methylene chloride to rinse the cylinder (or bottle) and transfer this rinse solvent to the separatory funnel. If the sample was transferred directly from the sample bottle, refill the bottle to the mark made in Sec. 9.1 with water and then measure the volume of sample that was in the bottle.

14.5 Seal and shake the separatory funnel vigorously for 2 minutes with periodic venting to release excess pressure.

NOTE: Methylene chloride creates excessive pressure very rapidly. Therefore, initial venting should be done immediately after the separatory funnel has been sealed and shaken once. The separatory funnel should be vented in a hood to avoid exposure of the analyst to solvent vapors.

14.6 Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends of the sample and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. Collect the solvent extract in an Erlenmeyer flask. If the emulsion cannot be broken (recovery of <80% of the methylene chloride corrected for the water solubility of methylene chloride), transfer the sample, solvent and emulsion into the extraction chamber of a continuous extractor and

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proceed as described in Method 3520- Continuous Liquid-Liquid Extraction.

14.7 Repeat the extraction two more times using fresh portions of methylene chloride. Combine the three solvent extracts.

14.8 Adjust the pH to >2 using sulfuric acid. Serially extract three times with 60 ml of methylene chloride, as outlined in Secs, 9.2 through 9.5. Collect and combine the extracts and label the combined extract appropriately. The acid and base/neutral extracts may be combined prior to concentration. However, in some situations, separate concentration and analysis of the acid/neutral and base extracts may be preferable.

14.9 Perform the concentration (if necessary) using the Kuderna-Danish Technique (Sec 9.10.1 through 9.10.4). Alternatively, the n-EVAP system may be used to concentrate sample extracts.

14.10 K-D technique

14.10.1 Assemble a Kuderna-Danish (K-D) concentrator by attaching a 10-ml concentrator tube to a 500-ml evaporation flask. Rinse the glassware one time with methylene chloride.

14.10.2 Dry the extract by passing it through a drying column containing about 10 cm of anhydrous sodium sulfate. Collect the dried extract in a K-D concentrator. Rinse the Erlenmeyer flask, which contained the solvent extract with 20 – 30 ml of methylene chloride and add it to the column to complete the quantitative transfer.

14.10.3 Add one or two clean boiling chips to the flask and attach a three ball Snyder column. Prewet the Snyder column by adding about 1-ml of methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 10 – 20 minutes. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of the liquid reaches 1 ml, remove the K-D apparatus form the water bath and allow it to drain and cool for at least 10 minutes.

14.10.4 Remove the Snyder column and rinse the flask and its lower joints into the concentrator tube with 1 – 2 ml of methylene chloride. If sulfur crystals are a problem, proceed to method 3660 for cleanup. The extract may be further concentrated by using the technique outlined in Sec. 9.11 or adjusted to 10.0 ml with the solvent last used.

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14.11 Concentrate the extract to 1.0 ml using either the micro-Snyder column or nitrogen blowdown technique.

14.11.1 Micro-Snyder column technique

Add another clean boiling chip to the concentrator tube and attach a two-ball micro-Snyder column. Prewet the column by adding 0.5 ml of methylene chloride to the top of the column. Place the K-D apparatus in a hot water bath so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature, as required to complete the concentration in 5 – 10 minutes. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of the liquid reaches 0.5 ml, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes. Remove the Snyder column, rinse the flask and its lower points into the concentrator tube with 0.2 ml of methylene chloride and adjust the final volume to 1 ml with solvent.

14.11.2 Nitrogen Blowdown techniques

Place the concentrator tube in a warm bath (35°C) and evaporate the solvent to 1 ml using a gentle stream of clean, dry nitrogen (filtered through activated carbon).

CAUTION: New plastic tubing must not be used between the carbon trap and the sample, since it may introduce contaminants.

The internal wall of the tube must be rinsed several times with methylene chloride or the appropriate solvent during the operation. During the evaporation, the tube must be positioned to avoid water condensation (i.e. the solvent level should be below the level of the water bath). The extract must not be allowed to become dry.

CAUTION: When the volume of solvent is reduced below 1 ml, semivolatile analytes may be lost.

14.12 After concentration of the extract to its final volume (1.0 ml unless viscous/dirty), the extracts are placed in the refrigerator in the SVOA laboratory. The internal chain of custody must be filled out relinquishing custody of the extracts from the extractions laboratory to the analytical laboratory.

Prior to analysis, the extracts are removed from the refrigerator with the analyst signing them out on the same internal chain of custody. 1.0 ul of the 4000 ug/ml internal standard solution (Table 3) is added to a 100 ul extract aliquot, giving a final internal standard concentration of 40 ug/l.

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15.0 Data Analysis and Calculations:

Refer to the determinative method.

16.0 Method Performance:

Refer to the determinative methods for performance data.

17.0 Pollution Prevention:

17.1 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. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the EPA recommends recycling as the next best option.

17.2 Quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18.0 Data Assessment, Acceptance and Corrective Action:

The following corrective actions should be instituted if deemed appropriate based on results obtained from the selected determinative method:

1) Potential sources of contamination should be investigated and eliminated.

2) Poor surrogate or matrix spike recoveries could be a function of matrix interference. If sample history does not indicate a matrix related problem, the affected samples should be re-extracted and re-analyzed to confirm matrix interference.

3) A corrective action form should be initiated by the individual that discovered the analytical problem. The corrective action form should be filled out by the appropriate personnel with documentation of the procedures to minimize reoccurrence of the problem. Finalized corrective action forms are filed with the laboratory quality assurance manager.

19.0 Waste Management:

Care should be taken to minimize waste generated during performance of this standard operating procedure. Waste should be disposed of in properly labeled

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containers following procedures documented in the chemical hygiene plan, Hazardous Waste Management and Spill Prevention Control and Contingency Plan of NEL.

20.0 References:

Method 3510C- SW846 Revision 3 December 1996

Method 3500B- SW846 Revision 2 December 1996

SW846 Chapter Four Organic Analytes- Revision 3 December 1996

NELAC Quality Systems Chapter 5, June 5, 2003

21.0 Tables, Diagrams, Flowcharts and Validation Data:

See attached tables.

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Table 1 – Surrogate Solution

Supplier	Standard	Initial Conc. (ug/ml)	Volume Added (ml)	Final Conc. (ug/ml)
Restek	Acid surrogate mix	10,000	0.5	100
Restek	Base/neutral surrogate mix	5,000	1.0	100
Restek	MeOH	-	48.5	-

Final Volume = 50 ml
Final Concentration = 100 ug/ml

Table 2 – Lab Control Sample and Matrix Spike Solutions

Supplier	Standard	Initial Conc. (ug/ml)	Volume Added (ml)	Final Conc. (ug/ml)
ECS	Phenols mix	2000	0.5	100
ECS	Benzidines Mix	2000	0.5	100
ECS	EPA 625 Base/neutrals mix 1	2000	0.5	100
ECS	8270 Add ons mix	2000	0.5	100
ECS	PAH mix	2000	0.5	100
Fisher Scientific	MeOH	-	7.5	-

Final Volume = 10 ml
Final Concentration = 100 ug/ml

Table 3 – Internal Standard Solution

Supplier	Standard	Concentration (ug/ml)
Restek	SV Internal Standard mix	4,000

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DOCUMENT HISTORY AND APPROVAL PAGE

TITLE:	**Separatory Funnel Liquid-Liquid Extraction Method 3510C- Pesticides and PCBs**
DEPARTMENTS AFFECTED:	**Organics**

ISSUE DATE:	REVISION CODE:	SOP #:	REVISION CHANGES:
2/05/02	-	3.3.30	Create an SOP in NELAC format
5/18/04	A	3.3.30	Update to include items identified during annual internal method audit and Navy assessment
2/11/05	B	3.3.30	Annual Review & Update
01/16/06	C	3.3.30	Annual Review & Update
12/17/07	D	3.3.30	Annual Review & Update
01/23/08	E	3.3.30	Annual Review & Update
04/09/08	F	3.3.30	Update to include new prep logbook

DOCUMENT APPROVAL		
Originator: Jim Galasyn	Signature/Date: J Galasyn 040908	Title Chemistry Manager
Approved by: Kelsey Anthony	Kelsey Anthony 042108	Chemistry Analyst
Reviewed by: Pamela Doughty	Pamela Doughty 4/21/08	Total Quality Manager

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NEL	STANDARD OPERATING PROCEDURE		
TITLE: **Separatory Funnel Liquid-Liquid Extraction Method 3510C- Pesticides and PCBs**			
SOP# 3.3.30	REV# F	EFFECTIVE 04/09/08	PAGE 2 OF 13
WRITTEN BY: Jim Galasyn	DEPARTMENTS AFFECTED: ORGANICS		

1.0 Test Method:

This standard operating procedure (SOP) is for EPA Separatory Funnel Liquid-Liquid Extraction Method 3510C.

2.0 Applicable Matrix or Matrices:

This SOP is applicable for the extraction of liquid matrices only.

3.0 Detection Limits:

The laboratory's reporting limit shall lie within the calibration range, at or above the lower quantitation limits. If client requires a reporting limit that lies below the lower limit of the calibration curve, then method modification is required.

4.0 Scope and Application:

4.1 This method describes a procedure for isolating organic compounds (water-insoluble and slightly water-soluble) from aqueous samples. The method also describes concentration techniques suitable for preparing the extract for analysis by Gas Chromatography/Electron Capture Detector (GC/ECD)

4.2 This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method.

5. 0 Summary of Method:

5.1 A measured volume of sample, usually 1 liter, is serially extracted (at a pH of 5 - 9) with methylene chloride using a separatory funnel.

5.2 The extract is dried and concentrated (if necessary) and exchanged with hexane prior to cleanup or analysis.

6.0 Definitions:

6.1 Surrogate Spiking Solution - Non-target analytes compounds that are similar in composition and behavior to the target analytes but are not expected to

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be found in environmental media and are added to every standard, quality control sample, and field sample at a known concentration prior to preparation and/or analysis. Surrogate responses are used to evaluate the accuracy of the laboratory's performance of the analytical method in a specific sample matrix.

6.2 Method Blank (BLK or Laboratory Reagent Blank) – An aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, and surrogates that are used with other samples. The BLK is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

6.2 Laboratory Control Spike (LCS) - A reagent water of method blank sample spiked with PCB or Pest spike and run through the method (including extraction) with samples as the quality control check.

6.4 Other terms are as defined in SW – 846.

7.0 Interferences:

7.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be necessary. Please refer to NEL SOP 3.7.1 for washing of glassware and preservation of sample bottles.

7.2 Interferences coextracted from the samples will vary considerably from source to source. If the analysis of an extracted sample is prevented due to interferences, further cleanup of the sample extract may be necessary.

7.3 Phthalate esters contaminate many types of products commonly found in the laboratory. Plastics, in particular, must be avoided because phthalates are commonly used as plasticizers and are easily extracted from plastic materials. Serious phthalate contamination may result at any time if consistent quality control is not practiced.

7.4 The decomposition of some analytes has been demonstrated under basic extraction conditions. Organochlorine pesticides may dechlorinate, phthalate esters may exchange and phenols may react to form tannates. These reactions increase with increasing pH, and are decreased by the shorter reaction times available in Method 3510. Method 3510 is preferred over method 3520 for the analysis of these classes of compounds.

8.0 Safety

8.1 Analysts should read and understand the laboratory chemical hygiene plan before handling any reagents specified in this standard operating procedure.

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8.2 Analysts should wear the proper protective attire when handling the chemicals referenced in this standard operating procedure. Proper protective attire includes labcoats, chemically resistant gloves and aprons, safety glasses and goggles. Open-toed shoes should not be worn in the laboratory.

8.3 Sample extraction should be performed in a hood with the hood sash pulled down to ensure that analyst exposure to solvents is minimized. Analysts should read the applicable MSDS sheets and be aware of hazards, odor thresholds and cleanup procedures required in case of an accidental spill.

8.4 Methylene chloride creates excessive pressure very rapidly. Therefore, initial venting should be done immediately after the separatory funnel has been sealed and shaken once. The separatory funnel should be vented into a hood to avoid exposure of the analyst to solvent vapors.

8.5 Syringes are used to spike surrogates and matrix spiking into samples or sample extracts. Care should be taken to ensure that the analyst is not mistakenly injected with a syringe.

9.0 Equipment and Supplies

9.1 Separatory funnel- 2-liter, with polytetrafluoroethylene (PFTE) stopcock.

9.2 Drying column- 20 mm ID Pyrex chromatographic column or glass funnels with glass wool at the bottom. Prewash the glass wool pad with 50 ml of methylene chloride prior to packing the column/funnel with adsorbent or equivalent.

9.3 Kuderna-Danish (K-D) apparatus.

- 9.3.1 Concentrator tube- 10 ml, graduated (Kontes K-5700250-1025 or equivalent). A ground glass stopper is used to prevent evaporation of extracts.
- 9.3.2 Evaporation flask- 500 ml (Kontes K-570001-500 or equivalent). Attach to concentrator tube with springs, clamps, or equivalent.
- 9.3.3 Snyder column- Three-ball macro (Kontes K-503000-0121 or equivalent)
- 9.3.4 Snyder column- Two-ball micro (Kontes K-569001-0219 or equivalent)
- 9.3.5 Springs- ½ inch (Kontes K-662750 or equivalent)
  Alternately, use of N-EVAP system may be used to concentrate the extracts.

9.4 Boiling chips- Solvent extracted, approximately 10/40 mesh (silicon carbide or equivalent)

9.5 Water bath- Heated, with concentric ring cover, capable of temperature control (+/_ 5°C). The bath should be used in a hood.

9.6 Vials- 2 ml, glass with PTFE-lined screw caps or crimp tops.

9.7 pH indicator paper- pH range including the desired extraction pH.

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9.8 Erlenmeyer flask- 250 ml.
9.9 Syringe- 1ml
9.10 Graduated cylinder- 1 liter

10.0 Reagents and standards

10.1 Reagent grade chemicals should be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. Reagents should be stored in glass to prevent the leaching of contaminants from plastic containers. Extraction solvent is methylene chloride- Pesticide grade. The exchange solvent is hexane- Pesticide grade.

10.2 Organic-free reagent water

10.3 Sodium sulfate (granular, anhydrous), Na_2SO_4. Purify by heating to 550 °C for 4 hours in a shallow tray, or by precleaning the sodium sulfate with methylene chloride. If the sodium sulfate is precleaned with methylene chloride, a method blank must be analyzed, demonstrating that there is no interference from the sodium sulfate. Please fill note the details of the sodium sulfate heating process in the logbook.

10.4 Stock standards for spiking solutions- Stock solutions may be prepared from the pure standard materials or purchased as certified solutions. The stock solutions for the calibration standards are acceptable (dilutions must be made in a water miscible solvent) except for the control check sample stock concentrate which must be prepared independently to serve as a check on the accuracy of the calibration solution.

10.4.1 Prepare the stock standard solutions by accurately weighing about 0.0100 g of pure compound. Dissolve the compound in a water miscible solvent (i.e. methanol, acetone, 2-propanol, etc) and dilute to volume in a 10-ml volumetric flask. If compound purity is 96 percent or greater, the weight can be used without correction to calculate the concentration of the stock standard solution. Commercially-prepared stock standard solutions can be used at the concentration if they are certified by the manufacturer or by an independent source.

10.4.2 Stock standard solutions should be stored in PTFE sealed containers at 4 °C or below. Refer to the determinative method for holding times of the stock solutions.

10.5 Surrogate standards- A surrogate (i.e., a compound that is chemically similar to the analyte group but is not expected to occur in an environmental sample) should be added to each sample, blank,

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laboratory control spike (LCS), and matrix spike sample just prior to extraction or processing. The recovery of the surrogate standard is used to monitor for unusual matrix effects, gross sample processing errors, etc. Surrogate recovery is evaluated for acceptance by determining whether the measured concentration falls within the acceptance limits. Surrogates used in this procedure are listed in Table 1.

10.5.1 Prepare a surrogate spiking concentrate by mixing stock standards prepared above and diluting with a water miscible solvent. Commercially prepared spiking solutions are acceptable. The concentration should be such that a 1-ml aliquot into 1000 ml of a sample provides a concentration of 10 times the quantitation limit or near the mid-point of the calibration curve. For matrices other than water, 1 ml of surrogate standard is still the normal spiking volume. However, if gel permeation chromatography will be used for sample cleanup, 2 ml should be added to the sample.

10.6 Matrix spike standards- The recommended matrix spike standard is listed in Table 2. Prepare a matrix spike concentrate by mixing stock standards prepared above and diluting with a water miscible solvent. Commercially-prepared spiking solutions are acceptable. The matrix spike standards should be independent of the calibration standard.

10.7 Laboratory control spike standard- Use the matrix spike standard prepared in Sec. 7.8 as the spike standard for the laboratory control sample (LCS). The LCS should be spiked at the same concentration as the matrix spike.

11.0 Sample collection, preservation, shipment and storage

Containers used to collect samples for the determination of semivolatile organics should be soap and water washed followed by methanol (or isopropanol) rinsing. Alternatively, containers pre-cleaned by the manufacturer should be rinsed with methylene chloride to minimize interferences. The sample containers should be of amber glass or Teflon and have screw-caps with Teflon liners. In situations where Teflon is not available, solvent-rinsed aluminum foil may be used as a liner. However, acidic or basic samples may react with the aluminum foil, causing eventual contamination of the sample. Plastic containers or lids may NOT be used for the storage of samples due to the possibility of sample contamination from the phthalate esters and other hydrocarbons within the plastic. Sample containers should be filled with care so as to prevent any portion of the collected sample coming in contact with the sampler's gloves, thus causing contamination. Samples should not be collected or stored in the presence of exhaust fumes. If the sample comes in contact with the sampler (e.g. if an automatic sampler is used), run organic-free reagent water through the sampler and use as a field blank. A minimum of two 1-liter amber glass containers should be collected for each sample location. Additional sample should be collected if matrix spike/matrix spike duplicates are to be performed. Samples should be cooled to 4 °C +/- 2 °C from the time of sampling to the time of extraction.

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Samples should be extracted within 7 days and extracts analyzed within 40 days following extraction.

12.0 Quality Assurance / Quality Control

12.1 Any reagent blanks, matrix spikes, or replicate samples should be subjected to exactly the sample analytical procedures as those used on actual samples.

12.2 Each laboratory using this method should maintain a formal quality assurance program. Each extraction batch of 20 or less samples should contain: a method blank, either a matrix spike/matrix spike duplicate or a matrix spike and duplicate samples and a laboratory control sample.

12.3 Initial demonstration of Proficiency- Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean reference matrix. This will include a combination of the sample extraction method and the determinative method. The laboratory should also repeat the following operations whenever new staff are trained or significant changes in instrumentation are made.

12.3.1 The reference samples are prepared from a spiking solution containing each analyte of interest. The reference sample concentrate (spiking solution) may be prepared from pure standard materials, or purchased as certified solutions. If prepared by the laboratory, the reference sample concentrate should be made using stock standards prepared independently from those used for calibration.

12.3.2 The procedure for preparation of the reference sample concentrate is dependent of the method being evaluated. The concentration of target analytes in the reference sample may be adjusted to more accurately reflect the concentrations that will be analyzed by the laboratory.

12.3.3 To evaluate the performance of the total analytical process, the reference samples must be handled in exactly the same manner as actual samples. Use a clean matrix for spiking purposes (e.g.. organic-free reagent water for the water matrix).

12.4 Sample Quality Control for Preparation and Analysis

12.4.1 Documenting the effect of the matrix should include the analysis of at least one matrix spike (MS) and one duplicate (DUP) unspiked sample or one matrix spike (MS)/matrix spike duplicate (DUP) pair daily per analytical batch up so 20 samples.

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12.4.2 A Laboratory Control Sample (LCS) should be included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the sample weight or volume. The LCS is spiked with the same analytes at the same concentration as the matrix spike sample. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. If there is insufficient sample to perform the MS/DUP or MS/MSD analyses, a laboratory control spike duplicate (LCSD) should be extracted. LCS (and/or LSCD if performed) should be extracted daily per analytical batch of up to 20 samples.

12.4.3 Analyze these QC samples (the LCS and the matrix spikes or the optional duplicates) following the procedure in the selected determinative method. Calculate and evaluate the QC data.

12.4.4 Blanks- Use of method blanks and other blanks are necessary to track contamination of samples during the sampling and analysis processes. Method blanks should not contain analytes of interest at concentrations great than one-half the reporting limit.

12.4.5 Surrogates- A surrogate is a compound that is chemically similar to the analyte group but not expected to occur in an environmental sample. Surrogates should be added to all samples, LCS, LCSD, MS and MSD samples.

13.0 Calibration and Standardization:

N/A

14.0 Procedure

14.1 Rinse all glassware, sodium sulfate and glass wool with approximately 60 ml methylene chloride and discard. Using a 1-liter graduated cylinder, measure 1 liter (nominal) of sample. Alternately, if the entire contents of the sample bottle are to be extracted, mark the level of the sample on the outside of the bottle. If high analyte concentrations are anticipated, a smaller sample volume may be taken and diluted to 1-L with organic-free reagent water, or the samples may be collected in smaller sample bottles and the whole sample used.

14.2 Spike 1.0 ml of the TCMX-DCB surrogate spiking solution into each sample and mix well.

14.2.1 If analyzing for pesticides only, add 1.0 ml of Pest matrix spike solution to the sample chosen for the matrix spike sample. If analyzing for PCBs only, add 1.0 ml of PCB matrix spike solution to the sample chosen for the matrix spike sample. If analyzing for both pesticides and PCBs, separate aliquots of the matrix spike

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sample should be spiked with the appropriate matrix spiking solution. This same format should be followed for the laboratory control sample/laboratory control sample duplicate (LCS/LCSD), except that the spikes are performed in 1liter aliquots of organic free water.

14.2.2 If method 3640, Gel-Permeation Cleanup, is to be employed, add twice the volume of the surrogate spiking solution and the matrix spiking standard, since half of the extract is not recovered from the GPC apparatus. (Alternately, use 1.0 ml of the spiking solutions and concentrate the final extract to half the normal volume, e.g. 0.5 ml instead of 1.0 ml).

14.3 Quantitatively transfer the sample from the graduated cylinder or sample bottle to the separatory funnel. Use 60 ml of methylene chloride to rinse the cylinder (or bottle) and transfer this rinse solvent to the separatory funnel. If the sample was transferred directly from the sample bottle, refill the bottle to the mark made in Sec. 14.1 with water and then measure the volume of sample that was in the bottle.

14.4 Seal and shake the separatory funnel vigorously for 2 minutes with periodic venting to release excess pressure.

NOTE: Methylene chloride creates excessive pressure very rapidly. Therefore, initial venting should be done immediately after the separatory funnel has been sealed and shaken once. The separatory funnel should be vented in a hood to avoid exposure of the analyst to solvent vapors.

14.5 Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends of the sample and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. Collect the solvent extract in an Erlenmeyer flask. If the emulsion cannot be broken (recovery of <80% of the methylene chloride corrected for the water solubility of methylene chloride), transfer the sample, solvent and emulsion into the extraction chamber of a continuous extractor and proceed as described in Method 3520- Continuous Liquid-Liquid Extraction.

14.6 Repeat the extraction two more times using fresh portions of methylene chloride. Combine the three solvent extracts.

14.7 Perform the concentration (if necessary) using the Kuderna-Danish Technique (Sec 9.10.1 through 9.10.4). Alternatively, the n-EVAP system may be used to concentrate sample extracts.

14.8 K-D technique

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14.8.1 Assemble a Kuderna-Danish (K-D) concentrator by attaching a 10-ml concentrator tube to a 500-ml evaporation flask. Rinse the glassware one time with methylene chloride.

14.8.2 Dry the extract by passing it through a drying column containing about 10 cm of anhydrous sodium sulfate. Collect the dried

extract in a K-D concentrator. Rinse the Erlenmeyer flask, which contained the solvent extract with 20 – 30 ml of methylene chloride and add it to the column to complete the quantitative transfer.

14.8.3 Add one or two clean boiling chips to the flask and attach a three ball Snyder column. Prewet the Snyder column by adding about 1-ml of methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 10 – 20 minutes. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of the liquid reaches 1 ml, remove the K-D apparatus form the water bath and allow it to drain and cool for at least 10 minutes.

14.8.4 Solvent exchange to hexane is required. Momentarily remove the Snyder column, add 50 ml of hexane, a new boiling chip, and reattach the Snyder column. Alternatively, pour the exchange solvent into the top of the Snyder column while the concentrator remains on the water bath. Concentrate the extract, as described in section 14.8.3, raising the temperature of the water bath, if necessary, to maintain proper distillation.

14.8.5 Remove the Snyder column and rinse the flask and its lower joints into the concentrator tube with 1 – 2 ml of hexane. If sulfur crystals are a problem, proceed to method 3660 for cleanup. The extract is then brought to a final volume of 10 ml with hexane.

14.8.6 If analysis of the extract will not be performed immediately, stopper the concentrator tube and store refrigerated. If the extract will be stored longer than 2 days, an aliquot of the extract should be transferred to a glass vial with a PTFE-lined screw-cap and labeled appropriately. A second aliquot should be transferred to a glass vial with a crimp top and labeled appropriately.

14.8.7 Extracts for polychlorinated biphenyls (PCBs) should be cleaned up following the procedure outlined in NEL SOP 3.3.23.

15.0 Calculations:

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N/A

16.0 Method Performance:

Refer to the determinative methods for performance data.

17.0 Pollution prevention

Care should be taken to minimize waste generated during performance of this standard operating procedure. Waste should be disposed of in properly labeled containers following procedures documented in the chemical hygiene plan.

18.0 Data Assessment, Acceptance Criteria, and Corrective Action

The following corrective actions should be instituted if deemed appropriate based on results obtained from the selected determinative method:

1) Potential sources of contamination should be investigated and eliminated.
2) Poor surrogate or matrix spike recoveries could be a function of matrix interference. If sample history does not indicate a matrix related problem, the affected samples should be re-extracted and re-analyzed to confirm matrix interference.
3) A corrective action form should be initiated by the individual that discovered the analytical problem. The corrective action form should be filled out by the appropriate personnel with documentation of the procedures to minimize reoccurrence of the problem. Finalized corrective action forms are filed with the laboratory quality assurance manager.

19.0 Waste Management:

Please refer to the NEL Chemical Hygiene Plan and Spill Prevention Control & Contingency Plan (SPCC) for information regarding waste management.

20.0 References

Method 3510C- SW846 Revision 3 December 1996

Method 3500B- SW846 Revision 2 December 1996

SW846 Chapter Four Organic Analytes- Revision 3 December 1996

NELAC Quality System Chapter 5, June 5, 2003

21.0 Tables, Diagrams, Flowcharts and Validation Data

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Table 1. Surrogates

Standard	Concentration	Compound
TCMX-DCB	1 ug/ml	Tetrachloro-m-xylene Decachlorobiphenyl

Table 2. Matrix spiking solutions

Standard	Concentration	Compound
Pesticide Spike	0.25 ug/ml each component	alpha-BHC beta-BHC gamma-BHC(Lindane) delta-BHC Heptachlor Aldrin
		Heptachlor Epoxide Gamma-Chlordane Endosulfan I Alpha-Chlordane Dieldrin 4,4'-DDE Endrin Endosulfan II 4,4'-DDD Endosulfan Sulfate Endrin Aldehyde 4,4'-DDT Endrin Ketone Methoxychlor
PCB Spike	10 ug/ml	Aroclor 1254

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EPA Method 3510C
Sodium Sulfate Prep Logbook
SOP 3.3.30

Sodium Sulfate Tag #	Date Prepared	Time In	Temp. In (C)	Time Out	Temp Out (C)

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REV:	H
PAGE:	1 of 33

DOCUMENT HISTORY AND APPROVAL PAGE

TITLE:	**Chlorinated Herbicides by Gas Chromatography (8151A)**
DEPARTMENTS AFFECTED:	**Organics**

ISSUE DATE:	REVISION CODE:	SOP #:	REVISION CHANGES:
04/22/02	-	3.3.5	Update to NELAC Format
01/29/03	A	3.3.5	Added extraction Procedures
05/10/04	B	3.3.5	Update to include items identified during annual internal audit & comments during Navy assessment
07/20/04	C	3.3.5	Modification to prep procedure
02/09/05	D	3.3.5	Modification to correct dates, titles and page numbers
10/24/06	E	3.3.5	Annual Review & Update
02/14/07	F	3.3.5	Annual Review & Update
01/23/08	G	3.3.5	Annual Review & Update
04/14/08	H	3.3.5	Update to Section 14.1.2.4 and 14.6

DOCUMENT APPROVAL		
Originator: James Galasyn, Ph.D.	Signature/Date: J Galasyn 04/14/08	Title Chemistry Lab Manager
Approved by: Joe Casarin	Joe Casarin 04/14/08	Organics Supervisor
Reviewed by: Pamela Doughty	Pamela Doughty 4/14/08	Total Quality Manager

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Northeast Laboratory Services, Inc.	Standard Operating Procedure		
TITLE: Chlorinated Herbicides by Gas Chromatography (8151A)			
SOP# 3.3.5	REV# H	EFFECTIVE 04/14/08	PAGE 2 of 33
WRITTEN BY: James Galasyn, Ph.D.	DEPARTMENTS AFFECTED: Organics		

1.0 Test Method:

8151A

2.0 Applicable Matrix or Matrices:

This standard operating procedure (SOP) is used to determine the concentrations of various chlorinated herbicides in aqueous, soil and waste matrices.

3.0 Detection Limit:

Reporting limits for aqueous samples are in the low part per billion range. The sensitivity of method 8151 depends on the level of interferences in addition to instrumental limitations. Current method detection limits studies are on file in the QA/QC manager's office. The laboratory's reporting limit shall lie within the calibration range, at or above the lower quantitation limit. If the client requires a reporting limit that lies below the lower limit of the calibration curve, then method modification is required.

4.0 Scope and Application:

4.1 Method 8151A is used to determine the concentrations of various chlorinated herbicides and related compounds in aqueous, soil and waste matrices, using fused-silica, open tubular, capillary columns with electron capture detectors (ECD). When compared to the packed columns, these columns offer improved resolution, better selectivity, increased sensitivity, and faster analysis. When this method is used to analyze unfamiliar samples for any or all of the compounds listed below, 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. Gas chromatograph/mass spectrometer (GC/MS) may be used for the qualitative and quantitative confirmation of results for all of the parameters listed below.

Compound	CAS Registry No.
2,4-D	94-75-7
2,4-DB	94-82-6
2,4,5-TP (Silvex)	93-72-1
2,4,5-T	93-76-5
Dalapon	75-99-0
Dicamba	1918-00-9

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Dichloroprop	120-36-5
Dinoseb	88-85-7
MCPA	94-74-6
MCPP	93-65-2
4-Nitrophenol	100-02-1
Pentachlorophenol	87-86-5

The following compounds may also be determined using this method:

Acifluorfen	50594-66-6
Bentazon	25057-89-0
Chloramben	133-89-0
DCPA diacid	2136-79-0
3,5-Dichlorobenzoic Acid	51-36-5
5-Hydroxydicamba	7600-50-2
Picloram	1918-02-1

4.2 The analyst must select columns, detectors and calibration procedures most appropriate for the specific analytes of interest in a study. Matrix specific performance data must be established and the stability of the analytical system and instrument calibration must be established for each analytical matrix.

4.3 Compound identification based on single-column analysis should be confirmed on a second column, or should be supported by at least one other 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. GC/MS is also recommended as a confirmation technique, if sensitivity permits.

4.4 This method includes a dual-column option. This option allows a hardware configuration of two analytical columns joined to a single injection port. This option allows one injection to be used for dual-column analysis. Analysts are cautioned that the dual-column option may not be appropriate when the instrument is subject to mechanical stress, many samples are to be run in a short period or when contaminated samples are analyzed.

4.5 This method is restricted to use by, or under the supervision of, analysts experienced in the use of gas chromatographs (GC) and skilled in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method.

5.0 Summary of the Method:

5.1 This SOP provides gas chromatographic conditions for the analysis of chlorinated acid herbicides in water, soil and waste samples.

5.2 Water samples are extracted with diethyl ether and then esterified with either diazomethane or pentafluorobenzyl bromide. The derivatives are determined by gas chromatography with an electron capture detector (GC/ECD). The results are reported as acid equivalents.

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5.3 Soil and waste samples are extracted and esterified with either diazomethane or pentafluorobenzyl bromide. The derivatives are determined by gas chromatography with an electron capture detector (GC/ECD). The results are reported as acid equivalents.

6.0 Definitions:

6.1 SOP-Standard Operating Procedure

6.2 GC- Gas chromatograph

6.3 ECD- Electron capture detector

6.4 PTFE- polytetrafluoroethylene

6.5 SURROGATE ANALYTE (SS) - Non-target analytes compounds that are similar in composition and behavior to the target analytes but are not expected to be found in environmental media (often, isotopically labeled target analytes are used for this purpose) and are added to every standard, quality control sample, and field sample at a known concentration prior to preparation and/or analysis. Surrogate responses are used to evaluate the accuracy of the laboratory's performance of the analytical method in a specific sample matrix.

6.6 STOCK STANDARD SOLUTION - A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.

6.7 PRIMARY DILUTION STANDARD SOLUTION - A solution of several analytes prepared in the laboratory from stock standard solutions and diluted as needed to prepare calibration solutions and other needed analyte solutions.

6.8 CALIBRATION STANDARD (CAL) - A solution prepared from the primary dilution standard solution or stock standard solutions and the internal standards and surrogate analytes. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

6.9 CONTINUING CALIBRATION CHECK STANDARD (CCC) - An analytical standard containing all target analytes, surrogate and internal standard compounds that is used to evaluate the performance of the instrument system with respect to a defined set of method criteria.

6.10 METHOD BLANK (BLK, similarly known as the LABORATORY REAGENT BLANK) - An aliquot of organic free reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The BLK is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

6.11 LABORATORY CONTROL SAMPLE (LCS) - The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or

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volume. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. When the results of the matrix spike analysis indicates a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. The LCS and its duplicate also serves as a second source check of the calibration, as it is prepared from a different vendor's standards.

6.12 MATRIX SPIKE/ MATRIX SPIKE DUPLICATE (MS/MSD) - An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS/MSD is analyzed exactly like a sample. Its purpose is used to evaluate the accuracy and precision of the laboratory performance of the analytical method in a specific sample matrix.

6.13 REAGENT WATER – Organic free water. NEL uses carbon filtration of water from its Milli-Q deionized water system, or equivalent.

7.0 Interferences:

7.1 Method interferences may be caused by contaminants in solvents, reagents, glassware and other sample processing hardware that lead to discrete artifacts or elevated baselines in gas chromatograms. All these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis through the analysis of reagent blanks.

7.1.1 Glassware must be scrupulously cleaned. Clean each piece of glassware as soon as possible after use by rinsing it with the last solvent used in it. This should be followed by detergent washing with hot water and rinses with tap water, then with organic-free reagent water. Glassware should be solvent rinsed with acetone and pesticide-quality hexane. After rinsing and drying, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. Store glassware inverted or capped with aluminum foil. Immediately prior to use, glassware should be rinsed with the next solvent to be used.

7.1.2 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.

7.2 Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerable from waste to waste, depending on the nature and diversity of the waste being sampled.

7.3 Organic acids, especially chlorinated acids, cause the most direct interference with the determination by methylation. Phenols, including chlorophenols, may also interfere with this procedure. The determination using pentafluorobenzylation is more sensitive, and

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more prone to interferences from the presence of organic acids or phenols than by methylation.

7.4 Alkaline hydrolysis and subsequent extraction of the basic solution removes many chlorinated hydrocarbons and phthalate esters that might otherwise interfere with the electron capture analysis. However, hydroysis may result in the loss of dinoseb and the formation of aldol condensation products if any residual acetone remains from the extraction of solids.

7.5 The herbicides, being strong organic acids, react readily with alkaline substances and may be lost during analysis. Therefore, glassware must be acid-rinsed and then rinsed to constant pH with organic-free water. Sodium sulfate must be acidified.

7.6 Sample extracts should be dry prior to methylation or else poor recoveries will be obtained.

8.0 Safety:

The laboratory is responsible for maintaining a current awareness of OSHA regulations regarding safe handling of chemicals. Please refer to the Northeast Laboratory Chemical Hygiene Plan and Spill Prevention Control and Contingency Plan for further information.

9.0 Equipment and Supplies:

9.1 Gas Chromatograph: an analytical system complete with gas Chromatograph suitable for on-column and/or split-splitless injection and all required accessories including syringes, autosampler vials and crimp top caps, autosampler vial inserts, autosampler vial cap crimper and decrimper, analytical columns, gases, electron capture detectors (ECD) and recorder/integrator or data system.

9.2 This method describes procedure for both single-column and dual-column analyses. The single-column approach involves one analysis to determine that a compound is present, followed by a second analysis to confirm the identity of the compound.

NEL usually uses Column pair 2. Other columns may be used provided they equal or exceed the performance described in this method, or as appropriate for the intended application.

9.2.1 Column pair 1 (two out of the following three columns)

30 m x 0.53 mm ID fused silica capillary column chemically bonded with SE-54 (RTX-5 or equivalent) 0.25 um film thickness

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30 m x 0.53 mm ID fused silica capillary column chemically bonded with 35 percent phenyl methylpolysiloxane (DB608 or equivalent) 0.83 um film thickness

30m x 0.53 mm ID fused silica capillary column chemically bonded with 50 percent phenyl methylpolysiloxane (RTX-1701 or equivalent) 0.25 um film thickness

Recommended oven temperature program and operating parameters:
Injection port temperature: 250 °C
Detector A or Detector B temperature:320 °C
Oven Temperature program:
Initial Temperature: 60°C
Ramp 1: 4.0 °C/min
Final Temperature: 300 °C
Final Hold: 0 minutes
Oven Max temperature: 310 °C
Equilibration Time: 1.00 minute
Delay Time: 0 minutes
Sampling Rate 1.2500 pts/s
Injection volume: 1.0 ul
Injection Speed: NORM
Sample washes: 5
Sample Pumps: 5
Viscosity Delay: 0
Solvent A Washes: 5
Solvent B Washes: 5
Carrier gas: Helium (5 ml/min)

9.2.2 Column pair 2 – NEL currently uses this option.

30 m x 0.25 mm ID fused silica capillary column chemically bonded with DB-XLB or equivalent- 0.5 um film thickness

30 m x 0.32 mm ID fused silica capillary column chemically bonded with DB-35MS or equivalent- 0.25 um film thickness

Recommended oven temperature program and operating parameters:
Injection port temperature: 250° C
Detector A or Detector B temperature:320° C
Oven Temperature program:
Initial Temperature: 50° C
Initial Hold: 0.5 minutes
Ramp 1: 25.0° C/min
Final Temperature: 100° C
Ramp 2: 12.0° C/min
Final Temperature: 320° C
Final Hold: 4.00 minutes
Oven Max temperature: 325° C
Equilibration Time: 3.0 minutes
Sampling Rate 1.2500 pts/s

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Injection volume: 1 ul
Injection Speed: NORM
Pre-injection sample washes: 1
Post-injection solvent washes (A) 2
Post-injection solvent washes (B) 2
Sample Pumps: 2
Carrier gas: Helium (5 ml/min)
Collection delay time: 3.00 minutes

9.3 Disposable Pasteur pipets- flint glass Fisherbrand Catalogue #13-678-6A (5 ¾ inch) and Fisherbrand Catalogue #13-678-6B (9 inch) or equivalent

9.4 Safeskin purple nitrile exam gloves Fisher Catalogue # 55082 or equivalent

9.5 Water bath- Heated, with concentric ring cover, capable of temperature control. The bath should be in a hood.

9.6 Balance- Analytical, capable of accurately weighing 0.0001 g.

9.7 Kuderna-Danish (K-D) apparatus

9.8 Diazomethane Generator

9.9 Funnel - 75 mm diameter

9.10 Separatory Funnel - 2000 ml and 125 ml with PTFE stopcock

9.11 Erlenmeyer Flask- 250 ml

9.12 Glass wool - acid washed

9.13 Ultrasonic extraction system - 1 horn-type device equipped with titanium tip or equivalent. The disrupter must have a minimum power wattage of 300 watts, with pulsing capability. Use a 3/4" horn for most samples.

9.14 Sonabox

9.15 pH paper - wide range.

10.0 Reagents and Standards:

10.1 Reagent grade or pesticide grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

10.1.1 Diethyl Ether

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10.1.2 Hexane
10.1.3 Methanol
10.1.4 Acetone
10.1.5 Isooctane
10.1.6 Organic-free reagent water
10.1.7 Methylene chloride
10.1.8 Carbitol
10.1.9 Sodium Hydroxide Solution (0.1 N), NaOH - Dissolve 4 g of NaOH in organic-free reagent water and dilute to 1 L.
10.1.10 Potassium Hydroxide Solution (37% aqueous w/v), KOH - dissolve 37 g of KOH in organic-free reagent water and dilute to 100 ml.
10.1.11 Diazald
10.1.12 Silicic acid - 100 mesh powder, store at 130 ° C.
10.1.13 Sodium Sulfate (granular, acidified, anhydrous), Na2SO4 - purify by heating at 400 ° C for 4 hours, or by pre-cleaning with methylene chloride. Acidify by slurrying 100 g Na2SO4 with enough diethyl ether or just cover the solid, then add 0.1 ml of concentrated sulfuric acid and mix thoroughly. Remove the ethyl ether under vacuum. Mix 1 g of the resulting solid with 5 ml of organic-free reagent water and measure the pH. It must be below a pH of 4. Store the acidified Na2SO4 at 130 ° C.
10.1.14 Sodium Hydroxide, NaOH, 6 N
10.1.15 Sulfuric Acid, H2S04, 12 N.
10.1.16 Sodium Chloride – Reagent grade

10.2 Stock standard solutions may be prepared from pure standard materials or can be purchased as certified solutions. Northeast Laboratory Services attempts to purchase standards as certified solutions whenever possible. In general, standards are purchased as composite stock standards whenever possible. Composite solutions purchased are listed in Table 1.

Note: Store the standard solutions (stock, composite, calibration, internal Standard and surrogate) at 4 °C in polytetrafluoroethylene (PTFE) sealed containers in the dark. Stock standard solutions should be checked frequently for signs of degradation or evaporation, especially prior to preparing calibration standards from them. Stock standard solutions of the derivatized acids must be replaced after 1 year, or sooner if comparison with check standards indicates a problem. Stock standard solutions of the free acids degrade more quickly and should be replaced after two months, or sooner if comparison with check standards indicates a problem.

10.3 Calibration standards should be prepared at a minimum of five different concentrations by dilution of the composite stock standard with diethyl ether or hexane. One of the standards should be at a concentration near, but above the method detection limit (MDL). The concentrations should correspond to the expected range of concentrations found in real samples and should bracket the linear range of the detector. Calibration solutions must be replaced after six months, or sooner if comparison with check standards indicates

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a problem. The following concentrations are used: See Table 2 for recommended calibration levels. Other concentrations may be used if deemed appropriate by the analyst.

10.4 Surrogate Standards

The performance of the method should be monitored using surrogate compounds. Surrogate standards are added to all samples, method blanks, matrix spikes and calibration standards. The recommended surrogate is 2,4-Dichlorophenylacetic acid (DCAA). Surrogate spiking solutions should be stored in the dark at 4 °C in PTFE-sealed containers and replaced after six months or sooner is comparison with check standards indicates a problem. DCAA is prepared at a concentration of 10 ug/ml.

11.0 Sample Collection, Preservation, and Storage:

11.1 Aqueous samples-Containers used to collect samples for the determination of semivolatile organics should be soap and water washed followed by methanol (or isopropanol) rinsing. Alternatively, containers pre-cleaned by the manufacturer should be rinsed with methylene chloride to minimize interferences. The sample containers should be of Amber glass or Teflon and have screw-caps with Teflon liners. In situations where Teflon is not available, solvent-rinsed aluminum foil may be used as a liner. However, acidic or basic samples may react with the aluminum foil, causing eventual contamination of the sample. Plastic containers or lids may NOT be used for the storage of samples due to the possibility of sample contamination from the phthalate esters and other hydrocarbons within the plastic. Sample containers should be filled with care so as to prevent any portion of the collected sample coming in contact with the sampler's gloves, thus causing contamination. Samples should not be collected or stored in the presence of exhaust fumes. If the sample comes in contact with the sampler (e.g. if an automatic sampler is used), run organic-free reagent water through the sampler and use as a field blank. A minimum of two 1-liter amber glass containers should be collected for each sample location. Additional sample should be collected if matrix spike/matrix spike duplicates are to be performed. Samples should be cooled to 4 °C +/- 2 °C from the time of sampling to the time of extraction. Samples should be extracted within 7 days and extracts analyzed within 40 days following extraction.

11.2 Solid samples should be collected in glass jars with teflon-lined caps. Solid samples should be extracted within 14 days of sample collection and analyzed within 40 days of extraction.

12.0 Quality Control:

12.1 For each 12-hour period during which analysis is performed, the performance of the entire analytical system should be checked. These checks should be part of a formal quality control program that

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includes the analysis of blanks, calibration standards, matrix spikes, laboratory control samples and replicate samples, although all of these checks need not be performed during each shift.

12.2 Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. The calibration should be verified each 12-hour shift by injecting calibration verification standards prior to conducting any sample analyses. A calibration standard must also be injected at intervals of not less than once every twenty samples and at the end of the analysis sequence. DOD requires that calibration standards be run every 10 samples.

12.2.1 The percent difference between the continuing calibration and the initial calibration should be within +/- 15 percent. Percent difference is calculated as follows:

$$\% \text{ difference} = \frac{\text{Concentration (actual)} - \text{Concentration (expected)}}{\text{Concentration (expected)}} \times 100$$

12.2.1.1 If this criterion is exceeded for any analyte, calculate the average percent difference across all analytes. If the average of the responses for all analytes is within +/- 15%, then the calibration has been verified. The data user must be provided with the calibration verification or a list of those analytes that exceeded the +/- 15% limit.

12.2.1.2 If the continuing calibration does not meet the +/- 15% limit (either on the basis of each compound or the average across all compounds), check the instrument operating conditions, and if necessary, restore them to the original settings and inject another aliquot of the calibration verification standard. If the response for the analyte is still not within +/- 15%, then a new initial calibration must be prepared. However, if the standard analyzed after a group of samples exhibits a response for an analyte that is above acceptance limit, i.e. >15%, and the analyte was not detected in the specific samples analyzed during the analytical shift, then the extracts for those samples do not need to be re-analyzed, as the verification standard has demonstrated that the analyte would have been detected were it present. In contrast, if an analyte above QC limits was detected in the sample, then re-injection is necessary to ensure accurate quantitation. If an analyte was not detected in the samples and the standard response is more than 15% below the initial calibration response, then re-injection is necessary to ensure that the

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detector response has not deteriorated to the point that the analyte would not have been detected even though it was present.

12.2.2 The absolute retention time for each analyte and surrogate should be centered around the retention time of the calibration verification standard at the beginning of the analytical shift. Each subsequent injection of a standard during the 12-hour analytical shift must be checked against the retention time windows. If any of these subsequent standards fall outside their absolute retention time windows, the GC system is out of control. Determine the cause of the problem and correct it. If the problem cannot be corrected, a new initial calibration must be performed.

12.3 In addition to the quantitative measure of comparison, analysts should evaluate chromatograms and instrument operation. Questions that should be asked include:

Do the peaks look normal (Gaussian)?
Is the response obtained compared to the response from previous calibrations?
Do the column fittings need tightening?
Are non-target peaks present in calibration analyses?
Are contaminants present in the blanks?
Is the injector leaking?

12.4 Recalibration of the instrument must take place when the performance changes to the point that the calibration verification acceptance criteria cannot be achieved. In addition, significant maintenance activities or hardware changes may also require recalibration. The sections below provide general guidance on the sorts of procedure that may or may not require recalibration.

12.4.1.1 There are various types of instrument maintenance that should not automatically require recalibration of the instrument. Examples include changing: septa; compressed gas cylinders;syringes; moisture, hydrocarbon or oxygen traps; split seals; column fittings; inlets or filaments. Other procedures include breaking off or changing a guard column or cleaning the inlet. Whenever such procedures are performed, the analyst must demonstrate that the results for a calibration verification standard meet the acceptance criteria before the analysis of any samples. Otherwise, recalibration is required.

12.4.1.2 In contrast to section 12.4.4.1, some maintenance procedures are so likely to affect the instrument response that recalibration is automatically required, regardless of the ability to meet the calibration verification acceptance criteria. These procedures include: changing, replacing or reversing the column or changing

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the injection port. Whenever such procedures are performed, the analyst must perform a new initial calibration that meets the requirements. Changing or replacing the column will also require that the retention time windows be redetermined.

12.5 Initial Demonstration of Proficiency

12.5.1 Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat the following operations whenever new staff are trained or significant changes in instrumentation are made.

12.5.2 Calculate the average recovery and standard deviation of the recoveries of the analytes in each of the four QC reference samples. Compare these values to those published in SW846 method 8151A. If any individual value falls outside the range for accuracy, then the system performance may be unacceptable for that analyte. When comparing the laboratory data to performance data developed from single-laboratory data, certain analytes may be outside the limits, however, the majority should be within the acceptance limits.

12.5.3 Even when the determinative method contains performance data, the development of in-house acceptance limits is strongly recommended.

12.6 Sample Quality Control for Preparation and Analysis

The laboratory must also have procedures for documenting the effect of matrix on method performance (precision, accuracy, and detection limit). At a minimum, this includes the analysis of QC samples including a method blank, a matrix spike, a duplicate or matrix spike duplicate, a laboratory control sample (LCS), and the addition of surrogates to each field sample and QC sample.

12.6.1 Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair. The decision on whether to prepare and analyze duplicate samples or matrix spike/matrix spike duplicate must be based on a knowledge of the samples in the sample batch. If samples are expected to contain target analytes, then laboratories may use one matrix spike and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, the laboratory should use a matrix spike and a matrix spike duplicate pair.

12.6.2 In-house method performance criteria should be developed using the guidance found in Sec. 8.0 of Method 8000 for procedures for evaluating method performance.

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12.6.3 A Laboratory Control Sample (LCS) should be included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicates a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. In instances where insufficient sample is provided for MS/MSD analyses the laboratory will prepare and analyze a LCS/LCSD pair.

12.6.4 Include a continuing calibration standard after each group of 20 samples (10 samples for DOD samples) and at the end in the analysis sequence as a calibration check. Thus, injections of method blank extracts, matrix spike samples and other non-standards are counted in the total. Solvent blanks, injected as a check on cross-contamination, need not be counted in the total. The response for the continuing calibration should be within +/- 15% of the initial calibration (see Sec. 12.2.1). When this calibration verification standard falls out of this acceptance window, the laboratory should stop analyses and take corrective action.

12.6.5 Method blanks should not contain analytes of interest above one-half the reporting limit.

12.7 Surrogate recoveries: The laboratory must evaluate surrogate recovery data form individual samples versus the surrogate control limits developed by the laboratory.

Surrogate recovery is calculated as:

$$\text{Recovery (\%)} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) added}} \times 100$$

If recovery is not within in-house surrogate recovery limits, the following procedures are necessary.

12.7.1 Check to be sure that there are no errors in the calculations or surrogate solutions. If errors are found, recalculate the data accordingly. Examine chromatograms for interfering peaks and integrated peak areas.

12.7.2 Check instrument performance. If an instrument performance problem is identified, correct the problem and re-analyze the extract.

12.7.3 Some samples may require dilution in order to bring target analytes within the calibration range or to overcome significant interferences with some analytes. This may result in the dilution of the surrogate responses to the point that the recoveries cannot be measured. If the surrogate recoveries are available from a less-diluted or undiluted aliquot of the sample or sample extract, those recoveries may be used to demonstrate that the surrogates were within the QC limits, and no further action is required. However, the

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results of both the diluted and undiluted (or less-diluted) analyses should be provided to the data user.

12.7.4 If no instrument problem is found, the sample should be re-extracted and re-analyzed.

12.7.5 If upon re-analysis, the recovery is again not within limits, report the data as an "estimated concentration". If the recovery is within the limits for the re-analysis, provide the re-analysis data to the data user. If the holding time for the method has expired prior to the re-analysis, provide both the original and re-analysis results to the data user and note the holding time problem.

12.8 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies. Please refer to Table 3 for a summary of QC acceptance criteria.

12.9 Please refer to NEL SOP 3.1.7 for proper documentation regarding manual integration of chromatographic peaks.

13.0 Calibration and Standardization:

13.1 Calibration of an analytical instrument involves the delineation of the relationship between the response of the instrument and the amount or concentration of an analyte introduced into the instrument. The graphical depiction of this relationship is often referred to as the calibration curve. In order to perform quantitative measurements, this relationship must be established prior to the analysis of any samples, and thus, is termed the initial calibration. The general procedure for performing an initial calibration is described here. Table 2 lists calibration levels used for routine 8151 analysis. A subset of this list may be used as long as all project analytes are included in all calibrations.

13.1.1 For each analyte and surrogate of interest, prepare calibration standards at a minimum of five different concentrations by adding volumes of one or more stock standards to volumetric flask and diluting to volume with an appropriate solvent. Alternatively, solvent may be measured with the use of a volumetric pipet. A known volume of solvent is then removed from the measured volume, and stock standard solution is added (the same volume as previously removed).

13.1.2 The lowest concentration calibration standard that is analyzed during an initial calibration establishes the method quantitation limit based on the final volume of extract (or sample) described in the preparative method or employed by the laboratory.

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13.1.3 The other concentrations should define the working range of the detector or correspond to the expected range of concentrations found in actual samples that are also within the working range of the detector.

13.1.4 For each analyte, at least one of the calibration standards should correspond to a sample concentration at or below that necessary to meet the data quality objectives of the project, which may include establishing compliance with a regulatory or action limit.

13.1.5 Once the standards have been prepared, the initial calibration begins by establishing chromatographic operating parameters that provide instrument performance required to meet the data quality objectives of this method.

13.1.6 Each calibration standard should be introduced into the instrument using the same technique that is used to introduce the actual samples into the gas chromatograph.

13.1.7 External calibration procedure will be used for quantitation of all samples, standards, etc. External standard calibration involves the comparison of instrument responses from the sample to the responses from the target compounds in the calibration standards. Sample peak areas (or peak heights) are compared to peak areas (or peak heights) of the standards. A calibration curve is then plotted. Linear or non-linear calibration (in situations where the analyst knows that the instrument response does not follow a linear model over a sufficiently wide working range) may be used. Six standards are required for a 2nd order calibration. The curve fit should be the same in the initial calibration as for each sample quantitated against it. The correlation coefficient of the initial calibration should be 0.99 or higher for all analytes and surrogates. A hardcopy of the initial calibration, including the raw data, the curve fit type and the resulting correlation coefficient should be maintained by the laboratory.

13.2 Retention Time Windows

Retention time windows are crucial to the identification of target compounds. Absolute retention times are used for compound identification. Retention time windows are established to compensate for minor shifts in absolute retention times as a result of sample loadings and normal chromatographic variability. The width of the retention time window should be carefully established to minimize the occurrence of both false positive and false negative results. Tight retention time windows may result in false negatives and/or may cause unnecessary reanalysis of samples when surrogates or spiked compounds are erroneously not identified. Overly wide retention time windows may result in false positive results that cannot be confirmed upon further analysis.

13.2.1 Before establishing retention time windows, make sure the chromatographic system is operating reliably and that the system conditions have been optimized for the target analytes and surrogates in the sample matrix to be analyzed. Make three injections of all single component standard mixtures and multi-component analytes over the course of a 72-hour period. Serial

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injections or injections over a period of less than 72 hours may result in retention time windows that are too tight.

13.2.2 Record the retention time for each single component analyte and surrogate to three decimal places. Calculate the mean and standard deviation of the three absolute retention times for each single component analyte and surrogate. For multi-component analytes, choose three to five major peaks and calculate the mean and standard deviation of those peaks.

13.2.3 If the standard deviation of the retention times for a compound is 0.000, then the laboratory may either collect data from additional injections of standards or use a default standard deviation of 0.01 minutes.

13.2.4 The width of the retention time window for each analyte, surrogate, and major constituent in multi-component analytes is defined as +/- 3 times the standard deviation of the mean absolute retention time established during the 72-hour period. If the default standard deviation is employed, the width of the window will be 0.03 minutes.

13.2.5 Establish the center of the retention time window for each analyte and surrogate by using the absolute retention time for each analyte and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration.

13.2.6 The laboratory must calculate absolute retention time windows for each analyte and surrogate on each chromatographic column and instrument. New retention time windows must be established when a new GC column is installed.

13.2.7 The surrogates are added to each sample, blank and QC sample and are also contained in each calibration standard. Although the surrogates may be diluted out of certain sample extracts, their retention times in the calibration standards may be useful in tracking retention time shifts. Whenever the observed retention time of a surrogate is outside of the established retention time windows, the analyst is advised to determine the cause and correct the problem before continuing analyses. The experience of the analyst should weigh heavily in the interpretation of the chromatograms.

14.0 Procedure:

14.1 Extraction and Hydrolysis of Soil, Sediment, and other Solid Samples

14.1.2 Ultrasonic Extraction

14.1.2.1 Rinse all glassware with methylene chloride. Discard all rinse solution. Add 30 g (dry weight) of the well-mixed solid sample to a 400-ml thick-wall beaker. Adjust the pH to 2 with concentrated

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hydrochloric acid and thoroughly mix the contents with a glass stirring rod. Spike the sample with 500 ul of DCAA solution (10 ug/ml).

14.1.2.2 The ultrasonic extraction of solids must be optimized for each type of sample. In order for the ultrasonic extractor to efficiently extract solid samples, the sample must be free flowing when the solvent is added. Acidified anhydrous sodium sulfate should be added to clay type soils (normally 1:1), or any other solid that is not a free flowing sandy mixture, until a free flowing is obtained.

14.1.2.3 Add 100 ml of methylene chloride/acetone (1:1 v/v) to the beaker. Perform ultrasonic extraction for 3 minutes, with output control knob set at 10 (full power) and with mode switch on Pulse (pulsing energy rather than continuous energy) and percent-duty cycle knob set at 50% of time and off 50% time). Allow the solids to settle. Filter through a glass funnel lined with filter paper and approximately 50 grams of acidified sodium sulfate and collect in a 500 ml flask.

14.1.2.4 Ultrasonically extract the sample once more using 100 ml of methylene chloride, then once more with 100 ml of methylene chloride. The same ultrasonic conditions are used

14.1.2.5 Filter each extract through the acidified sodium sulfate and combine extracts.

14.1.2.6 Quantitatively transfer the contents of the flask to a 500 ml K-D flask with a 10 ml concentrator tube attached. Add boiling chips and attach the macro Snyder column. Evaporate the extract on the water bath to a volume of approximately 1 ml. Remove the flasks from the water bath and allow them to cool. Refer to 14.3.3.1.

14.1 Preparation of Aqueous Samples

14.2.1 Rinse all glassware with diethyl ether. Using a graduated cylinder if it is a TCLP, transfer a 1-L sample aliquot to a 2-L separatory funnel. Spike the sample with 0.5 ml of 10 ug/ml DCAA surrogate.

14.2.2 Add 40 g of NaCl to the sample, seal, and shake to dissolve the salt.

14.2.3 <u>Use this step only if herbicide esters, in addition to herbicide acids, are to be determined.</u>

14.2.3.1 Add 3 ml of 6 N NaOH to the sample, seal and shake. Check the pH of the sample with pH paper. If the samples does not have a pH greater than or equal to 12, adjust the pH by adding more 6 N NaOH. Let the sample sit at room temperature until the hydrolysis step is completed (usually 1-2 hours), shaking and venting the separatory funnel and contents periodically.

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14.2.3.2 Add 60 ml of methylene chloride to the sample bottle and rinse both the bottle and the graduated cylinder. Transfer the methylene chloride to the separatory funnel and extract the sample by vigorously shaking the funnel for 2 minutes, with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase. If the emulsion interface between the layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration through glass wool, centrifugation, or other physical methods. Discard the methylene chloride phase.

14.2.3.3 Add a second 60 ml volume of methylene chloride to the separatory funnel and repeat the extraction procedure a second time, discarding the methylene chloride layer. Perform a third extraction in the same manner.

14.2.4 Add 6 ml of cold (4C) 12 N sulfuric acid to the sample (or hydrolyzed sample), seal, and shake to mix. Check the pH of the sample with pH paper. If the sample does not have a pH less than or equal to 2, adjust the pH by adding more acid.

14.2.5 Add 120 ml diethyl ether to the sample, seal, and extract the sample by vigorously shaking the funnel for 2 min with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase. If the emulsion interface between layers is more than one third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum techniques to complete the phase separation depends upon the sample, but may include stirring, filtration through glass wool, centrifugation, or other physical methods. Remove the aqueous phase to its original bottle, and collect the ether phase in a 500-ml Erlenmeyer.

14.2.6 Return the aqueous phase to the separatory funnel, add 60 ml of diethyl ether to the sample, and repeat the extraction procedure a second time, combining the extracts in the 500 ml Erlenmeyer flask. Perform a third extraction with 60 ml diethyl ether in the same manner. Add approximately 40 grams of acidified sodium sulfate and allow the extracts to remain in contact with the sodium sulfate for about 2 hours (preferably overnight). At room temperature.

NOTE: The drying step is very critical to ensuring complete esterification. Any moisture remaining in the ether will result in low herbicide recoveries. The amount of sodium sulfate is adequate if some free flowing crystals are visible when swirling the flask. If all of the sodium sulfate solidifies in a cake, add a few additional grams of acidified sodium sulfate and again test by swirling. The 2 hour drying time is a minimum, however, the extracts may be held in contact with the sodium sulfate overnight.

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14.3 Extract Concentration

14.3.1 Pour the dried extract through a funnel plugged with acid washed glass wool and collect the extract in the K-D concentrator. Use a glass rod to crush any caked sodium sulfate during the transfer. Rinse the flask with 20 to 30 ml of diethyl ether to complete the quantitative transfer.

14.3.2 Add one or two clean boiling chips to the flask and attach a three-ball Snyder column. Pre-wet the Snyder column by adding about 1 ml of diethyl ether to the top of the column. Place the K-D apparatus on a hot water bath (15-20C above the boiling point of the solvent) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration in 10-20 minutes. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reaches 1 ml, remove the K-D apparatus from the water bath and allow to cool for at least 10 minutes.

14.3.3 Remove the Snyder column and rinse the flask and its lower joints into the concentrator tube with 1-2 ml of diethyl ether. The extract may be further concentrated by using the nitrogen blowdown technique.

14.3.3.1 For soil samples, exchange solvent with two 50 ml portions of ether – each time bringing the volume below 10 ml. Then to a final volume of 1 ml.

14.3.3.2 For aqueous samples, concentrate to 1 ml.

14.3.4 Nitrogen Blowdown

14.3.4.1 Place the concentrator tube in a warm water bath (approximately 35C and evaporate the solvent volume to the required level using a gentle stream of clean, dry nitrogen (filtered through a column of activated carbon).

CAUTION: Do not use plasticized tubing between the carbon trap and the sample.

14.3.4.2 The internal wall of the tube must be rinsed down several times with diethyl ether during the operation. During evaporation, the solvent level in the tube must be positioned to prevent water from condensing into the sample (i.e., the solvent level should be below the level of the water bath). Under normal operating conditions, the extract should not be allowed to become dry.

14.3.5 Dilute the extract with 1 ml of isooctane and 0.5 ml of methanol. Dilute to a final volume of 4 ml with diethyl ether. The sample is now ready for methylation with diazomethane.

14.4 Esterification

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14.4.1 Diazomethane derivatization - Two methods may be used for the generation of diazomethane: the bubbler method and the Diazald kit method.

CAUTION: Diazomethane is a carcinogen and can explode under certain conditions.

The bubbler method is suggested when small batches of samples (10-15) require esterification. The bubbler method works well with samples that have low concentrations of herbicides (e.g., aqueous samples) and is safer to use than the Diazald kit procedure. The Diazald kit method is good for large quantities of samples needing esterification. The Diazald kit method is more effective than the bubbler method for soils or samples that may contain high concentrations of herbicides (e.g., samples such as soils that may result in yellow extracts following hydrolysis may be difficult to handle by the bubbler method).

The diazomethane derivatization procedures described below will react efficiently with all of the chlorinated herbicides described in this method and should be used only by experienced analysts, due to the potential hazards associated with its use.

The following precautions should be taken:

- Use a safety screen.
- Use mechanical pipetting aides.
- Do not heat above 90C - EXPLOSION may result.
- Avoid grinding surfaces, ground-glass joints, sleeve bearings, and glass stirrers - EXPLOSION may result.
- Store away from alkali metals - EXPLOSION may result.
- Solutions of diazomethane decompose rapidly in the presence of solid materials such as copper powder, calcium chloride, and boiling chips.

14.4.1.1 Bubbler Method - assemble the diazomethane bubbler

14.4.1.1.1 Add 5 ml of diethyl ether to the first test tube. Add 1 ml of diethyl ether, 3 ml (3 scoops) of carbitol, 3 ml of 37% KOH and 0.1-0.2 g of Diazald to the second test tube. Immediately place the exit tube into the concentrator tube containing the sample extract. Apply nitrogen flow (10 ml/min) to bubble diazomethane through the extract for 10 minutes or until the yellow color of diazomethane persists. The amount of diazald used is sufficient for esterification of approximately three sample extracts. An additional 0.1-0.2 g of Diazald may be added (after the initial Diazald is consumed) to extend the generation of the diazomethane. There is sufficient KOH present in the original solution to perform a maximum of approximately 20 minutes of total esterification.

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14.4.1.1.2 Remove the concentrator tube and seal it with a Neoprene or PTFE stopper. Store at room temperature in a hood for 20 minutes.

14.4.1.1.3 Destroy any unreacted diazomethane by adding 0.1-0.2 g of silicic acid to the concentrator tube. Allow to stand until the evolution of nitrogen gas has stopped. Vortex to mix well. Adjust the sample volume to 10.0 ml with hexane. Transfer to a vial and store refrigerated if further processing will not be performed immediately. Analyze by gas chromatography.

14.4.1.1.4 Extracts should be stored at 4°C away from light. Preservation study results indicate that most analytes are stable for 28 days; however, it is recommended that the methylated extracts be analyzed immediately to minimize the trans-esterification and other potential reactions that may occur.

14.5 Calibrate the GC system according to section 13 of this SOP. The same GC operating conditions used for the initial calibration must be employed for sample analyses.

14.6 Verify calibration each 12-hour shift, and after ten analyses (|AFCEE every ten samples) and at the end of the analytical sequence by injecting calibration verification standards prior to conducting any sample analyses. Analysts should alternate the use of high and low concentration mixtures of single and multi-component analytes for calibration verification. A calibration standard must also be injected at intervals of not less than once every ten samples and at the end of the analysis sequence.

14.6.1 The response for each analyte should not exceed a +/- 15% difference from that calculated for the initial calibration.

14.6.2 If this criterion is exceeded for any analyte, calculate the average percent difference across all analytes. If the average of the responses for all analytes is within 15%, then the calibration has been verified. The average must include all analytes in the calibration, regardless of whether they are target analytes for a specific project, and the data user must be provided with the calibration verification data or a list of those analytes that exceeded the +/- 15% limit.

14.7 If the calibration does not meet the +/- 15% limits (either on the basis of each compound or the average across all compounds), check the instrument operating conditions, and if necessary, restore them to the original settings and inject another aliquot of the calibration verification standard. If the response for the analyte is still not within +/- 15%, then a new initial calibration must be prepared.

14.8 Set the center of the retention time windows to the retention time of the calibration verification standard analyzed at the beginning of the 12-hour shift.

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14.9 Inject an aliquot of the concentrated sample aliquot. The injection volume should be the same as that used for the initial and continuing calibration standards.

14.10 Each sample analysis must be bracketed with an acceptable initial calibration, calibration verification standard(s) (each 12-hour analytical shift), or calibration standards interspersed within the samples. Although the analysis of a single mid-concentration standard (standard mixture or multi-component analyte) will satisfy the minimum requirements, analysts are urged to use different calibration verification standards during chlorinated herbicide analyses. The results from these bracketing standards must meet the calibration verification criteria in Sec. 12.2.1. When a calibration verification standard fails to meet the QC criteria, all samples that were injected after the last standard that last met the QC criteria must be evaluated to prevent mis-quantitations and possible false negative results, and re-injection of the sample extracts may be required. More frequent analyses of standards will minimize the number of sample extracts that would have to be re-injected if the QC limits are violated for the standard analysis. However, if the standard analyzed after a group of samples exhibits a response for an analyte that is above the acceptance limit, and the analyte was not detected in the specific samples analyzed during the analytical shift, then the extracts for those samples do not need to be re-analyzed, as the verification standard has demonstrated that the analyte would have been detected were it present. In contrast, if an analyte above QC limits was detected in a sample extract, then re-injection is necessary to ensure accurate quantitation. If an analyte was not detected in the sample and the standard response is more than 15% below the initial calibration response, then re-injection is necessary to ensure that the detector response has not deteriorated to the point that the analyte would not have been detected even though it was present.

14.11 Sample injections may continue for as long as the calibration verification standards and standards interspersed with the samples meet instrument QC requirements. It recommended that standards be analyzed after every 10 samples (required after every 20 samples and at the end of a set) to minimize the number of samples that must be re-injected when the standards fail the QC limits. The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria are exceeded.

14.12 If the peak response is less than 2.5 times the baseline noise level, the validity of the quantitative result may be questionable. The analyst should consult with the source of the sample to determine whether further concentration of the sample is warranted.

14.13 When sample concentrations exceed the upper limit of the calibration curve, samples shall be diluted and re-analyzed (if possible) to bring them within the calibration curve.

15.0 Data Analysis and Calculations:

15.1 Tentative identification of an analyte occurs when a peak from a sample extract falls within the absolute retention time window. Each

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tentative identification must be confirmed using either a second GC column of dissimilar phase or using another technique such as GC/MS. When results are confirmed using a second GC column of dissimilar phase, the analyst should check the agreement between the quantitative results on both columns once the identification has been confirmed. Calculate the relative percent difference (RPD) between the two results using the formula below.

$$RPD = \frac{|R1 - R2|}{\frac{(R1 + R2)}{2}} \times 100$$

where R1 and R2 are the results on the two columns and the vertical bars in the equation above indicate the absolute value of the difference.

15.1.1 If one result is significantly higher (e.g. >40%), check the chromatograms to see if an obviously overlapping peak is causing an erroneously high result. If no overlapping peaks are noted, examine the baseline parameters established by the instrument data system (or operator) during peak integration.

15.1.2 If no anomalies are noted, review the chromatographic conditions. If there is no evidence of chromatographic problems, report the higher result. This approach is conservative relative to protection of the environment. The data user should be advised of the disparity between the results on the two columns.

15.2 Determine the quantity of each component peak in the sample chromatogram which corresponds to the compounds used for calibration purposes according the following equations:

15.2.1 For aqueous samples

$$\text{Concentration (ug/L)} = \frac{(A)(V_t)(D)}{(V_i)(V_s)}$$

where:

A = ng of analyte detected in the sample (as determined by the initial calibration curve)

V_t = Total volume of the concentration extract (ul)

D = Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution was made, D = 1. The dilution factor is always dimensionless.

V_i = Volume of extract injected (ul). The injection volume for Samples and calibration standards must be the same.

V_s = Volume of the aqueous sample extracted in mL. If units of liters are used for this term, multiply the results by 1000

Using the units specified here for these terms will result in a

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Concentration in units of ng/ml, which is equivalent to ug/L.

15.2.2 For solid samples

$$\text{Concentration (ug/Kg)} = \frac{(A)(V_t)(D)}{(V_i)(V_s)(Ts)}$$

where:

A = ng of analyte detected in the sample (as determined by the initial calibration curve)

V_t = Total volume of the concentration extract (ul)

D = Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution was made, D = 1. The dilution factor is always dimensionless.

V_i = Volume of extract injected (ul). The injection volume for Samples and calibration standards must be the same.

V_s = Weight of the aqueous sample extracted in mL. If units of Kilograms are used for this term, multiply the results by 1000

Ts = total solids

Using the units specified here for these terms will result in a Concentration in units of ng/ml, which is equivalent to ug/Kg.

15.2.3 If responses exceed the calibration range of the system, dilute the extract and re-analyze. Peak height measurements are recommended over peak area integration when overlapping peaks cause errors in area integration.

15.3 GC/MS confirmation may be used in conjunction with either single-column or dual-column analysis if the concentration is sufficient for detection by GC/MS.

15.3.1 Full-scan GC/MS will normally require a concentration of approximately 10 ng/ul in the final extract for each single-component compound. Ion trap or selected ion monitoring will normally require a concentration of approximately 1 ng/ul.

15.3.2 The GC/MS must be calibrated for the specific target herbicides when it is used for quantitative analysis.

15.3.3 GC/MS may not be used for confirmation when concentrations are below 1 ng/ul in the extract.

15.3.4 GC/MS confirmation should be accomplished by analyzing the same extract that is used for GC/ECD analysis and the extract of the associated method blank.

15.3.5 The base/neutral/acid extract and the associated blank may be used for GC/MS confirmation if the surrogates and internal standards do not interfere and if it is demonstrated that the analyte is stable during

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acid/base partitioning. However, if the compounds are not detected in the base/neutral/acid extract, the GC/MS analysis of the herbicide extract should be performed.

15.3.6 A QC reference sample containing the compound must also be analyzed by GC/MS. The concentration of the QC reference sample must demonstrate that those herbicides identified by GC/ECD can be confirmed by GC/MS.

16.0 Method Performance:

16.1 The matrix specific method detection limits (MDLs) should be determined using the following equation:

With this data, method detection limits were calculate using the formula (3):

$$MDL = S \ t(n-1, 1\text{-alpha} = 0.99)$$

Where:

t(n-1, 1-alpha = 0.99) = Student's t value for the 99% confidence level with n-1 degrees of freedom

n = number of replicates

S = the standard deviation of the replicate analysis

16.2 The accuracy and precision obtainable with this method depend on the sample matrix, sample preparation technique, optional cleanup techniques, and the calibration procedures used. Table 3 contains criteria utilized by the laboratory for determining acceptability of surrogate recoveries, laboratory control spike recoveries, matrix spike/matrix spike duplicates and sample duplicates. The laboratory should determine statistical limits when sufficient data points become available. Per client request, the laboratory may also utilize project specific acceptance criteria.

17.0 Pollution Prevention:

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. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the EPA recommends recycling as the next best option.

18.0 Data Assessment, Acceptance and Corrective Action:

18.1 Sample data is initially reviewed by the analyst performing the analysis.

18.2 Secondary (or peer) review is required for all data before reporting to the client.

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18.3 Suggested chromatographic system maintenance- When system performance does not meet the established QC requirements, corrective action is required, and may include one or more of the following.

18.3.1 Splitter connections- For dual columns which are connected using a press-fit Y-shaped glass splitter or a Y-shaped fused-silica connector, clean and deactivate the splitter port insert or replace with a cleaned and deactivated splitter. Break off the first few centimeters of the injection port side of the column. Remove the columns and solvent backflush according to the Manufacturer's instructions. If these procedures fail to eliminate the degradation problem, it may be necessary to deactivate the metal injector body and/or replace the columns.

18.3.2 Metal injector body- Turn off the oven and remove the analytical columns when the oven has cooled. Remove the glass injection port insert. Lower the injection port temperature to room temperature. Inspect the injection port and remove any noticeable foreign material.

18.3.2.1 Place a beaker beneath the injector port inside the oven. Using a wash bottle, serially rinse the entire inside of the injector port with acetone and then hexane, catching the rinsate in the beaker.

18.3.2.2 Prepare a solution of deactivating agent (Sylon-CT or equivalent) following manufacturer's directions. After all metal surfaces inside the injector body have been thoroughly coated with the deactivation solution, rinse the injector body with toluene, methanol, acetone, then hexane. Reassemble the injector and replace the columns.

18.3.3 Column Rinsing- The column should be rinsed with several volumes of an appropriate solvent. Both polar and non-polar solvents are recommended. Depending on the nature of the sample residues expected, the first rinse might be water, followed by methanol and acetone. Methylene Chloride is a good final rinse and in some cases may be the only solvent required. The column should then be filled with methylene chloride and allowed to stand flooded overnight to allow materials within the stationary phase to migrate into the solvent. The column is then flushed with fresh methylene chloride, drained, and dried at room temperature with a stream of ultrapure nitrogen.

18.3.4 Autosampler syringes should be inspected daily and replaced as needed.

18.3.5 ECD detectors may become contaminated during use. Detectors may be baked overnight with a steady stream of hydrogen. If the signals do not return to useable levels, the detectors should be sent back to the manufacturer for replacement. ECD detectors (inlet, housing and exit) should be wiped every six months to ensure that radiation is not leaking. Records of wipe tests are maintained by the radiation safety officer.

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18.3.6 Autosampler rinse and Waste vials should be inspected on a daily basis and filled or emptied as needed.

19.0 Waste Management:

Any and all waste generated, through any procedure in the laboratory, is handled according to the Chemical Hygiene, Hazardous Waste Management and Spill Prevention Control and Contingency Plans of Northeast Laboratory Services, Inc.

20.0 References:

SW-846 Method 8151A, Revision 1 December 1996
SW-846 Method 8000B, Revision 2 December 1996
SW-846 Chapter 4 Organic Analytes, Revision 3 December 1996
NELAC Quality System Chapter 5, June 5, 2003

21.0 Tables, Diagrams, Flowcharts and Validation Data

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Table 1 Composite Standard Solutions

Mfgr.	Cat #	Mix Name	Concentration	Components
Restek	32054	Herbicide Mix #1 Free Acid Form	200 ug/ml each in methanol	2,4-D 2,4-DB 2,4,5-T 2,4,5-TP Dicamba Dichlorprop Dinoseb
	32055	Herbicide Mix #1 Derivatized Form	200 ug/ml each in hexane	2,4-D methyl ester 2,4-DB methyl ester 2,4,5-T methyl ester 2,4,5-TP methyl ester Dicamba methyl ester Dichlorprop methyl ester Dinoseb methyl ester
Restek	32056	Herbicide Mix #2 Free Acid Form	2000 ug/ml in methanol	Dalapon
	32057	Herbicide Mix #2 Derivatized Form	2000 ug/ml in hexane	Dalapon methyl ester
Restek	32058	Herbicide Mix #3 Free Acid Form	20,000 ug/ml each in methanol	MCPA MCPP
	32059	Herbicide Mix #3 Derivatized Form	20,000 ug/ml each in hexane	MCPA methyl ester MCPP methyl ester
Restek	32061	Herbicide Mix #4 Free Acid Form	200 ug/ml each in methanol	Acifluorfen Bentazon Chloramben DCPA diacid 3,5-Dichlorobenzoic Acid 4-Nitrophenol Pentachlorophenol Picloram
	32062	Herbicide Mix #4 Derivatized Form	200 ug/ml each in hexane	Acifluorfen methyl ester Bentazon methyl ester Chloramben methyl ester 3,5-Dichlorobenzoic acid methyl ester 4-Nitroanisole pentachloroanisole Picloram methyl ester
Restek	32049	Herbicide Surrogate Mix Free Acid Form	200 ug/ml in methanol	DCAA
	32050	Herbicide Surrogate Derivatized Form	200 ug/ml in hexane	DCAA methyl ester

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Table 2. Calibration Levels

Component	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
2,4-D	0.05	0.1	0.5	0.75	1.0	2.0
2,4-DB	0.05	0.1	0.5	0.75	1.0	2.0
2,4,5-T	0.05	0.1	0.5	0.75	1.0	2.0
2,4,5-TP	0.05	0.1	0.5	0.75	1.0	2.0
Dicamba	0.05	0.1	0.5	0.75	1.0	2.0
Dichlorprop	0.05	0.1	0.5	0.75	1.0	2.0
Dinoseb	0.05	0.1	0.5	0.75	1.0	2.0
Dalapon	0.5	1.0	5.0	7.5	10	20
MCPA	5.0	10	50	75	100	200
MCPP	5.0	10	50	75	100	200
Acifluorfen	0.05	0.1	0.5	0.75	1.0	2.0
Bentazon	0.05	0.1	0.5	0.75	1.0	2.0
Chloramben	0.05	0.1	0.5	0.75	1.0	2.0
DCPA	0.05	0.1	0.5	0.75	1.0	2.0
3,5-Dichlorobenzoic Acid	0.05	0.1	0.5	0.75	1.0	2.0
4-Nitroanisole	0.05	0.1	0.5	0.75	1.0	2.0
Pentachlorophenol	0.05	0.1	0.5	0.75	1.0	2.0
Picloram	0.05	0.1	0.5	0.75	1.0	2.0
DCAA (surrogate)	0.25	0.5	2.5	3.75	5	10

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Table 3 - Fixed Laboratory Analytical QC Sample Table
Groundwater, Surface Water, Solids
Herbicides

Medium/Matrix	Aqueous and Solid
Sampling SOP	NA
Analytical Parameter	Herbicides
Concentration Level	Aqueous-Low/Medium (1 – 5 ug/L) Solid (100 ug/Kg)
Analytical Method/SOP Reference	SW846 8151
Laboratory Name	NEL
No. of Sample Locations	

Laboratory QC:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	1 per 20 samples or daily whenever samples are extracted by the same procedure	(1) Less than or equal to ½ the PQL for all target analytes	(1) Investigate source of contamination (2) Evaluate the samples and the associated QC: i.e. If the blank results are above the QL, report sample results which are <QL or >10X the blank concentration for that analyte. If sample >QL and <10X the blank concentration for that analyte, reprep if less than 2X hold-time	Analyst/ Supervisor	Accuracy/ bias-Contamination	Less than or equal to ½ the PQL for all other target compounds
Reagent Blank	NA	NA	NA	NA	NA	NA
Storage Blank	NA	NA	NA	NA	NA	NA
Instrument Blank	NA	NA	NA	NA	NA	NA
Laboratory Duplicate	NA	NA	NA	NA	NA	NA
Laboratory Matrix Spike	One for each group of 20 samples of a similar matrix or concentration	2,4,5-TP: 20%-160% Dinoseb: 20%-160% Dicamba: 20%-160% 2,4,5-T: 20%-160% 2,4-D: 20%-160% Dichlorprop: 20%-160% 2,4-DB: 20%-160%	(1) No CA will be taken for samples where recoveries are outside limits and surrogate and LCS criteria are met. (2) Where LCS is low outside criteria , no MS CA will be taken if all samples in the batch are >25 ug/l. (3) Where LCS is low outside criteria, a LCS,MS (sample <25ug/l), and MSD may be re-extracted if Project Completeness< Completeness Criteria	Analyst/ Supervisor	Accuracy/bias	2,4,5-TP: 20%-160% Dinoseb: 20%-160% Dicamba: 20%-160% 2,4,5-T: 20%-160% 2,4-D: 20%-160% Dichlorprop: 20%-160% 2,4-DB: 20%-160%
Matrix Spike Duplicate	One for each group of 20 samples of a similar matrix or	2,4,5-TP: 20%-160% Dinoseb: 20%-160% Dicamba: 20%-160% 2,4,5-T: 20%-160%	(4) In the event that the MS or MSD surrogates are outside criteria and the the sample <25 ug/l the MS/MSD will be reextracted and reanalyzed if less than	Analyst/ Supervisor	Accuracy/bias	2,4,5-TP: 20%-160% Dinoseb: 20%-160% Dicamba: 20%-160% 2,4,5-T: 20%-160%

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Table 3 - Fixed Laboratory Analytical QC Sample Table
Groundwater, Surface Water, Solids
Herbicides

Medium/Matrix	Aqueous and Solid
Sampling SOP	NA
Analytical Parameter	Herbicides
Concentration Level	Aqueous-Low/Medium (1 – 5 ug/L) Solid (100 ug/Kg)
Analytical Method/SOP Reference	SW846 8151
Laboratory Name	NEL
No. of Sample Locations	

Laboratory QC:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria
	concentration. In the event insufficient volume is available, the lab will perform LCS/LCSD analyses	2,4-D: 20%-160% Dichlorprop: 20%-160% 2,4-DB: 20%-160%	2X hold-time. (5) No CA will be taken when both the MS and MSD surrogates are in criteria.			2,4-D: 20%-160% Dichlorprop: 20%-160% 2,4-DB: 20%-160%
LCS	Every 20 samples of a similar matrix or concentration or every batch of samples extracted	2,4,5-TP: 20%-160% Dinoseb: 20%-160% Dicamba: 20%-160% 2,4,5-T: 20%-160% 2,4-D: 20%-160% Dichlorprop: 20%-160% 2,4-DB: 20%-160%	(1) If an MS/MSD was performed and acceptable narrate. (1) If the surrogate recoveries in the LCS are also low but are acceptable in the blank and samples narrate. (2) If the LCS recovery is high but the sample results are <QL, narrate. Otherwise reextract affected sample batch if less than 2X Hold-time.	Analyst/ Supervisor	Accuracy/bias	2,4,5-TP: 20%-160% Dinoseb: 20%-160% Dicamba: 20%-160% 2,4,5-T: 20%-160% 2,4-D: 20%-160% Dichlorprop: 20%-160% 2,4-DB: 20%-160%
LFB	NA	NA	NA	NA	NA	NA
Surrogates	Every sample, blank, LCS, MS/MSD	All surrogates must meet the following acceptance limits: DCAA: 20%-160%	(1) If the surrogates are outside high and sample is <QL no CA taken. (2) If surrogates are low outside limits, the affected sample is reextracted and reanalyzed to confirm matrix interference.	Analyst/ Supervisor	Accuracy/bias	All surrogates must meet the following acceptance limits: DCAA: 20%-160%
Internal Standards (iSs)	NA	NA	NA	NA	NA	NA
Initial Calibration	Before sample analysis	Minimum of 5 calibration levels per analyte of interest. Correlation coefficient of 0.99 or higher for linear calibration. Non-linear calibration requires 6 standards.	Investigate source of problem. Re-analyze calibration curve making new standards or performing instrument maintenance as needed	Analyst/ Supervisor	Accuracy/bias	Correlation coeffiecient greater than or equal to 0.99

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Table 3 - Fixed Laboratory Analytical QC Sample Table
Groundwater, Surface Water, Solids
Herbicides

Medium/Matrix	Aqueous and Solid
Sampling SOP	NA
Analytical Parameter	Herbicides
Concentration Level	Aqueous-Low/Medium (1 – 5 ug/L) Solid (100 ug/Kg)
Analytical Method/SOP Reference	SW846 8151
Laboratory Name	NEL
No. of Sample Locations	

Laboratory QC:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action (CA)**	**Person(s) Responsible for CA**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Continuing Calibration standard	Daily before sample analysis and after 20 samples (10 samples for DOD) or at the beginning and end of each 12 hour clock	+/- 15% D	(1) Re-analyze standard. (2) Investigate and correct source of problem. (3) Re-analyze all samples analyzed after acceptable standard	Analyst/ Supervisor	Accuracy/bias	+/- 15% D
Other:						

GW = groundwater
SW = surface water
SL = solid

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DOCUMENT HISTORY AND APPROVAL PAGE

TITLE:	1,4-Dioxane By Gas Chromatography/Mass Spectroscopy (GC/MS)-Modified EPA Method 8260B
DEPARTMENTS AFFECTED:	Organics

ISSUE DATE:	REVISION CODE:	SOP #:	REVISION CHANGES:
10/25/05	-	3.3.50	Creation of SOP to be compliant with NELAC standards
6/19/07	A	3.3.50	Annual Review & Update
01/23/08	B	3.3.50	Annual Review & Update

DOCUMENT APPROVAL		
Originator: Abram Patenaude	Signature/Date: 012408	Title Organics Analyst
Approved by: Jim Galasyn	012308	Chemistry Lab Manager
Reviewed by: Pamela Doughty	1/23/08	Total Quality Manager

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NEL STANDARD OPERATING PROCEDURE			
TITLE **1,4-Dioxane By Gas Chromatography/Mass Spectroscopy (GC/MS)-Modified EPA Method 8260B**			
SOP# 3.3.50	REV# B	EFFECTIVE 01/23/08	PAGE 2 OF 18
WRITTEN BY: Abram Patenaude	DEPARTMENTS AFFECTED: ORGANICS		

1.0 Test Method:

This standard operating procedure (SOP) was developed to maintain high standards of analytical performance in order to generate data of known and documented quality for our clients. The test method is EPA Method 8260, modified to test for 1,4-dioxane only. This SOP is based on EIASOP-VOADIOX1 (The Office of Environmental Measurement and Evaluation, EPA Region NE, 11 Technology Drive, North Chelmsford, MA 01863), 05/21/03.

2.0 Applicable Matrix or Matrices:

This method is applicable to a variety of solid waste matrices. NEL uses this method for ground & surface waters, sludge, sediment, soils, and solid and waste oil samples.

3.0 Detection Limit:

Please see Table 1 for Standard Compound List and Reporting Limits for this method. Other analytes can be added by client request upon completion of a method detection limit study.

4.0 Scope and Application

This SOP is based on Method 8260B (USEPA Test Methods for Evaluating Solid Waste, Dec. 1996) and on EIASOP-VOADIOX1 (The Office of Environmental Measurement and Evaluation, EPA Region NE, 11 Technology Drive, North Chelmsford, MA 01863), 05/21/03.

Method 8260B describes the GC/MS procedure for the analysis of volatile organic compounds. The techniques by which compounds may be introduced into the GC/MS system are described in Method 5030 (purge-and-trap of aqueous samples) and Method 5035 (purge-and-trap of solid and waste oil samples). This method is applicable to nearly all types of samples regardless of water content, including ground and surface water, waste solvents, oily wastes, soils and sediments. A gas chromatograph utilizes a temperature program to separate the purgeables before detection on a mass spectrometer.

5.0 Summary of Method

5.1 Basis Principles - The analytes are introduced into the GC/MS by purge-and-trap techniques (Method 5030 or Method 5035). The high solubility of 1,4-dioxane in water causes a low purge efficiency. Heating the sample to 80 degrees C prior to purging increases the purge efficiency. Upon desorption the volatile compounds are introduced

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directly to the capillary column of a gas chromatograph equipped with a temperature program to separate the purgeables. The eluted analytes on the gas stream into the ion source of a mass spectrometer. The ionized molecules are focused and separated according to their mass/charge (m/z) ratio by the quadrupole analyzer. The signal is amplified by an electron multiplier and interpreted by the mass spectrometer data system to produce a total ion chromatogram with mass spectra for every data point on the chromatogram. The system is an HP 5890 Series II Gas Chromatograph with a 5970 Mass Spectrophotometer or equivalent.

5.2 General Method - The mass spectrometer is calibrated to recognize m/z values in the range of 35-550 amu. Reference spectra and retention times for analytes are obtained by the measurement of calibration standards under the same conditions used for samples. Analytes are quantitated based upon a minimum of a five part calibration. Hewlett-Packard Enviroquant ChemStation Ver. A.03.00 software (or equivalent) is utilized for identification and quantitation of data. The concentration of each identified component is measured by the relative response of the target compound to its respective internal standard.

5.3 The performance of the mass spectrometer is verified by the introduction of a 50ng 4-Bromofluorobenzene (BFB) tune standard. All analyses must occur within 12 hours of the passing BFB analysis time.

5.4 Before any samples can be analyzed a continuing calibration verification (CCV) standard must be analyzed to demonstrate that the instrument is stable relative to the initial calibration curve.

6.0 Definitions

6.1 INTERNAL STANDARD (IS) - Non-target analyte compounds that are similar to the target analytes but are not expected to be found in environmental media (generally, isotopically labeled target analytes are used for this purpose). Internal standards are added to every standard, quality control sample, and field sample at a known concentration prior to analysis. Internal standard responses are used as the basis for quantitation of target analytes.

6.2 SURROGATE ANALYTE (SS) - Non-target analytes compounds that are similar in composition and behavior to the target analytes but are not expected to be found in environmental media (often, isotopically labeled target analytes are used for this purpose) and are added to every standard, quality control sample, and field sample at a known concentration prior to preparation and/or analysis. Surrogate responses are used to evaluate the accuracy of the laboratory's performance of the analytical method in a specific sample matrix.

6.3 STOCK STANDARD SOLUTION - A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.

6.4 PRIMARY DILUTION STANDARD SOLUTION - A solution of several analytes prepared in the laboratory from stock standard solutions and diluted as needed to prepare calibration solutions and other needed analyte solutions.

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6.5 CALIBRATION STANDARD (CAL) - A solution prepared from the primary dilution standard solution or stock standard solutions and the internal standards and surrogate analytes. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

6.6 4-BROMOFLUOROBENZENE STANDARD (BFB)– A standard solution containing BFB in methanol and used to evaluate the performance of the MS system. The introduction and evaluation of a 50ng BFB standard by the MS system must meet the criteria in Table 2.

6.7 CONTINUING CALIBRATION VERIFICATION STANDARD (CCV) - An analytical standard containing all target analytes, surrogate and internal standard compounds that is used to evaluate the performance of the instrument system with respect to a defined set of method criteria.

6.8 METHOD BLANK (BLK, similarly known as the LABORATORY REAGENT BLANK) - An aliquot of organic free reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The BLK is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

6.9 TRIP BLANK - An aliquot of reagent water or other blank matrix that is placed in a sample container in the laboratory and treated as a sample in all respects, including shipment to the sampling site, exposure to sampling site conditions, storage, preservation, and all analytical procedures. The purpose of the trip blank is to determine if method analytes or other interferences are present in the field environment.

6.10 LABORATORY CONTROL SAMPLE (LCS) - The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. When the results of the matrix spike analysis indicates a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

6.11 MATRIX SPIKE/ MATRIX SPIKE DUPLICATE (MS/MSD) - An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS/MSD is analyzed exactly like a sample. Its purpose is used to evaluate the accuracy and precision of the laboratory performance of the analytical method in a specific sample matrix.

6.12 REAGENT WATER – Organic free water. NEL uses carbon filtration of water from its Milli-Q deionized water system, or equivalent.

7.0 Interferences

7.1 During analysis, major contaminant sources are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap. The use of Teflon tubing, Teflon thread sealants, or flow controllers and rubber components in the purging device should be avoided since such materials out-gas organic compounds which will

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be concentrated in the trap during the purge operation. Analyses of laboratory reagent blanks provide information about the presence of contaminants. Subtracting blank values from sample results is not permitted.

7.2 Interfering contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing relatively high concentrations of volatile organic compounds. A preventive technique is between-sample rinsing of the purging apparatus and sample syringes with two to three portions of reagent water. After analysis of a sample containing high concentrations of volatile organic compounds, one or more laboratory reagent blanks should be analyzed to check for cross-contamination. The blank must have a concentration less than one-half of the reporting level in order for analysis to proceed.

7.3 Traces of ketones, methylene chloride, and some other organic solvents can be present even in the highest purity methanol. This is another potential source of contamination, and should be assessed before standards are prepared in the methanol. Methanol is stored in a separate area from the extractions laboratory.

8.0 Safety

8.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials or procedures.

8.2 Material Safety Data Sheets (MSDS) are made available to all personnel involved in the chemical analysis. A written environmental health and safety plan is reviewed by all NEL staff.

8.3 Please note chemicals that have the potential to be highly toxic or hazardous, the appropriate MSDS must be reviewed by the employee before handling the chemical. The following method analytes have been tentatively classified as known or suspected human or mammalian carcinogens: benzene, carbon tetrachloride, 1,4-dichlorobenzene, 1,2-dichloroethane, hexachlorobutadiene, 1,1,2,2-tetrachloroethane, 1,1,2-trichloroethane, chloroform, 1,2-dibromoethane, tetrachloroethene, trichloroethene, and vinyl chloride. Pure standard materials and stock standard solutions of these compounds and all other target compounds will be handled in a hood.

9.0 Equipment and Supplies

9.1 **Containers**

9.1.1 Sample storage containers: 40 ml screw cap vials equipped with a Teflon faced silicone septum, certified clean, known volume of 44 ml (see also Method 5035)

9.1.2 Standard Storage Containers: 1-5 ml Mininert vials with Teflon lined screw caps.

9.2 **Syringes**

9.2.1 5 ml-50 ml gas tight hypodermic syringes with Luer-Lok or fixed needle tips.

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9.2.2 Micro syringe 10 - 1000 µl.

9.3 **Instrumentation**

9.3.1 Autosampler: Archon 51 Position Autosampler or equivalent.

9.3.2 LCS 2000 purge and trap or equivalent.

9.3.3 Trap: Tekmar Purge Trap K or equivalent

9.3.4 Gas Chromatograph: Hewlett-Packard 5890 Series II or equivalent.

9.3.5 Mass Spectrometer: Hewlett-Packard 5970 MSD or equivalent.

9.3.6 Primary Column: Fused silica capillary column, Restek RTX-VMS 30m x 0.25 mm x 1.4 µm or equivalent.

9.4 **Data Acquisition and Processing**

Data System - Hewlett-Packard ChemStation software is used for data acquisition and HP Enviroquant software is used for data processing.

10.0 Reagents and Standards

10.1 **Reagents**

10.1.1 Methanol - Purge and Trap Grade, demonstrated to be free of analytes.

10.1.2 Reagent water – organic free deionized water or equivalent.

10.2 **Stock Standard Solutions** - These solutions are purchased as certified solutions or prepared from pure standard materials. Commercial standards arrive ampulized in concentrations ranging from 1-5 mg/ml. Solutions are stored per manufacturer's recommendations. Once opened, these concentrated solutions expire at the manufacturer's expiration date or one year from opening, whichever is the most conservative.

10.3 **Primary Dilution Standards** - Stock standard solutions are used to prepare primary dilution standard solutions that contain all the analytes of interest in methanol. The primary dilution standards are prepared at concentrations that can be easily diluted to prepare aqueous calibration solutions that will bracket the working concentration range. The primary dilution standard solutions are stored with minimal headspace and checked frequently for signs of deterioration or evaporation. Methanol solutions of gaseous standards are not stable for more than one week at >0°C.

10.4 **Calibration Standards** – Five or more concentrations for calibration in the range 1 to 100 ug/L for 1,4-dioxane. Other concentrations and volumes may be used provided they meet method requirements.

10.5 **Calibration Verification Standard (CCV)** – Standard prepared at a concentration of 5 µg/L for 8260B analysis that is used to evaluate the initial calibration curve.

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10.6 **Method Blank (BLK)** – Sample of organic free reagent water.

10.7 **Laboratory Control Sample (LCS)** -.- Standards prepared at a target concentration of 5.0 ppb.

10.8 **Matrix Spike/Matrix Spike Duplicate (MS/MSD)** - Matrix spikes and matrix spike duplicates are prepared and analyzed with each batch of 20 client specific samples of the same matrix, assuming sufficient sample is provided by the client. Matrix spikes are at a concentration of 5 µg/L. If the client does not specify a sample to be used as a MS/MSD, NEL will analyze a LCS Duplicate (LCSD) to verify precision.

10.9 **Internal Standard and Surrogates Fortification Solutions**- The internal standard is prepared by diluting 12.5 ul of BFB solution (2000 µg/ml in methanol) to 5.0 ml in methanol, for a final concentration of 5.0 ppb. The surrogate standard is prepared by diluting 10 ul of 1,4-dioxane-d8 (neat) to 5.0 ml in methanol. Then 250 ul of this solution is diluted a second time to 5 ml in methanol. This results in a surrogate solution of 100 ppb.

11.0 Sample Collection, Preservation, shipment and storage

11.1 **Sample collection** - Sample collection for aqueous samples is described in Method 5030. Sample collection for solid samples is described in Method 5035.

11.2 **Preservation** - Sample preservation for aqueous samples is described in Method 5030. Sample preservation for solid samples is described in Method 5035. Samples should **not** be acid preserved.

11.3 **Storage** - Samples are analyzed within fourteen days of collection. Other holding times may be selected by the client to conform to local regulatory requirements. Samples are stored at 4°C ± 2°C in a storage area free of organic solvent vapors and direct or intense light. The pH of each aqueous sample is monitored after analysis, with the value recorded in the injection logbook.

12.0 Quality Assurance/Quality Control

12.1 **4-Bromofluorobenzene (BFB)** - Prior to any analyses a 50ng BFB standard is introduced into the GC/MS for 8260B analysis. The BFB spectrum must meet all criteria in Table 2 before analysis of any standards or samples can follow.

12.2 **Method detection limits (MDLs)** are determined annually in accordance with the method described in 40 CFR, Part 136, Appendix B. The results of these studies are kept on file by the QA Manager.

12.3 **Method Blank** – One method blank must be run with every batch of samples after the calibration standards. An acceptable blank must not contain 1,4-dioxane at a concentration greater than the reporting level, 1.0 ug/l. If the method blank exceeds these criteria, the analytical system may be out of control. The source of the contamination must be investigated and appropriate corrective measures must be taken and documented before further sample analysis proceeds

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12.4 **Laboratory Control Sample (LCS)** - The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. When the results of the matrix spike analysis indicates a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

12.5 **Continuing Calibration Verification Standard (CCV)** - A standard sample of target compounds made from the same standard used to prepare the calibration curve. The CCV is run at 5µg/L.

12.6 **Matrix Spike and Matrix Spike Duplicate (MS/MSD)** – The laboratory will analyze a MS/MSD pair when requested by the client. If a client MS/MSD request has not been received every twenty sample analyses, a laboratory control spike duplicate (LCSD) will be analyzed to serve as a measure of the precision of the batch. The MS/MSD will be prepared at a spike concentration of 5 µg/L.

12.7 **Surrogates/Internal Standards** - One compound is used as the surrogate: 1,4-dioxane-d_8. One compound is used as the internal standard: bromofluorobenzene (BFB). Acceptable recovery of the surrogate 1,4-dioxane-d_8 is 60%-140%.

12.8 **Internal Standard Area Response**

12.8.1 The relative retention time (RRT) of the sample analyte must be within ± 30 seconds of the RT of the standard analyte.

12.8.2 If the extended ion current profile (EICP) area in the CCV changes by a factor of two (-50% to +100%) from that in the midpoint lead in the most recent initial calibration curve, corrective action must be taken.

12.8.3 If the EICP area of any internal standard in a QC or field sample changes by more than a factor of two (-50% to +100%) from that in the CCV, reanalysis must occur to confirm matrix interference.

13.0 Calibration and standardization

13.1 **BFB** - The ion abundances shown in Table 2 must be met before analysis of any standards or samples may proceed.

13.2 **Initial Calibration** - The concentrations for the initial calibration are in the range of 1 to 100 ug/L. A minimum of five points are used to calibrate each analyte. A response factor (RF) is calculated for each analyte and/or isomer pair for each calibration solution using the appropriate internal standard. The calculation is performed as follows:

$$RF = \frac{(Ax)\,(Qis)}{(Ais)(Qx)}$$

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Where:
Ax = integrated abundance of the quantitation ion of the analyte
Ais = integrated abundance of the quantitation ion of the internal standard
Qx = quantity of analyte purge in nanograms or concentration units
Qis = quantity of internal standard purged in ng or concentration units

For each analyte and surrogate, calculate the mean response factor from analyses of the calibration solutions. Calculate the standard deviation (SD) and relative standard deviation (RSD) from each mean (M).

$$\%RSD = 100 * \frac{SD}{M}$$

The %RSD of 1,4-dioxane and 1,4-dioxane-d8 must be ≤30%. If this criteria is not met maintenance is performed on the instrument and/or a new calibration curve is acquired.

13.3 **Continuing Calibration Verification (CCV)** – The calibration curve must be verified at the beginning of each run. The concentration of the CCV is 5 µg/L. The RF is calculated for both 1,4-dioxane and 1,4-dioxane-d8 The percent difference is calculated using the following equation:

$$\% \ Difference = \frac{RFv - RF}{RF} \quad (100)$$

Where:
RFv = Response Factor from the analyses of the verification standard
RF = Mean Response Factor from the initial calibration

The percent difference for both analytes must be ≤ 30%. In addition, the internal standard retention time should not change by more than 30 seconds from the mid-point standard level of the most recent initial curve sequence. The integrated areas of the internal standards in the calibration verification standard should not change by more than a factor of two (-50% to +100%) from that in the mid-point standard level of the most recent initial calibration sequence. If the above criteria are not met, maintenance is performed on the instrument and/or a new calibration curve is acquired.

14.0 Procedure

14.1 **Sample Introduction and Purging** - See preparation and introduction Methods 5030 and 5035. Again, all calibration standards, field samples, and QC samples must be heated to 80 degrees C prior to initiation of purging.

14.1.1 See Tables 8 and 9 for preparation of daily check standards and calibration curves.

14.2 **Gas Chromatography/Mass Spectrometry** - Data is acquired and stored over the nominal mass range 35-550 with a total cycle time (including scan overhead time) of two seconds or less. The cycle time is adjusted to measure five or more spectra during the elution of each GC peak. A multi-stage temperature ramp is used to separate the components of interest for this analysis. A typical GC temperature program is described below.

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Initial temperature 40°C, initial time 7 min.
Ramp1: 9°C/min. to 50°C, hold for 0 min.
Ramp2: 27°C/min. to 110°C, hold for 1 min.
Ramp3: 40°C/min. to 220°C, hold for 2.5 min.

Instrument control and acquisition parameters are defined on the ChemStation software for each instrument.

14.3 **Identification of Analytes** - A sample is identified by comparison of its mass spectrum (after background subtraction) to a reference spectrum in the user-created data base. The GC retention time for each analyte should be with ± 30 seconds of the midpoint standard's retention time in the initial calibration curve.

14.3.1 In general, all ions that are present above 10% relative abundance in the mass spectrum of the standard should be present in the mass spectrum of the sample component and should agree within absolute 20%. For example, if an ion has a relative abundance of 30% in the standard spectrum, its abundance in the sample spectrum should be in the range of 10-50%. Some ions, particularly the molecular ion, are of special importance, and should be evaluated even if they are below 10% relative abundance.

14.3.2 Identification requires expert judgment when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When GC peaks obviously represent more than one sample component (i.e., broadened peak with shoulder(s) or valley between two or more maxima), appropriate analyte spectra and background spectra can be selected by examining plots of characteristic ions for tentatively identified components. When analytes coelute (i.e., only one GC peak is apparent), the identification criteria can be met but each analyte spectrum will contain extraneous ions contributed by the coeluting compound. Because purgeable organic compounds are relatively small molecules and produce comparatively simple mass spectra, this is not a significant problem for most method analytes.

14.3.3 Structural isomers that produce very similar mass spectra can be explicitly identified only if they have sufficiently different GC retention times. Acceptable resolution is achieved if the height of the valley between two peaks is less than 25% of the average height of the two peaks. Otherwise, structural isomers are identified as isomeric pairs. Two of the three isomeric xylenes are examples of structural isomers that are not resolved on the capillary column. These groups of isomers will be reported as isomeric pairs.

14.3.4 Methylene chloride, acetone, carbon disulfide, and other background components appear in variable quantities in laboratory and field reagent blanks and generally cannot be accurately measured. Subtraction of the concentration in the blank from the concentration in the sample is not performed because the concentration of the background in the blank is highly variable.

15.0 Calculations

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15.1 Concentrations of Unknowns – This method does not include reporting of tentatively identified compounds (TIC). However, reporting of up to 10 TIC's whose peak heights are ≥ 10% of the nearest internal standard may be requested or ≥ 40% of the nearest internal standard may be requested for low concentration analyses. Concentrations are calculated using the following formula using an assumed response factor (RF) of 1:

$$C(s) = \frac{C(is) * A(s) * DF}{A(is)}$$

Where:
C(s) = Concentration of Unknown (ug/L)
C(is) = Concentration of internal standard (ug/L)
DF = Dilution Factor
A(s) = Area of Unknown
A(is) = Area of associated internal standard

Tentatively identified compounds (TIC) are estimated concentrations and are reported with a "J" qualifier.

15.2 Concentrations of Calibrated Compounds:

$$C(s) = \frac{A(s) * C(is) * DF}{A(is) * RRF}$$

Where:
C(s) = Concentration of compound (ug/L)
C(is) = Concentration of associated internal standard (ug/L)
DF = Dilution Factor.
A(is) = Area of quantitation ion for associated internal standard
A(s) = Area of quantitation ion for compound
RRF = Average Relative Response Factor from five-point initial calibration

15.3 Calculation of Recovery - Calculate the recovery of the spiked 1,4-dioxane in the MS/MSD, LCS, and LCSD by the following equation:

$$Recovery = \%R = \frac{Cs - Cu}{Cn} \times 100$$

Where:
Cs = Measured concentration of the spiked sample aliquot
Cu = Measured concentration of the unspiked sample aliquot (use 0 for LCS and ICV)
Cn = Nominal (theoretical) concentration increase that results from spiking the sample, or the nominal concentration of the spike aliquot (for LCS and ICV)

15.4 Calculation of Precision - Precision is estimated from the relative percent difference (RPD) of the concentrations (not the recoveries) measured for matrix spike/matrix spike duplicate pairs, or for duplicate analyses of unspiked samples. The RPD is calculated according to the following equation below.

$$RPD = \frac{2*(\%Rec1 - \%Rec2) * 100}{(\%Rec1 + \%Rec2)}$$

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Where:
%Rec1 = Calculated % Recovery of the compound in the 1st sample aliquot
%Rec2 = Calculated % Recovery of the compound in the 2nd sample aliquot

15.5 Data Reporting - Based on the mass spectra, it is appropriate to report values that are at a level at or above the MDL, but less than the reporting level (PQL). In this region, an analyte can be qualitatively detected, but not accurately quantified. Any concentrations calculated to be less than the reporting level are flagged with a "J" qualifier. Sample specific RL's are derived by taking into account various sample specific data, which can include the amount of the sample subject to testing, % moisture, dilution factor, interferences and the base RL's for the analysis.

15.5.1 Reporting qualifiers are as follows:

B = Analyte is found in the associated method blank as well as the sample
E = Compound quantitation is above the instrument's calibration range for this analysis
J = Indicates an estimated quantitation value
U = Compound was analyzed for but not detected

15.5.2 Data Package Definitions - three levels of reporting are available. The difference between these levels has nothing to do with the quality of the work being performed, only how it is presented.

- Level 1: A level 1 data package consists of sample results only
- Level 2: A level 2 data package consists of sample results and simple Excel quality control forms.
- Level 3: A level 3 data package consists of sample, CLP-like forms with Quality Control results.
- Level 4: A level 4 data package consists of a full set of CLP-like forms, and all supporting documentation.

Electronic-Diskette is available upon request.

16.0 Method performance

16.1 Laboratory accuracy and precision data were obtained for the method analytes using laboratory control spikes. The analytes were at a concentration of 1 µg/L for the procedural combination of Methods 5030 and 8260. Results were obtained using the analytical instrumentation described in section 9.

16.2 With this data, method detection limits were calculate using the formula:

MDL = S t (n-1, 1-alpha = 0.99)

Where:
t(n-1, 1-alpha = 0.99) = Student's t value for the 99% confidence level with n-1 degrees of freedom
n = number of replicates
S = the standard deviation of the replicate analysis

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16.3 Precision and MDL study results are received and retained by the NEL Quality Assurance Manager.

17.0 Pollution prevention and waste management

17.1 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. Please refer to NEL's Chemical Hygiene Plan for further information.

17.2 The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18.0 Data Assessment, Acceptance criteria and Corrective Action

18.1 Data is initially reviewed by the analyst in the lab and signed as such. Following this, the data is secondarily reviewed by the organics supervisor or his/her designee before being put into its final data package form (where the data is reviewed by the QA/QC department before being sent to the client). Please refer to NEL's Quality Assurance Plan for further information.

18.2 Data that is out of control is marked as such and slated for re-analysis. Any corrective action undertaken is documented on a corrective action form (detailing the client information, problem, investigation findings and solution). This form is kept in the Quality Assurance Manager's office. Refer to Table 7 for a list of possible corrective actions.

18.3 When sample concentrations exceed the upper limit of the calibration curve, samples shall be diluted and reanalyzed (if possible) to bring the concentrations within the range of the calibration curve.

If dilution is not possible due to a lack of sample and/or sample extract, the analyte whose concentration lies over the upper limit of the calibration curve shall be reported with an "E" qualifier.

18.4 Routine Maintenance. On a daily basis the sparger tube is rinsed and the trap and column are baked for a minimum of 10 minutes. The following maintenance is performed as needed.

18.4.1 Change trap when response factors indicate a problem
18.4.2 Change sparger liner, septum, and clip column when contamination is suspected
18.4.3 Change column when peak separation is no longer adequate
18.4.4 Change or solvent rinse transfer lines to eliminate contamination
18.4.5 Clean source if BFB tune does not pass
18.4.6 Change rough pump oil when source is cleaned or annually at a minimum
18.4.7 Change compressed gas cylinders before they empty, preferably before they go under 500 psi

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19.0 Corrective action and contingencies for out of control data

Generally, any data that is out of control is considered unusable. There are, however, cases in which the laboratory supervisor will be made aware of the issue and, if the data is used, it will be thoroughly narrative noted. Refer to Table 7 for a list of possible corrective actions.

20.0 Waste Management

Please refer to the NEL Chemical Hygiene Plan and Spill Prevention Control & Contingency Plan (SPCC) for information regarding waste management.

21.0 References

Test Methods for EvaluatingSolid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996.

Standard Operating Procedure for Measurement of Purgeable 1,4-dioxane in Water by GC/MS, EIASOP-VOADIOX1, 05/21/03, EPA Region New England, North Chelmsford, MA

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TABLE 1: PRACTICAL QUANTITATION LIMITS

COMPOUND	WATER µg/L	Solid, METHANOL EXT., µg/kg*	Soil, Low Level µg/kg
1,4-dioxane	1	50	5

* NEL method routinely used for analysis of soils, sludges, and other solid materials; the low-level (heated purge) is used at the request of the client to achieve lower reporting levels.

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TABLE 2: ACCEPTABLE BFB TUNING CRITERIA

MASS	ION 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	LESS THAN 2% OF MASS 174
174	50% To 100% OF MASS 95
175	5 TO 9% OF MASS 174
176	GREATER THAN 95% BUT LESS THAN 101% OF MASS 174
177	5 TO 9% OF MASS 176

TABLE 3: CALIBRATION CRITERIA

Compound	Minimum RRF	Maximum % RSD	Maximum %D
1,4-dioxane	NA	30	30
1,4-dioxane-d8	NA	30	30

TABLE 4: SURROGATE RECOVERY FOR AQUEOUS SAMPLES

Surrogate Name	Aqueous Samples %Recovery
1,4-dioxane-d8	40-160

% Recovery Limits are from USEPA Region I SOP (EIASOP-VOADIOX1)

TABLE 5: SURROGATE RECOVERY FOR SOLID SAMPLES

Surrogate Name	Soil Samples %Recovery
1,4-dioxane-d8	40-160

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TABLE 6: INTERNAL STANDARDS AND ASSOCIATED TARGET COMPOUNDS

Bromofluorobenzene
1,4-dioxane 1,4-dioxane-d8

TABLE 7: 1,4-DIOXANE ORGANIC ANALYTICAL RUN SEQUENCE AND CORRECTIVE ACTION

Quality Control Criteria	Frequency	Acceptance	Corrective Action
BFB	Before Analysis and every 12 hours	Criteria Table 2	Reanalyze. Manual tune clean source.
ICAL: Minimum of five points	As Required.	maximum 30% RSD for 1,4-dioxane and 1,4-dioxane-d8	System check, mix new standards, recalibrate, reanalyze.
CCV	Beginning of each 12-hour window, after tune, or as a part of curve. End of each 12-hour sequence	maximum 30% RSD for 1,4-dioxane and 1,4-dioxane-d8	System check, recalibrate. If closing CCV os out, reanalyze batch
LCS/LCSD	Every analytical batch if MS and MSD not analyzed. Prepared from second source as compared to calibration	% Recovery 60-140%,	Check Std., check quantitation, evaluate MS/MSD, reanalyze analytical batch if data impacted.
Method Blank	Every analytical batch.	1,4-dioxane less than 1.0 ug/l	Check for contamination, reanalyze batch if data impacted.
Samples	Until 12-hour window is closed.	Results < Highest Calibration Std., Surrogates meeting Table 4 and 5 criteria, int. std. Areas – 50% to +100% of CCV istd area.	Dilute and reanalyze if over curve. If surrogate/internal std. Recovery fails, reanalyze to confirm matrix interference.
Matrix Spike/Matrix Spike Duplicate	Every analytical batch if sufficient sample. Prepared from second source as compared to calibration	% Recoveries 60-140%	Reanalyze if analytical problem. Evaluate LCS/LCSD if poor matrix.

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TABLE 8: DAILY 8260 ANALYSES

8260 Calibrations
(Standard VOC Mix in a 10 ml reagent water)

Final Concentration ppb	ul 1,4-dioxane 10 ug/ml	ul 1,4-dioxane-d8 10 ug/ml	ul BFB 10 ug/ml
1	1.0	1.0	1.0
5	5.0	5.0	1.0
10	10	10	1.0
20	20	20	1.0
50	50	50	1.0
100	100	100	1.0

1,4-dioxane: Restek, cat no. 30287, 2000 ug/ml in methanol. Dilute 5.0 ul of the Restek standard to 1 ml with methanol. (10 ppm final concentration)

1,4-dioxane (second source for LCS, LCSD, MS, MSD): 100 ul of 100 ug/ml stock (Ultra Scientific part number NV-150)) diluted to 1 ml with methanol (10 ppm final concentration)

1,4-dioxane-d8: dilute 4 ul of the neat compound to 2 ml in methanol (2000 ug/ml resultant). Add 5 ul of this 2000 ug/ml solution to 1 ml if methanol for a 10 ppm stock.

BFB IS: dilute 5.0 ul of BFB solution (Ultra, part # STS-110N, 2000 ug/ml in methanol) to 1.0 ml in methanol for a 10 ppm stock.

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PAGE:	1 OF 16

DOCUMENT HISTORY AND APPROVAL PAGE

TITLE:	**1,1-Dichloroethene By Gas Chromatography/Mass Spectrometry (GC/MS)-Modified EPA Method 8260B-SIM**
DEPARTMENTS AFFECTED:	**Organics**

ISSUE DATE:	REVISION CODE:	SOP #:	REVISION CHANGES:
08/01/08	-	3.3.52	Creation of SOP to be compliant with NELAC standards

DOCUMENT APPROVAL		
Originator: Abram Patenaude	Signature/Date: 080108	Title Organics Analyst
Approved by: Jim Galasyn	080108	Chemistry Lab Manager
Reviewed by: Joseph Casarin	080108	Organics Supervisor

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NEL	STANDARD OPERATING PROCEDURE		
TITLE **1,1-Dichloroethene By Gas Chromatography/Mass Spectrometry (GC/MS)-Modified EPA Method 8260B-SIM**			
SOP# 3.3.52	REV# -	EFFECTIVE 08/04/08	PAGE 2 OF 18
WRITTEN BY: Abram Patenaude	DEPARTMENTS AFFECTED: ORGANICS		

1.0 Test Method:

This standard operating procedure (SOP) was developed to maintain high standards of analytical performance in order to generate data of known and documented quality for our clients. The test method is EPA Method 8260, modified to test for 1,1-dichloroethene only.

2.0 Applicable Matrix or Matrices:

This method is applicable to a variety of solid waste matrices. NEL uses this method for ground & surface waters, drinking water, sludge, sediment, soils, and solid and waste oil samples.

3.0 Detection Limit:

Please see Table 1 for Standard Compound List and Reporting Limits for this method. Other analytes can be added by client request upon completion of a method detection limit study.

4.0 Scope and Application

This SOP is based on Method 8260B (USEPA Test Methods for Evaluating Solid Waste, Dec. 1996). Method 8260B describes the GC/MS procedure for the analysis of volatile organic compounds. The techniques by which compounds may be introduced into the GC/MS system are described in Method 5030 (purge-and-trap of aqueous samples) and Method 5035 (purge-and-trap of solid and waste oil samples). This method is applicable to nearly all types of samples regardless of water content, including ground and surface water, drinking water, waste solvents, oily wastes, soils and sediments. A gas chromatograph utilizes a temperature program to separate the purgeables before detection on a mass spectrometer.

5.0 Summary of Method

5.1 Basis Principles - The analytes are introduced into the GC/MS by purge-and-trap techniques (Method 5030 or Method 5035). Upon desorption the volatile compounds are introduced directly to the capillary column of a gas chromatograph equipped with a temperature program to separate the purgeables. The eluted analytes on the gas stream into the ion source of a mass spectrometer. The ionized molecules are focused and separated according to their mass/charge (m/z) ratio by the quadrupole analyzer. The signal is amplified by an electron multiplier and interpreted by the mass spectrometer data system to produce a total ion chromatogram with mass spectra for

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every data point on the chromatogram. The system is an HP 5890 Series II Gas Chromatograph with a 5970 Mass Spectrophotometer or equivalent.

5.2 General Method - The mass spectrometer is calibrated to recognize m/z values in the range of 35-550 amu. Reference spectra and retention times for analytes are obtained by the measurement of calibration standards under the same conditions used for samples. Analytes are quantitated based upon a minimum of a five part calibration. Hewlett-Packard Enviroquant ChemStation Ver. A.03.00 software (or equivalent) is utilized for identification and quantitation of data. The concentration of each identified component is measured by the relative response of the target compound to its respective internal standard.

5.3 The performance of the mass spectrometer is verified by the introduction of a 50ng 4-Bromofluorobenzene (BFB) tune standard. All analyses must occur within 12 hours of the passing BFB analysis time.

5.4 Before any samples can be analyzed a continuing calibration verification (CCV) standard must be analyzed to demonstrate that the instrument is stable relative to the initial calibration curve.

6.0 Definitions

6.1 INTERNAL STANDARD (IS) - Non-target analyte compounds that are similar to the target analytes but are not expected to be found in environmental media (generally, isotopically labeled target analytes are used for this purpose). Internal standards are added to every standard, quality control sample, and field sample at a known concentration prior to analysis. Internal standard responses are used as the basis for quantitation of target analytes.

6.2 SURROGATE ANALYTE (SS) - Non-target analytes compounds that are similar in composition and behavior to the target analytes but are not expected to be found in environmental media (often, isotopically labeled target analytes are used for this purpose) and are added to every standard, quality control sample, and field sample at a known concentration prior to preparation and/or analysis. Surrogate responses are used to evaluate the accuracy of the laboratory's performance of the analytical method in a specific sample matrix.

6.3 STOCK STANDARD SOLUTION - A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.

6.4 PRIMARY DILUTION STANDARD SOLUTION - A solution of several analytes prepared in the laboratory from stock standard solutions and diluted as needed to prepare calibration solutions and other needed analyte solutions.

6.5 CALIBRATION STANDARD (CAL) - A solution prepared from the primary dilution standard solution or stock standard solutions and the internal standards and surrogate analytes. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

6.6 4-BROMOFLUOROBENZENE STANDARD (BFB)– A standard solution containing BFB in methanol and used to evaluate the performance of the MS system. The

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introduction and evaluation of a 50ng BFB standard by the MS system must meet the criteria in Table 2.

6.7 CONTINUING CALIBRATION VERIFICATION STANDARD (CCV) - An analytical standard containing all target analytes, surrogate and internal standard compounds that is used to evaluate the performance of the instrument system with respect to a defined set of method criteria.

6.8 METHOD BLANK (BLK, similarly known as the LABORATORY REAGENT BLANK) - An aliquot of organic free reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The BLK is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

6.9 TRIP BLANK - An aliquot of reagent water or other blank matrix that is placed in a sample container in the laboratory and treated as a sample in all respects, including shipment to the sampling site, exposure to sampling site conditions, storage, preservation, and all analytical procedures. The purpose of the trip blank is to determine if method analytes or other interferences are present in the field environment.

6.10 LABORATORY CONTROL SAMPLE (LCS) - The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. When the results of the matrix spike analysis indicates a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

6.11 MATRIX SPIKE/ MATRIX SPIKE DUPLICATE (MS/MSD) - An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS/MSD is analyzed exactly like a sample. Its purpose is used to evaluate the accuracy and precision of the laboratory performance of the analytical method in a specific sample matrix.

6.12 REAGENT WATER – Organic free water. NEL uses carbon filtration of water from its Milli-Q deionized water system, or equivalent.

7.0 Interferences

7.1 During analysis, major contaminant sources are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap. The use of Teflon tubing, Teflon thread sealants, or flow controllers and rubber components in the purging device should be avoided since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. Analyses of laboratory reagent blanks provide information about the presence of contaminants. Subtracting blank values from sample results is not permitted.

7.2 Interfering contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing relatively high concentrations of volatile organic compounds. A preventive technique is

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between-sample rinsing of the purging apparatus and sample syringes with two to three portions of reagent water. After analysis of a sample containing high concentrations of volatile organic compounds, one or more laboratory reagent blanks should be analyzed to check for cross-contamination. The blank must have a concentration less than one-half of the reporting level in order for analysis to proceed.

7.3 Traces of ketones, methylene chloride, and some other organic solvents can be present even in the highest purity methanol. This is another potential source of contamination, and should be assessed before standards are prepared in the methanol. Methanol is stored in a separate area from the extractions laboratory.

8.0 Safety

8.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials or procedures.

8.2 Material Safety Data Sheets (MSDS) are made available to all personnel involved in the chemical analysis. A written environmental health and safety plan is reviewed by all NEL staff.

8.3 Please note chemicals that have the potential to be highly toxic or hazardous, the appropriate MSDS must be reviewed by the employee before handling the chemical. The following method analytes have been tentatively classified as known or suspected human or mammalian carcinogens: benzene, carbon tetrachloride, 1,4-dichlorobenzene, 1,2-dichloroethane, hexachlorobutadiene, 1,1,2,2-tetrachloroethane, 1,1,2-trichloroethane, chloroform, 1,2-dibromoethane, tetrachloroethene, trichloroethene, and vinyl chloride. Pure standard materials and stock standard solutions of these compounds and all other target compounds will be handled in a hood.

9.0 Equipment and Supplies

9.1 **Containers**

9.1.1 Sample storage containers: 40 ml screw cap vials equipped with a Teflon faced silicone septum, certified clean, known volume of 44 ml (see also Method 5035)

9.1.2 Standard Storage Containers: 1-5 ml Mininert vials with Teflon lined screw caps.

9.2 **Syringes**

9.2.1 5 ml-50 ml gas tight hypodermic syringes with Luer-Lok or fixed needle tips.

9.2.2 Micro syringe 10 - 1000 µl.

9.3 **Instrumentation**

9.3.1 Autosampler: Archon 51 Position Autosampler or equivalent.

9.3.2 LCS 2000 purge and trap or equivalent.

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9.3.3 Trap: Tekmar Purge Trap K or equivalent

9.3.4 Gas Chromatograph: Hewlett-Packard 5890 Series II or equivalent.

9.3.5 Mass Spectrometer: Hewlett-Packard 5970 MSD or equivalent.

9.3.6 Primary Column: Fused silica capillary column, Restek RTX-VMS 30m x 0.25 mm x 1.4 µm or equivalent.

9.4 **Data Acquisition and Processing**

Data System - Hewlett-Packard ChemStation software is used for data acquisition and HP Enviroquant software is used for data processing.

10.0 Reagents and Standards

10.1 **Reagents**

10.1.1 Methanol - Purge and Trap Grade, demonstrated to be free of analytes.

10.1.2 Reagent water – organic free deionized water or equivalent.

10.2 **Stock Standard Solutions** - These solutions are purchased as certified solutions or prepared from pure standard materials. Commercial standards arrive ampulized in concentrations ranging from 1-5 mg/ml. Solutions are stored per manufacturer's recommendations. Once opened, these concentrated solutions expire at the manufacturer's expiration date or one year from opening, whichever is the most conservative.

10.3 **Primary Dilution Standards** - Stock standard solutions are used to prepare primary dilution standard solutions that contain all the analytes of interest in methanol. The primary dilution standards are prepared at concentrations that can be easily diluted to prepare aqueous calibration solutions that will bracket the working concentration range. The primary dilution standard solutions are stored with minimal headspace and checked frequently for signs of deterioration or evaporation. Methanol solutions of gaseous standards are not stable for more than one week at >0°C.

10.4 **Calibration Standards** – Five or more concentrations for calibration in the range 0.2 to 2.5 µg/L for 1,1-dichloroethene. Other concentrations and volumes may be used provided they meet method requirements.

10.5 **Calibration Verification Standard (CCV)** – Standard prepared at a concentration of 1 µg/L for 8260B analysis that is used to evaluate the initial calibration curve.

10.6 **Method Blank (BLK)** – Sample of organic free reagent water.

10.7 **Laboratory Control Sample (LCS)** -.- Standards prepared at a target concentration of 1.0 ppb from a secondary source as compared to the CCV.

10.8 **Matrix Spike/Matrix Spike Duplicate (MS/MSD)** - Matrix spikes and matrix spike duplicates are prepared and analyzed with each batch of 20 client specific samples of the same matrix, assuming sufficient sample is provided by the client. Matrix spikes

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are at a concentration of 1 µg/L. If the client does not specify a sample to be used as a MS/MSD, NEL will analyze a LCS Duplicate (LCSD) to verify precision.

10.9 **Internal Standard and Surrogates Fortification Solutions**- The internal standard is prepared by diluting 5 µl of fluorobenzene solution (2000 µg/ml in methanol) to 1.0 ml in methanol, for a final concentration of 1.0 ppm. The surrogate standard is prepared by diluting 4 µl of 1,2-dichloroethane-d_4 (2500 µg/ml in methanol) solution to 1.0 ml in methanol, for a final concentration of 1.0 ppm.

11.0 Sample Collection, Preservation, shipment and storage

11.1 **Sample collection** - Sample collection for aqueous samples is described in Method 5030. Sample collection for solid samples is described in Method 5035.

11.2 **Preservation** - Sample preservation for aqueous samples is described in Method 5030. Sample preservation for solid samples is described in Method 5035.

11.3 **Storage** - Samples are analyzed within fourteen days of collection. Other holding times may be selected by the client to conform to local regulatory requirements. Samples are stored at 4°C ± 2°C in a storage area free of organic solvent vapors and direct or intense light. The pH of each aqueous sample is monitored after analysis, with the value recorded in the injection logbook.

12.0 Quality Assurance/Quality Control

12.1 **4-Bromofluorobenzene (BFB)** - Prior to any analyses a 50ng BFB standard is introduced into the GC/MS for 8260B analysis. The BFB spectrum must meet all criteria in Table 2 before analysis of any standards or samples can follow.

12.2 **Method detection limits (MDLs)** are determined annually in accordance with the method described in 40 CFR, Part 136, Appendix B. The results of these studies are kept on file by the QA Manager.

12.3 **Method Blank** – One method blank must be run with every batch of samples after the calibration standards. An acceptable blank must not contain 1,1-dichloroethene at a concentration greater than the reporting level, 0.2 µg/l. If the method blank exceeds these criteria, the analytical system may be out of control. The source of the contamination must be investigated and appropriate corrective measures must be taken and documented before further sample analysis proceeds

12.4 **Laboratory Control Sample (LCS)** - The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. When the results of the matrix spike analysis indicates a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

12.5 **Continuing Calibration Verification Standard (CCV)** - A standard sample of target compounds made from the same standard used to prepare the calibration curve. The CCV is run at 1 µg/L.

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12.6 **Matrix Spike and Matrix Spike Duplicate (MS/MSD)** – The laboratory will analyze a MS/MSD pair when requested by the client. If a client MS/MSD request has not been received every twenty sample analyses, a laboratory control spike duplicate (LCSD) will be analyzed to serve as a measure of the precision of the batch. The MS/MSD will be prepared at a spike concentration of 1 µg/L.

12.7 **Surrogates/Internal Standards** - One compound is used as the surrogate: 1,2-dichloroethane-d_4. One compound is used as the internal standard: fluorobenzene (BFB). Acceptable recovery of the surrogate 1,2-dichloroethane-d_4 is 60%-140%.

12.8 **Internal Standard Area Response**

12.8.1 The relative retention time (RRT) of the sample analyte must be within ± 30 seconds of the RT of the standard analyte.

12.8.2 If the extended ion current profile (EICP) area in the CCV changes by a factor of two (-50% to +100%) from that in the midpoint lead in the most recent initial calibration curve, corrective action must be taken.

12.8.3 If the EICP area of any internal standard in a QC or field sample changes by more than a factor of two (-50% to +100%) from that in the CCV, reanalysis must occur to confirm matrix interference.

13.0 Calibration and standardization

13.1 **BFB** - The ion abundances shown in Table 2 must be met before analysis of any standards or samples may proceed.

13.2 **Initial Calibration** - The concentrations for the initial calibration are in the range of 0.2 to 2.5 µg/L. A minimum of five points are used to calibrate each analyte. A response factor (RF) is calculated for each analyte and/or isomer pair for each calibration solution using the appropriate internal standard. The calculation is performed as follows:

$$RF = \frac{(Ax)\,(Qis)}{(Ais)(Qx)}$$

Where:
Ax = integrated abundance of the quantitation ion of the analyte
Ais = integrated abundance of the quantitation ion of the internal standard
Qx = quantity of analyte purge in nanograms or concentration units
Qis = quantity of internal standard purged in ng or concentration units

For each analyte and surrogate, calculate the mean response factor from analyses of the calibration solutions. Calculate the standard deviation (SD) and relative standard deviation (RSD) from each mean (M).

$$\%RSD = 100 * \frac{SD}{M}$$

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The %RSD of 1,1-dichloroethene must be ≤30%. If this criteria is not met maintenance is performed on the instrument and/or a new calibration curve is acquired.

13.3 **Continuing Calibration Verification (CCV)** – The calibration curve must be verified at the beginning of each run. The concentration of the CCV is 1 µg/L. The RF is calculated for both 1,1-dichloroethene and 1,2-dichloroethane-d_4. The percent difference is calculated using the following equation:

$$\% \text{ Difference} = \frac{RFv - RF}{RF} (100)$$

Where:
RFv = Response Factor from the analyses of the verification standard
RF = Mean Response Factor from the initial calibration

The percent difference for both analytes must be ≤ 30%. In addition, the internal standard retention time should not change by more than 30 seconds from the mid-point standard level of the most recent initial curve sequence. The integrated areas of the internal standards in the calibration verification standard should not change by more than a factor of two (-50% to +100%) from that in the mid-point standard level of the most recent initial calibration sequence. If the above criteria are not met, maintenance is performed on the instrument and/or a new calibration curve is acquired.

14.0 Procedure

14.1 **Sample Introduction and Purging** - See preparation and introduction Methods 5030 and 5035. Again, all calibration standards, field samples, and QC samples must be heated to 80 degrees C prior to initiation of purging.

14.1.1 See Tables 8 and 9 for preparation of daily check standards and calibration curves.

14.2 **Gas Chromatography/Mass Spectrometry** - Data is acquired and stored over the nominal mass range 35-550 with a total cycle time (including scan overhead time) of two seconds or less. The cycle time is adjusted to measure five or more spectra during the elution of each GC peak. A multi-stage temperature ramp is used to separate the components of interest for this analysis. A typical GC temperature program is described below.

Initial temperature 35°C, initial time 5 min.
Ramp1: 10°C/min. to 90°C, hold for 0 min.
Ramp2: 35°C/min. to 220°C, hold for 2.8 min.

Instrument control and acquisition parameters are defined on the ChemStation software for each instrument.

14.3 **Identification of Analytes** - A sample is identified by comparison of its mass spectrum (after background subtraction) to a reference spectrum in the user-created data base. The GC retention time for each analyte should be with ± 30 seconds of the midpoint standard's retention time in the initial calibration curve.

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14.3.1 In general, all ions that are present above 10% relative abundance in the mass spectrum of the standard should be present in the mass spectrum of the sample component and should agree within absolute 20%. For example, if an ion has a relative abundance of 30% in the standard spectrum, its abundance in the sample spectrum should be in the range of 10-50%. Some ions, particularly the molecular ion, are of special importance, and should be evaluated even if they are below 10% relative abundance.

14.3.2 Identification requires expert judgment when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When GC peaks obviously represent more than one sample component (i.e., broadened peak with shoulder(s) or valley between two or more maxima), appropriate analyte spectra and background spectra can be selected by examining plots of characteristic ions for tentatively identified components. When analytes coelute (i.e., only one GC peak is apparent), the identification criteria can be met but each analyte spectrum will contain extraneous ions contributed by the coeluting compound. Because purgeable organic compounds are relatively small molecules and produce comparatively simple mass spectra, this is not a significant problem for most method analytes.

14.3.3 Structural isomers that produce very similar mass spectra can be explicitly identified only if they have sufficiently different GC retention times. Acceptable resolution is achieved if the height of the valley between two peaks is less than 25% of the average height of the two peaks. Otherwise, structural isomers are identified as isomeric pairs. Two of the three isomeric xylenes are examples of structural isomers that are not resolved on the capillary column. These groups of isomers will be reported as isomeric pairs.

14.3.4 Methylene chloride, acetone, carbon disulfide, and other background components appear in variable quantities in laboratory and field reagent blanks and generally cannot be accurately measured. Subtraction of the concentration in the blank from the concentration in the sample is not performed because the concentration of the background in the blank is highly variable.

15.0 Calculations

15.1 Concentrations of Calibrated Compounds:

$$C(s) = \frac{A(s) * C(is) * DF}{A(is) * RRF}$$

Where:

C(s) = Concentration of compound (µg/L)
C(is) = Concentration of associated internal standard (µg/L)
DF = Dilution Factor.
A(is) = Area of quantitation ion for associated internal standard
A(s) = Area of quantitation ion for compound
RRF = Average Relative Response Factor from five-point initial calibration

15.2 Calculation of Recovery - Calculate the recovery of the spiked 1,1-dichloroethene in the MS/MSD, LCS, and LCSD by the following equation:

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$$Recovery = \%R = \frac{Cs - Cu}{Cn} \times 100$$

Where:
Cs = Measured concentration of the spiked sample aliquot
Cu = Measured concentration of the unspiked sample aliquot (use 0 for LCS and ICV)
Cn = Nominal (theoretical) concentration increase that results from spiking the sample, or the nominal concentration of the spike aliquot (for LCS and ICV)

15.3 Calculation of Precision - Precision is estimated from the relative percent difference (RPD) of the concentrations (not the recoveries) measured for matrix spike/matrix spike duplicate pairs, or for duplicate analyses of unspiked samples. The RPD is calculated according to the following equation below.

$$RPD = \frac{2*(\%Rec1 - \%Rec2) *100}{(\%Rec1+\%Rec2)}$$

Where:
%Rec1 = Calculated % Recovery of the compound in the 1st sample aliquot
%Rec2 = Calculated % Recovery of the compound in the 2nd sample aliquot

15.4 Data Reporting - Based on the mass spectra, it is appropriate to report values that are at a level at or above the MDL, but less than the reporting level (PQL). In this region, an analyte can be qualitatively detected, but not accurately quantified. Any concentrations calculated to be less than the reporting level are flagged with a "J" qualifier. Sample specific RL's are derived by taking into account various sample specific data, which can include the amount of the sample subject to testing, % moisture, dilution factor, interferences and the base RL's for the analysis.

15.4.1 Reporting qualifiers are as follows:

B = Analyte is found in the associated method blank as well as the sample
E = Compound quantitation is above the instrument's calibration range for this analysis
J = Indicates an estimated quantitation value
U = Compound was analyzed for but not detected

15.4.2 Data Package Definitions - three levels of reporting are available. The difference between these levels has nothing to do with the quality of the work being performed, only how it is presented.

- Level 1: A level 1 data package consists of sample results only
- Level 2: A level 2 data package consists of sample results and simple Excel quality control forms.
- Level 3: A level 3 data package consists of sample, CLP-like forms with Quality Control results.
- Level 4: A level 4 data package consists of a full set of CLP-like forms, and all supporting documentation.

Electronic-Diskette is available upon request.

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16.0 Method performance

16.1 Laboratory accuracy and precision data were obtained for the method analytes using laboratory control spikes. The analytes were at a concentration of 1 µg/L for the procedural combination of Methods 5030 and 8260. Results were obtained using the analytical instrumentation described in section 9.

16.2 With this data, method detection limits were calculate using the formula:

MDL = S t (n-1, 1-alpha = 0.99)

Where:

t(n-1, 1-alpha = 0.99) = Student's t value for the 99% confidence level with n-1 degrees of freedom
n = number of replicates
S = the standard deviation of the replicate analysis

16.3 Precision and MDL study results are received and retained by the NEL Quality Assurance Manager.

17.0 Pollution prevention and waste management

17.1 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. Please refer to NEL's Chemical Hygiene Plan for further information.

17.2 The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18.0 Data Assessment, Acceptance criteria and Corrective Action

18.1 Data is initially reviewed by the analyst in the lab and signed as such. Following this, the data is secondarily reviewed by the organics supervisor or his/her designee before being put into its final data package form (where the data is reviewed by the QA/QC department before being sent to the client). Please refer to NEL's Quality Assurance Plan for further information.

18.2 Data that is out of control is marked as such and slated for re-analysis. Any corrective action undertaken is documented on a corrective action form (detailing the client information, problem, investigation findings and solution). This form is kept in the Quality Assurance Manager's office. Refer to Table 7 for a list of possible corrective actions.

18.3 When sample concentrations exceed the upper limit of the calibration curve, samples shall be diluted and reanalyzed (if possible) to bring the concentrations within the range of the calibration curve.

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If dilution is not possible due to a lack of sample and/or sample extract, the analyte whose concentration lies over the upper limit of the calibration curve shall be reported with an "E" qualifier.

18.4 Routine Maintenance. On a daily basis the sparger tube is rinsed and the trap and column are baked for a minimum of 10 minutes. The following maintenance is performed as needed.

18.4.1 Change trap when response factors indicate a problem
18.4.2 Change sparger liner, septum, and clip column when contamination is suspected
18.4.3 Change column when peak separation is no longer adequate
18.4.4 Change or solvent rinse transfer lines to eliminate contamination
18.4.5 Clean source if BFB tune does not pass
18.4.6 Change rough pump oil when source is cleaned or annually at a minimum
18.4.7 Change compressed gas cylinders before they empty, preferably before they go under 500 psi

19.0 Corrective action and contingencies for out of control data

Generally, any data that is out of control is considered unusable. There are, however, cases in which the laboratory supervisor will be made aware of the issue and, if the data is used, it will be thoroughly narrative noted. Refer to Table 7 for a list of possible corrective actions.

20.0 Waste Management

Please refer to the NEL Chemical Hygiene Plan and Spill Prevention Control & Contingency Plan (SPCC) for information regarding waste management.

21.0 References

Test Methods for EvaluatingSolid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996.

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TABLE 1: PRACTICAL QUANTITATION LIMITS

COMPOUND	WATER µg/L	Solid, METHANOL EXT., µg/kg*	Soil, Low Level µg/kg
1,1-dichloroethene	0.20	10	1.0

* NEL method routinely used for analysis of soils, sludges, and other solid materials; the low-level (heated purge) is used at the request of the client to achieve lower reporting levels.

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TABLE 2: ACCEPTABLE BFB TUNING CRITERIA

MASS	ION 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	LESS THAN 2% OF MASS 174
174	50% To 100% OF MASS 95
175	5 TO 9% OF MASS 174
176	GREATER THAN 95% BUT LESS THAN 101% OF MASS 174
177	5 TO 9% OF MASS 176

TABLE 3: CALIBRATION CRITERIA

Compound	Minimum RRF	Maximum % RSD	Maximum %D
1,1-dichloroethene	NA	30	30
1,2-dichloroethane-d_4	NA	NA	NA

TABLE 4: SURROGATE RECOVERY FOR AQUEOUS SAMPLES

Surrogate Name	Aqueous Samples %Recovery
1,2-dichloroethane-d_4	60-140

TABLE 5: SURROGATE RECOVERY FOR SOLID SAMPLES

Surrogate Name	Soil Samples %Recovery
1,2-dichloroethane-d_4	60-140

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TABLE 6: INTERNAL STANDARDS AND ASSOCIATED TARGET COMPOUNDS

fluorobenzene
1,1-dichloroethene 1,2-dichloroethane-d_4

TABLE 7: 1,1-DCE ORGANIC ANALYTICAL RUN SEQUENCE AND CORRECTIVE ACTION

Quality Control Criteria	Frequency	Acceptance	Corrective Action
BFB	Before Analysis and every 12 hours	Criteria Table 2	Reanalyze. Manual tune clean source.
ICAL: Minimum of five points	As Required.	maximum 30% RSD for 1,1-dichloroethene	System check, mix new standards, recalibrate, reanalyze.
CCV	Beginning of each 12-hour window, after tune, or as a part of curve. End of each 12-hour sequence	maximum 30% RSD for 1,1-dichloroethene	System check, recalibrate. If closing CCV os out, reanalyze batch
LCS/LCSD	Every analytical batch if MS and MSD not analyzed. Prepared from second source as compared to calibration	% Recovery 1,1-DCE 60-140%	Check Std., check quantitation, evaluate MS/MSD, reanalyze analytical batch if data impacted.
Method Blank	Every analytical batch.	1,1-DCE less than 0.20 µg/l	Check for contamination, reanalyze batch if data impacted.
Samples	Until 12-hour window is closed.	Results < Highest Calibration Std., Surrogates meeting Table 4 and 5 criteria, int. std. Areas – 50% to +100% of CCV istd area.	Dilute and reanalyze if over curve. If surrogate/internal std. Recovery fails, reanalyze to confirm matrix interference.
Matrix Spike/Matrix Spike Duplicate	Every analytical batch if sufficient sample. Prepared from second source as compared to calibration	% Recoveries 60-140%	Reanalyze if analytical problem. Evaluate LCS/LCSD if poor matrix.

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TABLE 8: DAILY 8260 ANALYSES

8260 Calibrations
(Standard VOC Mix in a 100 ml reagent water)

Final Concentration ppb	µl 1,1-dichloroethene 10 µg/ml	µl 1,2-dichloroethane-d_4 10 µg/ml	µl fluorobenzene 10 µg/ml
0.2	2.0	2.0	10
0.5	5.0	5.0	10
1.0	10	10	10
1.5	15	15	10
2.0	20	20	10
2.5	25	25	10

1,1-dichloroethene: Ultra Scientific, cat no. HC-170, 100 µg/ml in methanol. Dilute 100 µl of the Ultra Scientific standard to 1 ml with methanol. (10 ppm final concentration)

1,1-dichloroethene: Supelco, cat no. 4-8601 (second source for LCS, LCSD, MS, MSD), 200 µg/ml in methanol. Dilute 50 µl of the Supelco standard to 1 ml with methanol. (10 ppm final concentration)

1,2-dichloroethane-d_4: Restek, cat no. 30004, 2500 µg/ml in methanol. Dilute 4 µl of the Restek standard to 1 ml in methanol. (10 ppm final concentration)

fluorobenzene: Ultra Scientific, cat no. STS-160, 2000 µg/ml in methanol. Dilute 5.0 µl of the Ultra Scientific standard to 1 ml with methanol. (10 ppm final concentration)

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REV:	O
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DOCUMENT HISTORY AND APPROVAL PAGE

TITLE:	Procedure for Proper Sample Receipt and Log-In
DEPARTMENTS AFFECTED:	Micro, Inorganic and Organic Chemistry Sections

ISSUE DATE:	REVISION CODE:	SOP #:	REVISION CHANGES:
12/18/01	A	3.4.0	Update to Standardized SOP
05/28/02	B	3.4.0	Update SOP to comply with client audit
6/12/02	C	3.4.0	Update SOP to comply with client audit
5/19/04	D	3.4.0	Update SOP to comply with client audit
6/9/05	E	3.4.0	Annual Review & Update
4/24/06	F	3.4.0	Annual Review & Update
06/19/06	G	3.4.0	Update to Improve Lab/Client Communication
09/25/06	H	3.4.0	Update to Navy Standards
10/06/06	I	3.4.0	Update to Include NELAC Audit Findings
11/09/06	J	3.4.0	Update for Corrections
12/11/06	K	3.4.0	Update for Corrections to include pH paper range Clarification
03/01/07	L	3.4.0	Annual Review & Update
11/5/07	M	3.4.0	Update to Include Microbiology Sample Plan
01/17/08	N	3.4.0	Annual Review & Update
04/10/08	O		Update to include Sample Acceptance Policy

DOCUMENT APPROVAL		
Originator: Pamela Doughty	Signature/Date: Pamela Doughty 4/10/08	Title Total Quality Manager
Approved by: James Galasyn	J Galasyn 041008	Chemistry Lab Manager
Reviewed by: Brett Goodrich	Brett Goodrich 4-10	Microbiology Manager

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NEL	STANDARD OPERATING PROCEDURE		
TITLE: **Procedure for Proper Sample Receipt and Log-in**			
SOP# 3.4.0	REV# O	EFFECTIVE 04/10/08	PAGE 2 of 23
WRITTEN BY: Pamela M. Doughty	DEPARTMENTS AFFECTED: Micro, Inorganic and Organic Chemistry Sections		

1.0 Test Method:

N/A

2.0 Applicable Matrix or Matrices:

N/A

3.0 Detection Limit:

N/A

4.0 Scope and Application:

This standard operating procedure (SOP) was developed to establish and maintain standards for the receipt and log-in process for samples that are accepted at Northeast Laboratory Services.

5.0 Summary of the Methods for the Proper Procedures for Sample Receipt and Log-in Process.

Procedures for sample receipt include a comparison of the chain of custody to the sample containers that are received. The condition of the samples, collection date and sample receipt date are reviewed to ensure samples can be analyzed with-in the recommended holding time. A sample receipt form and an internal chain of custody are filed with the chain of custody.

6.0 Definitions

6.1 SRCR: Sample Receipt Condition Report (An example is attached)

6.2 COC: Chain of Custody (An example is attached)

6.3 ICOC: Internal Chain of Custody (An example is attached)

6.4 CAF: Corrective Action Form (An example is attached)

6.5 RSO: Radiation Safety Officer

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6.6 Thermometer: An instrument by which temperatures are measured

6.7 Neon Label: A colored label preprinted with a sequential number used to track the sample throughout the lab.

6.8 Sample Receipt Fax/E-mail Transmittal Form: Used for any discrepancies between the client's chain-of-custody and samples received to document that discrepancy.

7.0 Interferences

N/A

8.0 Safety

No specific safety measures beyond good laboratory practices are necessary.

9.0 Equipment and Supplies

9.1 COC

9.2 SRCR

9.3 IOC

9.4 Thermometer

9.5 pH indicator strips (wide range alkacid test ribbon)

10.0 Reagents and Standards

N/A

11.0 Sample Collection, Preservation, Shipment and Storage

11.1 Once samples arrive at NEL, the pH of preserved samples and sample volume is verified using pH indicator paper to encompass a pH range of 2-10 (Fisher Alkacid Test, Cat. No. A-979, range pH 2-10) and noted on the IOC. Samples are then stored in the chemistry storage cooler or in the volatile organic cooler. Refrigerator temperatures will be monitored seven days a week. For more information on maintenance of equipment please see SOP 3.7.2. The temperature of the cooler is recorded on the SRCR after taking the temperature of the cooler blank. If it is out of range, a call for instructions is placed to the customer.

11.2 For microbiology customers, *Standard Methods* 9060A sample collection procedures are followed. Please see the attached chain of custody forms with the procedures that are sent to customers.

12.0 Quality Assurance/Quality Control

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12.1 Sample log-in information is reviewed by the Chemistry Project Manager from review of the chain-of-custody records. Microbiology chain of custodies are reviewed by the bench analyst at the time of sample analysis, and with the final report prior to submittal to the client by the Microbiology Laboratory Manager. The following information is reviewed and corrected if necessary:

- Client Information for Reporting
- Client Information for Invoicing
- Purchase Order Number
- Project Name
- Sample Description
- Sample Collection Date & Time
- Sample Submitted Date & Time
- Sample Matrix
- Sample Type
- Analysis Requested
- Pricing
- Discrepancies noted on the Sample Receipt Fax/E-mail Transmittal Form

12.2 Once the information is verified as correct, the Chemistry Project Manager will pass the chain-of-custodies onto Administration. If any discrepancies are noted, the chemistry project manager will pass those onto the chemistry lab manager who will relay the necessary information to the analysts.

12.3 Changes that affect the analyst should be reported to the analysts in written form and attached to the chain of custody copy in the chemistry laboratory. Typographical errors, wrong tests, dates, etc. should be noted on a discrepancy report and filed with the QA Manager.

12.4 Level IV data packages require the following procedure for sample receipt quality assurance:

12.4.1 Samples are checked against the chain of custody. Inconsistencies are noted on the SRCR. The client is contacted for instructions using the Sample Receipt Fax/E-mail Transmittal Form. Any e-mail or fax correspondence received pertaining to the samples are attached to the COC packet and a copy is also photocopied and placed into the Chain of Custody Book in the chemistry laboratory.

12.4.2 An SDG number is assigned to the COC.

12.4.3 Samples are numbered following 14.1.8.

12.4.4 Samples are checked for preservation. When it is necessary to add acid, a notation is made one the ICOC and the client is contacted. The ICOC is completed and filed in the chemistry department.

12.4.5 Once samples are logged into the system, a sample log in summary is printed following this procedure. The log in summary is then compared to this COC. Any changes will be made following the procedures set in 12.1

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- 12.3. After this, a sample receipt printout is printed. Enter into Access, go to Labworks/dater/accrep/accrep. Select "rpt Sample Receipt", enter SBT number, and enter the date required to be printed, select print. The sample receipt printout, sample receipt form, copy of the COC and a cover letter are then faxed and/or e-mailed if required by the client to the client as soon as possible. This packet is then forwarded to the reporting clerk to file for later use. A copy of the Sample Receipt Report is always placed in the Chain of Custody Book in the chemistry laboratory with a copy of the chain of custody.

12.4.6 Depending on the project, if necessary, samples are then logged into the EDD system. After this is complete, a case narrative is started for that data package.

12.5 The Laboratory Manager, prior to the report going out to the client, performs a final chain of custody review. The above information is checked against a trial report along with other non-log-in related information.

13.0 Calibration and Standardization

N/A

14.0 Procedures

14.1 Sample Receipt

14.1.1 Radioactive samples are received by the RSO. See SOP # 3.6.3

14.1.2 Non-radioactive coolers or containers are opened and the temperature is recorded on the SRCR. Containers with samples that are known to be or most likely are, particularly hazardous are opened underneath a fume hood in the chemistry department.

14.1.3 Sample containers are checked against the COC. Inconsistencies, such as mislabeled containers, missing or damaged containers, incorrectly preserved containers, hold times or incomplete COC's and the condition of the containers are noted on the SRCR. The client contact is notified via fax, e-mail or phone. The Sample Receipt Fax/E-mail Transmittal form is used to document (see attached form) any inconsistencies or problems and ask for instructions. The final decision is noted in the SRCR comment section. A copy of the Sample Receipt Fax/E-mail Transmittal form is attached to the chain of custody copy in the Chain of Custody Book in the chemistry laboratory so that all analysts are aware of any discrepancies and the client's final decision on the sample.

14.1.4 The collection date and time is observed to ensure samples arrive within the recommended holding time. Sample containers are also inspected for proper preservation. Based on the client's instructions, samples that are too old or are not preserved can be processed (preserved in-house and analyzed) or may be cancelled. A note concerning the condition of the

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sample(s) upon arrival at the laboratory and the client's instructions regarding that condition must appear on the final report.

14.1.5 The COC is signed and dated by the technician receiving the sample.

14.1.6 An SRCR form is completed for each project and attached to the COC.

14.1.7 Samples are assigned a unique sequential laboratory identification number. Prefixes to these numbers vary by year and department, PA represents 2001 potability samples, AD represent 2001 chemistry and samples with both microbiological and chemistry analyses, MA represents 2001 samples for strictly microbiological testing of air and food samples. The sequential numbers are fixed to the sample containers using neon labels. Yellow signifies chemistry samples and pink signifies microbiology samples. Permanent markers are used if the number of bottles per sample exceed 3 for chemistry samples and 1 for microbiology samples.

14.1.8 Samples containing more than one sample container per site are assigned an additional unique sample ID. Within the set the original sample number is followed by an additional sequential number - starting with 1 which is preceded by a decimal. For example, AD13652.1, AD13652.2 and so on until all bottles are labeled.

14.1.9 Samples with the laboratory ID number are then placed on a transport cart for the sample custodian to continue with the sample process procedure. The pH of all samples, except volatile organic samples, will be taken under a hood in the radon room by removing an aliquot of sample and then using a small disposable glass rod to sample from the aliquot and then touch the rod to a pH indicator strip as noted in Section 11.0 of this SOP, and noted on the IOC. Samples are then placed in the appropriate sample storage cooler. The corresponding storage location and date and time the sample was placed there, is noted on the IOC. Samples that need immediate attention are given to the appropriate analyst with a notation on the IOC.

14.1.9 (a) The pH of volatile organic samples is taken by the bench analyst and recorded on the instrument run log for the particular analytical method.

NEL is a full service lab; however, some analyses need to be subcontracted. Customers are notified of this, either through quotes, fax or e-mail. For more information on subcontracted samples see SOP 3.4.1.

14.1.10 For residual chlorine checks the following procedures are used:

14.1.10.1 For volatile organics in drinking water, EPA Method 524.2, 40 ml amber ascorbic acid preserved vials are sent to all clients.

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14.1.11 Copies of the COC are then distributed to any department that may need them. A copy of the COC is also placed in a binder in the chemistry department.

14.2 Sample Log-in

14.2.1 Samples are logged into the Labworks system by entering the corresponding information provided on the COC.

14.2.2 Once samples are entered into the LabWorks System, the COC is forwarded to the Chemistry Project Manager for review and then to the administrative department for filing.

14.2.3 Once preliminary reports and invoices are printed, the COC is matched with the report and the Chemistry/Microbiology Laboratory Manager will review the information on the COC to the reports.

15.0 Calculations

N/A

16.0 Method Performance

N/A

17.0 Pollution Prevention

N/A

18.0 Data Assessment, Acceptance Criteria and Corrective Action

Modifications made to samples for any reason are relayed to the appropriate department manager. If the changes are due to an amendment to the COC at the request of the client, the changes are noted on the COC and the COC is dated and signed by the sample receipt technician.

19.0 Waste Management

Any and all waste generated, through the above procedures, is handled according to the Chemical Hygiene and Hazardous Waste Management and Spill Prevention Control and Contingency Plan.

20.0 References

NELAC Quality Systems Chapter 5, June 3, 2005

Current Revision of Northeast Laboratory Services Quality Program Manual

21.0 Tables, Diagrams, Flowcharts and Validation Data

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See attached forms

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Northeast Laboratory SERVICES

SAMPLE RECEIPT FAX/E-MAIL TRANSMITTAL FORM

To:		From:	Client Service Representative
Fax/E-mail:		Pages:	1
Re:	Problem w/sample(s) received	Date:	
Project Name or Number:			

YOU MUST REPLY TO THIS FAX/E-MAIL WITH THE NECESSARY INFORMATION TO ASSURE THE PROCESSING OF YOUR SAMPLE(S).

We are holding up the processing of your sample project pending the resolution of the following:

	Past Hold Time		Not Preserved		Not Labeled
	Not at Appropriate Temperature		Analysis Not Specified		Incomplete Paperwork
	Samples Not Received		Other		

We will hold your project and wait for instructions on whether to proceed with the project. If hold time appears likely to expire before you can respond, we will proceed with analyses.

Please check one of the following and either fax to 207/873-7022 or e-mail to mbawcum@nelabservices.com:

	I am aware of the discrepancy – please proceed with the analyses.
	Discard or return the sample(s) – we will resubmit or resolve the issue in another manner.

Name:	Date:

This message is intended only for the use of the individual or entity to which it is addressed and may contain information that is privileged, confidential and exempt from disclosure under applicable law. If the reader of this message is not the intended recipient or the employee or agent responsible for delivering the message to the intended recipient, you are hereby notified that any dissemination, distribution or copying of the communication is strictly prohibited. If you receive this communication in error, please notify us by telephone immediately (toll free 1-800-244-8378) to arrange for return of the original document(s) to NEL.

NORTHEAST LABORATORY SERVICES
227 CHNA ROAD, WINSLOW, ME 04901
PO BOX 788, WATERVILLE, ME 04903-0788
1-800-244-8378

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Dr #_____
Date:_____

DISCREPANCY REPORT

A)

Person who discovered the problem			Possible ramifications if not discovered:
Sample #:	Client:	Lab Area:	

B) Describe Discrepancy:

Problem Type:

	Equipment		Insufficient Sample		QC Failure
	Process Change Effectivity		Lab Accident		Proficiency Failure
	Log-in		Labeling Error		Other (describe below)
Initiator:				Date:	

C) Recommended Disposition:

	Accept as is/No Process Change Required (provide justification below)		Reanalyze Sample
	Accept as is/Process Change Required (P.C.#:)		Reissue Report
	Discrepancy Corrected or Void		
Comments:			
Initiator/Supervisor:		Date:	

D) Authorization:

Department	Signature	Date
QA Manager		
Laboratory Manager		
Technical Manager		
Supervisor		
Other Appropriate Party		
Comments:		

E) Final Disposition:

F) Corrective/Preventive Action:

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CC#_____
Date_____

CUSTOMER COMPLAINT FORM

Date:		By:		Type:	Other
					Laboratory Use Only

Laboratory Number:		Customer:	
Date Received:		Customer #:	
Date Reported:		Address:	
Department Affected:		Inquiry Made by:	
P.O. #		Phone #:	

Customer Report:

Technical Service:
Summary Response & Recommended Action (if any):

Disposition:					
Issue Credit:		Yes		No	Credit Memo #:
Laboratory #					Amount:
Credit Issued:	Date:				By:
Reanalyze Sample:		Yes		No	Parameter(s) Reanalyzed:
New Lab # for Reanalysis:					By:
Reissue Report:		Yes		No	Report Reissue Date:
Reporting/Accounting Notified to Issue Credit:					

RETURN FORM TO QA UPON COMPLETION OF DISPOSITION

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CUSTOMER COMPLAINT FORM
(pg. 2)

QA FOLLOW-UP TO CUSTOMER COMPLAINT

Client Name:		Sample No.(s)	

COMPLAINT:

Contamination		Other:	
Performance			

RETEST PROCEDURE & RESULTS:

Similar Complaints for this Dept?	Yes		No		How Many?	
Date Investigation Completed:				By:		
Date Technical Service Review:				By:		
Conclusions:						
Date:			Laboratory Manager:			

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207-873-7711

Northeast Laboratory Services

EXPRESS **TEST**

Instructions for Collecting Your sample

To collect sample:

One sealed bottle may contain a pill or powder – this is intentional and should not be removed. To take sample, remove strainer, aerator and/or any purification equipment from the faucet before sampling. Allow water to run for 3 to 5 minutes to clear pipes on used wells; ½ to 1 hour on new wells. Fill sample container(s) to shoulder of bottle(s), **allowing about 1/2 inch of airspace between water level and cap**. Take care not to touch rim or inside of cap.

Whenever possible, collect sample from a faucet since collection directly from the well or spring is difficult and usually results in the accidental contamination of the sample. Perform sampling in early afternoon just prior to shipping, or bring sample directly to the laboratory if possible.

Waters From Sources Other Than Wells

Ponds, streams and other bodies of water are usually not tested for total coliform bacteria, since these bacteria are common in soils, vegetation and other environments that are in contact with surface waters. These waters may be tested for other indicator bacteria, such as *E. coli*, *E. coil* 0157, fecal coliforms and enterococci as indicators of various types of contamination and health risks. Typical swimmer suitability testing includes *E. coli* and enterococci for fresh waters and enterococci for marine waters. Please call Customer Service for additional information.

Bringing Samples to the Laboratory

Samples may be brought directly to the laboratory, which is located on the China Road, Route 137, in Winslow. Samples may be dropped off between the hours of 8 AM and 5 PM Mon through Friday. We make every effort to start testing on the same day that samples are received at the laboratory, but please note that samples dropped off after 3:00 PM. may be set up on the following day.

CAUTION: Important Information

Be sure to fill in all requested information on the reverse side of this sheet. **You must include the date and time of sampling**, as results are inaccurate (and invalid) if samples are tested beyond 30 hours of age. If you were required to disinfect your well, be sure that there is **no detectable chlorine odor** in your water before sampling. Samples with detectable chlorine cannot be tested. Perform sampling in early afternoon just prior to shipping, or bring sample directly to the laboratory if possible. Do not ship samples on Saturday.

Disclaimer: NEL will make every effort to adhere to the data reporting times (turnaround times) mentioned on this sheet. However, we are not responsible for delays caused by circumstances beyond our control, such as instrument malfunction. Some samples, such as those with strong color, high turbidity or high concentrations of some metals may take extra time to analyze.

Northeast Laboratory Services
P.O. Box 788, Waterville, ME 04903-0788
1-800-244-8378

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Northeast Laboratory SERVICES

PO Box 786, Waterville, ME 04903-786
227 China Road, Winslow, ME 04901
207/873-7711 1-800-244-6375 Fax: 207/873-7022
508 Riverside Street, Portland, ME 04104
207/878-8481
www.nelabservices.com

DRINKING WATER ANALYSIS

VERY IMPORTANT! Do not collect or ship samples on Saturday, Sunday or Holidays. Samples more than 30 hours old or without collection date and time cannot be tested.
To submit your sample please either (A): Hand deliver to the laboratory located at 227 China Road, Winslow, ME
(B): Hand deliver to the Portland Office located at: 508 Riverside Street
(C): Use the enclosed return label & mail from the nearest U.S. Post Office

LABORATORY USE ONLY

Date Rec'd	Check #
SAMPLE #	

CUSTOMER & WELL INFORMATION

REPORT TO BE SENT TO:					
Name:					
Address:					
City:		State:		Zip:	
Daytime Phone:					

DATE SAMPLED:		TIME SAMPLED:	

WELL OWNER/LOCATION:					
Name:					
Address:					
City:		State:		Zip:	
Daytime Phone:					

PAYMENT INFORMATION (enclose check or complete below to avoid a delay in processing):			
Mastercard / Visa / American Express #			
3 or 4 Digit Security Code from back of card:		Expiration Date:	

ANALYSIS INFORMATION

MORTGAGE APPROVAL TEST
(Conventional Mortgage Test)

Analysis includes:

Arsenic	Lead
Chloride	Manganese
Copper	Nitrate Nitrogen
Fluoride	Nitrite Nitrogen
Hardness	pH
Iron	Sodium

Coliform Bacteria + E. Coli
(Select the box with your preference for reporting Bacteria results):
Negative/Positive Reading ☐ $69.00
Numeric Bacteria Count ☐ $72.00

BASIC SAFETY TEST

Analysis includes:

Nitrate Nitrogen Nitrite Nitrogen

Coliform Bacteria + E. Coli
(Select the box with your preference for reporting Bacteria results):
Negative/Positive Reading ☐ $39.00
Numeric Bacteria Count ☐ $42.00

Other test kits available upon request. Please call the lab for details on the following:

Iron Bacteria	Radon Air / Radon Water
1st Draw Lead	Diesel Range Organics (Fuel Oil)
Mercury	Gasoline Range Organics & VOA 524.2

SAMPLING INSTRUCTIONS

Samples with a detectable odor of bleach will be rejected!
Whenever possible, collect sample from a faucet. Collection directly from the well or spring is difficult and usually results in the accidental contamination of the sample.
Fill all containers to the shoulder. *One sealed bottle may contain a pill or powder - this is intentional and should not be removed.*
To take sample, remove strainer, aerator and/or any purification equipment from the faucet. Allow water to run for 3-5 minutes to clear pipes on used wells; 1/2 to 1 hour on new well. Take care not to touch rim or inside of cap.

WINSLOW OFFICE: Samples received by 3:00 PM Monday thru Friday will have reportable results by 5:00 PM the next business day.
PORTLAND OFFICE: Samples received by 2:00 PM Monday thru Thursday will have reportable results as above. Samples received after 2:00 PM will have reportable results in 2 business days. *No samples are accepted after 2:00 PM on Friday in the Portland Office.*
NEL will make every effort to adhere to this reporting schedule. However, instrument and computer failures may result in unforeseen delays. Some samples, such as those with strong color, high turbidity or high concentrations of some metals may take extra time to analyze. Testing not completed within the guaranteed time frame will warrant a refund to client if not due to interferences listed above. Verbal phone results are discouraged in order to ensure confidentiality of your sample.

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Northeast Laboratory SERVICES

P.O. Box 788 • Waterville, ME 04903-0788
207-873-7711 / Fax: 207-873-7022

Shipping: Rt. 137, China Road
Winslow, Maine 04901

CHAIN OF CUSTODY RECORD

Company Name:

Reporting Address:

Client Contact: **Phone** **Fax**

Billing Name:

Billing Address:

PO #:
Quote #
Project Name
Lab Comments:

Sample Designation	Sample Date/Time	Preservation: 1 H_2SO_4, 2=HNO_3, 3=HCL, 4=NaOH, 5=Thio, 6=4°C, 7=None	Container Size, Type	QTY	Sample Type Code: G = Grab C = Composite	Matrix Code Choose One: CW = Ground Water WW = Waste Water DW = Drinking Water SW = Surface Water O = Oil WP = Wipe S = Soil F = Food Other (Please List)	Analyses Requested	Lab Sample ID

Notes:

Known hazards associated with samples:

Sampled By	Date/Time	Received By	Date/Time	pH	The recommended temperature of the internal container is 2-6°C. Do you authorize analysis if the temp is out of range?
Relinquished By	Date/Time	Received By	Date/Time	Rush Yes 5 days 50% surcharge 2 days 100% surcharge	
Relinquished By	Date/Time	Received By	Date/Time	Field Filtered Y / N	Yes No

Chain of Custody <u>must</u> be filled out completely. This includes signature, sample description, analysis requested, date and time sampled.

<u>If not completed, samples will be rejected.</u>

Rev. 5/01 Form #NEL 100

If a sample is hazardous or radioactive, the sample will be returned to the client at the client's expense.
Standard turn-around-time is 10 business days - depending on season.

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Northeast Laboratory Services

Sample Custody Record

Microbiology Analysis Services

Ship Samples To:
Route 137, China Road
Winslow, Maine 04901

Ph: 1-800-244-8378
Ph: (207) 873-7711
FAX: (207) 873-7022

Lab Use Only

Client

Address

City, State, Zip

Contact

Phone: FAX:

P.O. Number

Project No:

Sample Type Codes

AND = Andersen Plate	BURK = Burkard/Allergenco Slide	S = Surface Swab	Wp = Wipe	FS = Food/Solid
AOC = Air-O-Cell Cassette	RCS = RCS Air Strip	T = Tape Lift	O = Other (describe)	
BS = Bulk Solid	SAS = SAS Plate	W = Water	FL = Food/Liquid	

Sample Information

Lab No.	Sample Identification	Date/Time	Type	Analysis Requested

Flow rates and sampling times (if applicable):

Special Sample Information, Testing or Reporting Instructions:

Sample Temperature Upon Receipt at Laboratory:

Custody Record

Date	Time	Samples Relinquished By	Samples Received By	Comments

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NORTHEAST LABORATORY SERVICES
SAMPLE RECEIPT CONDITION REPORT

Client:_____ **Date/Time Received:**_____ **Intials:**_____

Project:_____ **Rush? Yes No TAT**

NEL Sample Numbers:_____

Delivered by:_____ **Tracking Number:**_____

Cooler _____ **of** _____

	Yes	No	Comment
1. Are custody seals present?	___	___	_____
2. Is the COC present in cooler?	___	___	_____
3. Is the COC properly filled out?	___	___	_____ { } Completed at NEL
4. Is a temp blank present?	___	___	_____
5. Is the temperature of the cooler 4^0C +/- 2^0C?	___	___	Temp:_____ { } NA Collected same day
6. Are ice pacs/ice present in cooler?	___	___	_____
7. Do sample labels match the COC?	___	___	_____
8. Are samples in proper sample containers with proper preservation?*	___	___	_____
9. Are samples within the analytical hold time?	___	___	_____
10. Is there sufficient volume to perform tests?	___	___	_____
11. Are samples in good condition?	___	___	_____
12. Are aqueous VOA/GRO vials free of headspace?	___	___	_____
13. If necessary, has the cooler been screened for radiological activity?	___	___	_____

*See internal COC for preservation verification

Comments:_____

_____

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NORTHEAST LABORATORY SERVICES
SAMPLE RECEIPT CONDITION REPORT

NEL Sample Numbers:_____

Client:_____
Date/Time Received:_____ **Initials:**_____

Delivered by:_____
Tracking Number:_____

Project:_____
Rush? Yes No TAT

FOOD AND WATER

	Yes	No
1. Are custody seals present?	____	____
2. Is the COC properly filled out?	____	____
3. Do sample labels match the COC?	____	____
4. Are samples within the analytical hold time?	____	____
5. Is there sufficient volume to perform tests?	____	____
6. Are samples in good condition?	____	____

Comments:_____

_____

INDOOR AIR QUALITY

	Yes	No
1. Is the COC properly filled out?	____	____
2. Do sample labels match the COC?	____	____
3. Are slides broken or damaged?	____	____

Comments:_____

_____

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NEL INTERNAL CHAIN OF CUSTODY FORM

Date Received: _____ Customer Name: _____ Project: _____ Immediate: _____ Initials: _____

		CONTAINER				LOCATION		IN	OUT	IN	OUT	IN	OUT
LAB ID #	# of Bottles	Type (a)	Size (b)	Preserv-tive (c)	pH	Analyst	Shelf (d)	Date/Time	Date/Time	Date/Time	Date/Time	Date/Time	Date/Time

(a) TYPE OF CONTAINER			(b) CONTAINER SIZE				(c) PRESERVATIVE					(d) SHELF	
P =	G =	Bg =	1 = 1L	3 = 250	5 = 8oz	7 = 100ml	1 = H_2SO_4	3 = HCL	5 = Thio	7 = None		M = Metals	W = Wet Chem
Plastic	Glass	Baggie	2 = 2L	4 = 40ml	6 = 5oz	8 = 5 ml	2 = HNO_3	4 = NaOH	6 = 4°C	8 = Zn/Ac		O = Organics	

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NEL Microbiology Internal Chain of Custody Record

Date Rec'd by NEL	Client Name	Prod. Code	NEL Sample ID	Date Rec'd by Micro	Micro Analyst	Storage Area	Moved to Storage Area/Date	Moved to Storage Area/Date	Moved to Storage Area/Date	Sample sent to:	Rec. Initials	Date/ Analyst

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Document storage locations of all samples. Samples received in the laboratory that remain at ambient temperature shall be documented as "RT".
Samples that require additional testing from another NEL department shall be documented as being sent to that department.

Product Codes: F)Food A) Aqueous S) Swab W) Wipe O) Other

Sample Receipt Report

Sample ID	Sample Description	Project	Matrix	Sampled	Received	Analyte	Due Date	Location	SDG Number
AE03066	LFB001 *qc4 + afcee QC FOR*	x	WATER	4/10/02	04/10/02	DRO in water	5/1/02		MW041002
	LFB001 *qc4 + afcee QC FOR*	x	WATER	4/10/02	04/10/02	TPH Corrected DRO	5/1/02		MW041002
AE03067	LFD001 *qc4 + afcee QC FOR*	x	WATER	4/10/02	04/10/02	CLP Electronic Data	5/1/02		MW041002
	LFD001 *qc4 + afcee QC FOR*	x	WATER	4/10/02	04/10/02	Contract Laboratory	5/1/02		MW041002
	LFD001 *qc4 + afcee QC FOR*	x	WATER	4/10/02	04/10/02	GRO in water	5/1/02		MW041002

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Northeast Laboratory SERVICES
PO. Box 788
Waterville, Maine 04903-0788

Rt. 137, Cline Road
Winslow, Maine 04901

Administrative Offices
Phone: 207-873-7711
Fax: 207-873-7022
Customer Service
Phone: 800-244-6378
Fax: 207-873-7022

SUBJECT:	Sample Acceptance	EFFECTIVE DATE:	June 2007
		APPROVED BY:	Total Quality Manager

NEL SAMPLE ACCEPTANCE POLICY

- **All samples should arrive with proper, full and complete documentation** which includes:
  - Sample identification
  - Location of sample collection
  - Date & time of collection
  - Collector's name
  - Preservation type
  - Sample type
  - Any special remarks concerning the sample
- **Proper sample labeling** must include unique identification on the sample bottle(s)/containers(s) printed with indelible ink or other form of permanent marking.
- **Appropriate sample containers** must be used and obtained from NEL only.
- **Adequate sample volume** must be submitted to perform the necessary tests. All bottles/containers must be filled to the rim except in the case of 40 ml glass VOA vials which should be filled to over-flowing with no air space. If **soil samples** are submitted, please fill all bottles/containers to the rim except in the case of Volatile Organic or Gasoline Range Organics samples where a plug of soil is inserted into a 40 ml VOA vial. Please see attached Terra Core sampler instructions for additional instructions if this sampling is necessary.
- **All samples must be shipped on ice** so that temperature taken at the laboratory, through the use of an enclosed temperature blank, ranges between 0 and 6 degrees Celsius.
- **Frozen samples will not be accepted.** Clients will be called and the lab will proceed with the analyses requested if the client gives his/her permission to do so. In this case, reports and applicable documentation will be noted appropriately.

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REV:	F
PAGE:	1 OF 9

DOCUMENT HISTORY AND APPROVAL PAGE

TITLE:	Standard Operating Procedure for Sample Disposal
DEPARTMENTS AFFECTED:	Analytical Divisions

ISSUE DATE:	REVISION CODE:	SOP #:	REVISION CHANGES:
5/28/02	-	3.7.11	Create a new SOP in NELAC format
5/19/04	A	3.7.11	Revised to account for new procedure
6/13/05	B	3.7.11	Annual Review & Update
4/3/06	C	3.7.11	Annual Review & Update
2/13/07	D	3.7.11	Annual Review & Update
01/10/08	E	3.7.11	Annual Review & Update
03/14/08	F	3.7.11	Update to Section 14.4

DOCUMENT APPROVAL		
Originator: Paul Lynch	Signature/Date: 3/14/08	Title RSO
Approved by: Jim Galasyn	03/14/08	Chemistry Lab Manager
Reviewed by: Pamela Doughty	3/14/08	Total Quality Manager

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Northeast Laboratory Services, Inc. Standard Operating Procedure			
TITLE: Standard Operating Procedure for Sample Disposal			
SOP# 3.7.11	REV# F	EFFECTIVE 03/14/08	PAGE 2 OF 9
WRITTEN BY: Paul Lynch	DEPARTMENTS AFFECTED: Inorganics, Organics, Sample Log in		

1.0 Test Method:

N/A

2.0 Applicable Matrix or Matrices:

All samples.

3.0 Detection Limit:

N/A

4.0 Scope and Application:

This standard operating procedure (SOP) will serve as a guideline for proper disposal of samples that have been analyzed.

5.0 Summary of the Method:

This method will enable personnel to properly dispose of samples that have been analyzed.

6.0 Definitions:

ENU- Elemental Neutralization Unit. The unit used to neutralize caustic and acidic only waste.

LIMS- Laboratory Information System. A database used to store and organize all information concerning samples.

7.0 Interferences:

N/A

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8.0 Safety:

The laboratory is responsible for maintaining a current awareness of OSHA regulations regarding safe handling of chemicals. Please refer to the Northeast Laboratory Chemical Hygiene Plan and Spill Prevention Control and Contingency Plan for further information. While handling radioactive material all personnel are required to wear personal protective equipment that consists of a long sleeve lab coat, safety glasses, gloves, and a dosimetry badge. All personnel are required to practice ALARA as described in SOP # 3.6.6 Radioactive Material Handling, while handling radioactive material.

9.0 Equipment and Supplies:

ENU

10.0 Reagents and Standards:

10.1 Base neutralization solution (ie. 1:1 H2SO4).
10.2 Acid neutralization solution (ie. Sodium Bicarbonate)

11.0 Sample Collection, Preservation, and Storage:

N/A

12.0 Quality Control:

The pH of effluent from the ENU is to be checked periodically using pH paper. This will ensure that the pH does not reach hazardous levels.

13.0 Calibration and Standardization:

N/A

14.0 Procedure:

14.1 Samples are to be disposed of no earlier than thirty days after the report is issued. Clients are made aware of this in writing with the quotation for any particular project. If the client requests so, they will be contacted prior to disposal.

14.2 The LIMS is used to determine samples that are ready for disposal. This is done by a cross-reference search by report date. This data is then exported to a sample disposal log where the disposal date, disposal method, and the initials of the disposer are recorded. The sample disposal log is a spreadsheet stored on the local network. Internal chains of custody are maintained until the sample is disposed of.

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14.2.1 Sample extracts/digests are to be disposed of no earlier than six months after the report is issued. The same procedures for disposal of samples are followed for the disposal of sample extracts/digests.

14.3 After the sample and/or extract/digest has been designated for disposal refer to SOP # 3.7.3 Hazardous Waste Determination to determine them as hazardous or non-hazardous.

14.4 If it is not possible to determine whether or not a samples is hazardous by the analyses already performed, further analysis may be required. In the case of soil or solids, the samples will be composited in 5 gallon buckets. A sample from there will be analyzed to determine its hazardous nature. In the case of water or liquid samples, matrix type (i.e. drinking water vs. waste water) and generator knowledge can be used to determine a sample's hazardous nature. If it is still not possible to determine whether or not the sample is hazardous then it will be composited in a 30 gallon steel drum in the hazardous waste shed. A sample from this drum will then be analyzed to determine its hazardous nature.

14.5 If the sample and/or extract/digest is determined to be hazardous it is to be sent back to the customer. If this is not an option, the sample and/or extract/digest is to labeled as hazardous waste and be placed in the hazardous waste shed for disposal within 90 days by a hazardous waste broker.

14.6 If the sample and/or extract/digest is liquid and non-hazardous or has the hazardous characteristic of corrosivity **only**, it is to be disposed of through the ENU.

14.7 Solid non-hazardous samples are to be disposed of via landfill.

15.0 Data Analysis and Calculations:

N/A

16.0 Method Performance:

N/A

17.0 Pollution Prevention:

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. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the EPA recommends recycling as

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the next best option.

18.0 Data Assessment, Acceptance and Corrective Action:

The following corrective actions should be instituted if deemed appropriate:

1) Potential sources of any problems associated with sample disposal, should be investigated and eliminated prior to beginning the disposal process.

2) A corrective action form should be initiated, if any problems are discovered or encountered, by the individual that discovered the problem. The corrective action form should be filled out by the appropriate personnel with documentation of the procedures to minimize reoccurrence of the problem. Finalized corrective action forms are filed with the laboratory quality assurance manager.

3) The Health and Safety Manager should be contacted immediately if a problem is encountered while disposing of samples.

19.0 Waste Management:

Any and all waste generated, through any procedure in the laboratory, is handled according to the Chemical Hygiene, Hazardous Waste Management and Spill Prevention Control and Contingency Plans of Northeast Laboratory Services, Inc.

20.0 References:

NELAC Quality Systems Chapter 5, June 5, 2003

21.0 Tables, Diagrams, Flowcharts and Validation Data

Hazardous Waste Label
Sample disposal log
Cross Reference Search Results
Analysis Results

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HAZARDOUS WASTE

FEDERAL LAWS PROHIBIT IMPROPER DISPOSAL

IF FOUND, CONTACT THE NEAREST POLICE OR PUBLIC SAFETY AUTHORITY OR THE U.S. ENVIRONMENTAL PROTECTION AGENCY

GENERATOR INFORMATION:

NAME _____

ADDRESS _____

CITY _____ STATE _____ ZIP _____

EPA ID NO. _____ EPA WASTE NO. _____

ACCUMULATION START DATE _____ MANIFEST DOCUMENT NO. _____

[_____]

D.O.T. PROPER SHIPPING NAME AND UN OR NA NO. WITH PREFIX

HANDLE WITH CARE!

Lab Safety Supply Inc., Janesville WI 53547-1368 Reorder No. 433

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Sample Disposal Log

Disposal Methods
1 Sanitary District (Elemental Neutralization)
2 Composited Solid Sample (samples that do not exhibit any hazardous properties from the requested analysis)
3 Sent back to customer
4 Disposed of through Hazardous Waste Broker
5 Sample Consumed During Analysis

Report Date	Sample ID	Client	Submittal Date	Sample Description	Sample Disposal Date	Extract/Digest Disposal Date	Disposal Method	Disposer
4/1/04	AG01841	Yankee Atomic Electric Co-Rowe	3/16/04	MW 104B	Hold			
4/1/04	AG01984	Hampden Water Dist	3/22/04	Mary Parsons	5/5/04		1	PL
4/1/04	AG01985	Hampden Water Dist	3/22/04	Rudolph Butterworth	5/5/04		1	PL
4/1/04	AG01986	Hampden Water Dist	3/22/04	Lisa Gadoury	5/5/04		1	PL
4/1/04	AG01987	Hampden Water Dist	3/22/04	Anthony Gilberti	5/5/04		1	PL
4/1/04	AG01988	Hampden Water Dist	3/22/04	Charles Hamel	5/5/04		1	PL
4/1/04	AG01989	Hampden Water Dist	3/22/04	Troy Bailey	5/5/04		1	PL
4/1/04	AG01990	Hampden Water Dist	3/22/04	Tim Scott/Deb Scott	5/5/04		1	PL
4/1/04	AG01991	Hampden Water Dist	3/22/04	Dana Tardiff	5/5/04		1	PL
4/1/04	AG01992	Hampden Water Dist	3/22/04	Edward Gardella/Michael Li	5/5/04		1	PL
4/1/04	AG01993	Hampden Water Dist	3/22/04	Aniko Fulep/Miklos Simon	5/5/04		1	PL
4/1/04	AG02296	Woodard & Curran Inc.-Portland	3/31/04	WMT03HS02	5/5/04		1	PL
4/1/04	AG02297	Woodard & Curran Inc.-Portland	3/31/04	WMT03HS03	5/5/04		1	PL
4/1/04	AG02298	Woodard & Curran Inc.-Portland	3/31/04	WMT03HS07	5/5/04		1	PL
4/1/04	AG02299	Woodard & Curran Inc.-Portland	3/31/04	WMT03MW11	5/5/04		1	PL
4/1/04	AG02300	Woodard & Curran Inc.-Portland	3/31/04	WMT03HS06	5/5/04		1	PL
4/1/04	AG02301	Woodard & Curran Inc.-Portland	3/31/04	WMT03HS05	5/5/04		1	PL
4/1/04	AG02302	Woodard & Curran Inc.-Portland	3/31/04	WMT03HS04	5/5/04		1	PL
4/1/04	AG02303	Woodard & Curran Inc.-Portland	3/31/04	WMT03HS04	5/5/04		1	PL
4/1/04	AG02304	Woodard & Curran Inc.-Portland	3/31/04	WMT03HSDW	5/5/04		1	PL
4/1/04	AG02305	Woodard & Curran Inc.-Portland	3/31/04	WMT03WHLBLK	5/5/04		1	PL
4/1/04	AG02306	Woodard & Curran Inc.-Portland	3/31/04	WMT03HSBL2	5/5/04		1	PL
4/2/04	AG02140	Waste Water Services Corp	3/25/04	MSAD 47	5/5/04		1	PL
4/2/04	AG02141	Waste Water Services Corp	3/25/04	Goodwill	5/5/04		1	PL
4/2/04	AG02142	Waste Water Services Corp	3/25/04	D6-001	5/5/04		1	PL
4/2/04	AG02168	J M C Wastewater Services	3/26/04	WCCCC Effluent	5/5/04		1	PL
4/2/04	AG02169	Penobscot Frozen	3/26/04	Final Effluent	5/5/04		1	PL
4/2/04	AG02170	Penobscot Frozen	3/26/04	Aeration	5/5/04		1	PL

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Sample Cross Reference Search Results

Northeast Laboratory Services
User: Lynch, Paul
Date: 05/20/2004 Time: 09:31:39

Search routine: **REPDATE**
Search performed: **05/20/2004 at 09:30**
Samples found: **22**

No.	Rep Date	Sample ID	SampleDesc	OtherComment	SBT Number	Sub Date	Sub Time
1	04/01/2004	AG01841	MW 104B	QC3	250125	03/16/2004	10:15
2	04/01/2004	AG01984	Mary Parsons		080114	03/22/2004	10:45
3	04/01/2004	AG01985	Rudolph Butterworth		080114	03/22/2004	10:45
4	04/01/2004	AG01986	Lisa Gadoury		080114	03/22/2004	10:45
5	04/01/2004	AG01987	Anthony Gilberti		080114	03/22/2004	10:45
6	04/01/2004	AG01988	Charles Hamel		080114	03/22/2004	10:45
7	04/01/2004	AG01989	Troy Bailey		080114	03/22/2004	10:45
8	04/01/2004	AG01990	Tim Scott/Deb Scott		080114	03/22/2004	10:45
9	04/01/2004	AG01991	Dana Tardiff		080114	03/22/2004	10:45
10	04/01/2004	AG01992	Edward Gardella/Michael Little		080114	03/22/2004	10:45
11	04/01/2004	AG01993	Aniko Fulep/Miklos Simon		080114	03/22/2004	10:45
12	04/01/2004	AG02296	WMT03HS02	qc2	2848	03/31/2004	16:55
13	04/01/2004	AG02297	WMT03HS03	qc2	2848	03/31/2004	16:55
14	04/01/2004	AG02298	WMT03HS07	qc2	2848	03/31/2004	16:55
15	04/01/2004	AG02299	WMT03MW11	qc2	2848	03/31/2004	16:55
16	04/01/2004	AG02300	WMT03HS06	qc2	2848	03/31/2004	16:55
17	04/01/2004	AG02301	WMT03HS05	qc2	2848	03/31/2004	16:55
18	04/01/2004	AG02302	WMT03HS04	qc2	2848	03/31/2004	16:55
19	04/01/2004	AG02303	WMT03HS04	qc2	2848	03/31/2004	16:55
20	04/01/2004	AG02304	WMT03HSDW	qc2	2848	03/31/2004	16:55
21	04/01/2004	AG02305	WMT03WHLBLK	qc2	2848	03/31/2004	16:55
22	04/01/2004	AG02306	WMT03HSBL2	qc2	2848	03/31/2004	16:55

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User: Lynch, Paul
Northeast Laboratory Services
Date: 05/20/2004 Time: 09:31

Sample: AG01984 SBT Number: 080114 Clientname: Hampden Water Dist LoginBatch: 03221153 Status: Inactive
SampleDesc: Mary Parsons ProjectNumber: MD01954-93
Collected: 03/08/2004 06:00 Submitted: 03/22/2004 10:45 Sample due date: 04/05/2004
Collector: --- Matrix: D-WATER PAM: ---
Page: 1

Anl Code	Analysis Name	Units	Result	Violation	Value	Sec Result	Qualifi
CU2007	Copper Total	mg/L	0.097	---	---	---	---
PB2009	Lead, Total	mg/L	0.0042	---	---	---	---
200MNC	Metals Sample Prep for DW and WW		Complete	---	---	---	---

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Northeast Laboratory Services

QUALITY ASSURANCE PROGRAM MANUAL

AND

Statement of Qualifications

STANDARD OPERATING PROCEDURES

April 2008

Revision 21

This manual is approved by the following approved signatories of NEL:

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This document pertains primarily to the analytical chemistry and microbiology departments of NEL.

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STATEMENT OF QUALIFICATIONS & QUALITY ASSURANCE PROGRAM MANUAL

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Northeast Laboratory SERVICES

GENERAL OVERVIEW

Northeast Laboratory Services is located on the China Road in Winslow, Maine. A location at 508 Riverside Drive, Portland, Maine is open for business and will primarily be used for the receipt of samples that will be delivered to the Winslow facility for analysis. The business was founded in 1972 as Maine Poultry Consultants and later became Maine Poultry Consultants/Northeast Laboratory Services. After a decline in the poultry business in Maine, causing the closure of Maine Poultry Consultants, the business served its environmental and microbiological products clients under the name of Northeast Laboratory Services (NEL) for the next several years. In 1997, NEL merged with Binax, Inc. a Portland-based research and development firm. Binax/NEL operated as a separate entity to the Portland facility, complete with it's own management team and accounting functions. In April of 2001, the Laboratory was purchased back by it's original founder, Dr. Kenneth Eskelund who will act as a consultant to the business and Corporate Treasurer and Mr. Rodney "Beau" Mears, NEL CEO and President. Although Mr. Mears is in charge of both divisions, there are managers present in all sections who ultimately make the decisions for their functional groups, eliminating the potential for conflicts of interest.

The business units of NEL are departmentalized into three (3) areas:

- Microbiological Products (Media Manufacturer)
- Environmental Analytical Services (Chemistry)
- Microbiology Services (Microbiology)

The Microbiological Products Department is an FDA registered in-vitro diagnostic manufacturer and provides approximately half of revenues for NEL. Approximately twenty-eight (20) full time employees are in this department.

The market segment that generates sales is approximately 75% from industrial (pharmaceutical, biotechnology, food and government) and 25% clinical (hospital, physicians). The product lines include custom and preparatory media as well as standardized microbiological products.

In the industrial market, NEL is committed to work directly with food manufacturers, cosmetic companies, pharmaceutical companies and the biotechnology industry offering custom products to meet their specific microbiology needs.

The service sector (chemistry and microbiology) produces the other 50% of the sales revenue and employs approximately twenty-two (22) full-time individuals.

To reach our customer service representatives and place orders for prepared media, please dial 207/873-7711extension 304 or 305.

Environmental Services includes four (4) distinct units: wet or general chemistries, inorganic, organic and radiological chemistry.

The analytical chemistry laboratory performs a variety of analyses for private clients as well as government agencies. NEL is committed to providing analytical testing services and generating scientifically valid data of known and documented quality. It is the laboratory's policy to conduct all projects in a timely manner using USEPA, FDA, ASTM, AOAC and other Standard Reference Methods as well as state of the art technology/instrumentation. With the increase in decommissioning of nuclear power plants throughout the United States, NEL has become increasingly active in the analyses of radioactive materials, producing results for several large nuclear power plants such as Yankee Rowe, CT., Yankee, Big Rock, Fermi 2 of Michigan, Millstone, CT., and Maine Yankee.

Jim Galasyn, Chemistry Lab Manager, can be reached at extension 337.

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The Microbiology Services includes two (2) units, IAQ Microbiology and Food Microbiology.

The Microbiology Services Unit routinely analyzes air, water and soil samples for a variety of microorganisms and for a wide range of industries.

Drinking water is assayed for coliform bacteria and E. coli as indicators of surface water and septic, respectively, intrusion into drinking water systems. Public bathing beaches and pools are tested for indicators of fecal contamination and potential infectious agents. Sludges and composts are assayed for clients primarily in the field of wastewater treatment. Assays for molds and bacteria in air (bioaerosols) and in building materials are conducted to assess and control exposure to workers and products.

Increased emphasis on the safety of the food supply is spawning an increase in food testing. Food manufacturers are required to monitor and control the manufacturing environment. NEL Microbiology Laboratory offers unique support serves and improved test turn-around-times for food manufacturers.

Brett Goodrich is the Manager of the Microbiology Laboratory and can be reached at extension 321.

Other key personnel are listed below:

Employee	Position	873-7711/Ext
Francis Reed	IT Manager	329
Melissa Higgins	Director of Finance	336
Vickie Massey	Human Resource Manager	311
Lauraly LeBrasseur	Quality Assurance Manager, Media Div.	317
Eva Chase	Media & Production Manager, Media Div.	318
Paul Lynch	Radiation Haz Mat Manager	341
Pamela Doughty	Total Quality Manager	310
Vanessa Vigue	Food Microbiology Supervisor/Project Manager	342
Jeremy Boutin	Materials Handling Supervisor	303
Bill Sargent	Manager, Portland Facility	878-6481

Northeast Laboratory Services welcomes all old and new clients and is anxious to service all of your analytical requirements as well as supply the medical and biotechnology communities with their microbiological needs. We are proud of our products and services and we believe you, as one of our customers, will be equally proud. Our desire is to serve all of our customers faithfully. More is humanly possible; less, we will not tolerate.

We can be reached from 8:00 AM to 5:00 PM EST at (207)873-7711, toll free 1-800-244-8378 or via fax at (207)873-7022. We look forward to servicing your microbiological prepared media and/or environmental analysis needs. Our President and CEO, Beau Mears, is also available for your calls and can be reached personally at extension 331.

Please visit us online @ www.nelabservices.com

Thank you for your interest in NEL.

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CHAPTER 1

LABORATORY QUALITY ASSURANCE PROGRAM INTRODUCTION

1.1.0 INTRODUCTION

1.1.1 The analytical chemistry and microbiology laboratory performs a variety of analyses for private clients as well as government agencies. Each client may have different analytical requirements with respect to data quality objectives and intended use of data. Northeast Laboratory Services (here after referred to as NEL) QA/QC program was developed to establish and maintain a high standard of performance consistently applied to all analyses performed.

1.1.2 This Quality Assurance/Quality Control (QA/QC) Manual documents NEL facility, personnel, policies, practices and analytical procedures established in order to meet the requirements of the National Environmental Laboratory Accreditation Conference (NELAC) and the International Organization of Standardization (ISO) policies, ISO/IEC 17025 Standards in conjunction with the requirements of our clients. NEL is a privately owned company with one location on the China Road in Winslow, Maine (mailing address PO Box 788, Waterville, ME 04903-0788). The telephone and fax numbers are (207) 873-7711 and (207) 873-7022 respectively. The e-mail address is pdoughty@nelabservices.com. The NEL website address is www.nelabservices.com. NEL's laboratory is open to the public from 8:00 AM to 5:00 PM eastern standard time, Monday through Friday. NEL is open on Saturdays to receive samples.

NEL is certified in Maine, New Hampshire, Vermont, Connecticut, Massachusetts, Rhode Island, New York, Virginia, Utah, Florida, Colorado, Michigan and pending in Arizona. NEL is also certified for work with the United States Navy. NEL is also certified for food microbiology by the American Association for Laboratory Accreditation (A2LA) and for indoor air quality by the American Industrial Hygiene Association (AIHA). Please see the attachment to this LQAP Chapter for additional information on NEL's certifications and accredidations. The Laboratory is a State of the Art facility with modern analytical instrumentation and a professional staff of highly skilled analytical chemists, microbiologists and technicians. We extend an open invitation to visit our facility.

1.2.0 QUALITY ASSURANCE POLICY STATEMENT

NEL is committed to providing analytical testing services, and generating scientifically valid data of known and documented quality. It is the laboratory's policy to conduct all projects in a timely manner using USEPA, FDA, ASTM, AOAC, and other Standard Reference Methods as well as state of the art technology/instrumentation. To achieve this end this document was prepared and a copy distributed to each section of the Laboratory.

1.3.0 MANAGEMENT QUALITY POLICY

1.3.1 The owners and management of NEL are dedicated to the adherence of this Quality Assurance Manual. This manual is designed to provide "quality data" and represents the management's requirements for the laboratory's standard of service as it relates to quality. Deviations substandard from this manual will result in employee dismissal. Any data manipulation to misrepresent quality control will be considered fraud and will result in employee dismissal. The following practices are not tolerated at NEL and will result in termination:

1) Reporting the analysis date incorrectly to meet holding time.
2) Manual integration of chromatography without justification in order to meet QC requirements.
3) Modifying a regulated method without client permission to cut costs or save time.
4) Falsifying data.
5) Conduct. While NEL may use oral warnings, written warnings and suspensions, as well as discharge for violation of the Rules of Conduct or NEL policies, it is not restricted to using

Initiated by: Total Quality Manager

Approved By: Pamela Doughty
Total Quality Manager

Approved By: [illegible]
Chemistry Lab Manager

Approved By: [illegible]
Microbiology Manager

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progressive discipline. In each case, NEL will select the form of discipline that it believes is justified in the circumstances.

Every disciplinary action will be discussed with the employee and a record of the discipline will be made a part of the employee's personnel file.

NEL reserves the right to discipline employees for violation of the following Rules of Conduct or for violation of other written NEL policies:

- Reporting to work under the influence of drugs, alcohol or other intoxicants, or the use or possession of drugs, alcohol or other intoxicants during the work day, except in each case where the use of such drugs or intoxicants has been prescribed by a physician and does not prevent the employee from performing the full scope of his or her duties. The employee is responsible for informing his or her supervisor of this situation.
- No employee at any work site will possess any quantity of any unlawful controlled substance. "Work site" means any NEL motor vehicle, office, building, yard or any other location at which the employee is to perform work. "Possess" means to have either in or on the employee's person, personal effect, NEL motor vehicle or areas substantially entrusted to the control of the employee.
- Dishonesty with NEL or its customers.
- Theft of NEL property or personal property of others, or concealing information about thefts.
- Negligence in the performance of work.
- Unauthorized absence from work or workstation.
- Violation of NEL safety rules and regulations.
- Fighting or horseplay or causing intentional injury to another associate.
- Damage to NEL property or reputation.
- Removal of NEL property without authorization of your supervisor.
- Unauthorized possession of dangerous weapons.
- Arrest and conviction of serious criminal or civil offenses.
- Falsification of time records.
- Falsification of employment application.
- Insubordination after a written warning by a supervisor.

All analytical employees are required to read and sign off that they understand what is contained in this LQAP including the ramifications for partaking in any of the above practices. Laboratory fraud is clearly described with the statement that "these practices are not acceptable at NEL and will result in your termination".

1.3.2 Through the use of ethics and data integrity training, NEL will ensure that all analytical employees are aware of the relevance and importance of their activities and how each one contributes to the achievement of the objectives of the management system. As stated above, NEL has a stringent policy on laboratory fraud.

1.4.0 This Laboratory Quality Assurance Program Manual (LQAP) describes analytical activities having a direct or indirect effect on data quality. The LQAP manual shall be updated whenever necessary and at least annually reviewed and updated by the Quality Assurance Manager with input from all technical managers. NEL defines quality data as follows: "Quality data are data produced from regulatory accepted methods and quality control that meet or exceed our clients' data quality objectives and are reported correctly and on time". This document only provides the routine and minimum quality control practices at NEL.

1.4.1 As a minimum requirement there shall be LQAPs for the following critical topics:

* Quality Assurance Policy
* Staff Responsibilities, Organization and Training
* Data Quality Objectives (DQOs)
* Sample Preservation Procedures, Holding times & Amounts Needed for Aqueous & Solid Samples
* Sample Handling and Transmittal Procedures
* Analytical Procedures, and Quality Control Samples
* Data Verification, Review, and Reports
* Corrective Action Procedures
* Performance and System Audits

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* Instrument and Equipment Maintenance
* Good Laboratory Practices, and Documentation Procedures

1.5.0 Standard Operating Procedures (SOP's)

Standard Operating Procedures are documents used to identify the operations and QA/QC requirements to be followed for specific analysis. The following SOP's have been developed by the Laboratory Analytical Staff:

* Wet Chemistry SOP's
* Inorganic SOP's
* Organic SOP's
* Environmental Microbiology SOP's
* Radiation SOP's
* Quality Assurance SOP's
* Procedural SOP's
* Sample Log-In SOP's

All SOP's must contain the method reference, effective date, and revision number. Any revisions to laboratory procedures should be written, dated, and distributed to all affected individuals to ensure implementation of changes. The Quality Assurance Manager will be sure that the current version of each SOP is on file in the Master Book and a copy is in each applicable section of the lab. Expired SOP's must be maintained for a period of three years.

1.6.0 Quality Assurance Project Plans (QAPP)

The Client/Customer specific requirements shall dictate the basis of which QAPPs will be initiated. These plans may supersede specific requirements identified in the Laboratory's analysis specific SOP's.

1.7.0 Clients that request a copy of this manual for a specific project or laboratory introduction will receive an uncontrolled copy. Clients that require an on-going relationship with NEL and must be aware of any changes to the QA/QC manual will receive a controlled copy. Holders of uncontrolled copies will not receive revisions to this manual but controlled copyholders will. We strongly recommend the request for an uncontrolled copy unless absolutely required to have the very latest revision.

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Northeast Laboratory SERVICES

STATE CERTIFICATIONS
CHEMISTRY & ENVIRONMENTAL MICROBIOLOGY DIVISIONS

State	Contact	Address	Certification Number	Date of Expiration	Matrix				
					Waste Water	Drinking Water	Ground Water	Solid Waste	Other
Arizona	Arizona Department of Health Services	State Laboratory Services Office of Laboratory Licensure and Certification 1740 W. Adams, Suite 203 N Phoenix, Arizona 85007	AZ0711	10/29/08		X			Micro Only
Colorado	Ken Johnson	Colorado Department of Public Health and Environment/Lab Services Division/Pending Certification 8100 Lowry Boulevard Denver, Colorado 802030-6928	N/A	06/30/08		X			Chemistry only
Connecticut	Mr. Jeffrey Curran, Supervising Environmental Laboratory Consultant	Environmental Laboratory Certification Division of Environmental Health, MS#51Lab 410 Capitol Avenue PO Box 340308 Hartford, CT 06134-0308	PH-0264	06/30/08	X	X	X	X	
Florida	Mr. Stephen Arms	Florida Dept. of Health Bureau of Laboratories PO Box 210 Jacksonville, FL 32231	E87820	06/30/08	X	X	X	X	
Maine	Mr. Matthew Sica	State of Maine Division of Health Engineering, Bureau of Health Department of Health and Human Services 11 State House Station 286 Water Street, 3rd Floor Key Plaza Augusta, ME 04333	ME009	10/04/08	X	X			
Massachusetts	Ms. Anne Marie Allen	Laboratory Approval Program Massachusetts DEP Lawrence Experiment Station 37 Shattuck Street Lawrence, MA 01843	M-ME009	06/30/08	X	X			Chemistry accredited – Micro pending
Michigan	Mr. George L. Krisztian Certification Officer	DEQ Environmental Science & Services Division Michigan Department of Environmental Quality Laboratory Services Section, Bldg 44, 3rd Floor 3350 N. Martin Luther King Jr. Blvd. Lansing, MI 48906-2933	9965	06/30/08		X'			Organic & Inorganic Chemistry
New Hampshire	Mr. William Hall	State of New Hampshire Dept. of Environmental Services 29 Hazen Dr., PO Box 95 Concord, NH 03302-0095	253403	12/07/08	X	X			
New Jersey	Amy Bowman Certification Officer	State of New Jersey Department of Environmental Protection Office of Quality Assurance 9 Ewing St., 2nd Flr, P.O. Box 424 Trenton, NJ 08625	ME003	6/30/08		X			Micro Only
New York	Ms. Joyce Reilly	New York State Dept. of Health Wadsworth Center Environmental Laboratory Approval Program (ELAP) PO Box 509 Albany, NY 12201-0509	11786	4/01/08	X	X	X	X	
Pennsylvania	Stacy Howerter, Certificaiton Officer	Commonwealth of Pennsylvania Department of Environmental Protection Bureau of Laboratories P.O. Box 1487 Harrisburg, PA 17105-1467	68-04295	06/30/08		X			Micro Only
Rhode Island	Dr. Henry Leibovitz, Ph.D.	State of Rhode Island and Providence Plantations Rhode island Dept. of Health Laboratory 50 Orms Street Providence, RI 02904	00272	12/30/08	X	X			

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Texas	Frank Jamison	Texas Commission on Environmental Quality P.O. Box 13087 Austin, TX 78711-3087	T104704288-08-TX	10/31/08		X			Micro Only
Utah		Bureau of Laboratory Improvement Environmental Laboratory Certification Program 46 North Medical Drive Salt Lake City, Utah 84113-1105	NELS	06/30/08	X	X	X	X	
Vermont	George Mills	State of Vermont Department of Health Drinking Water Certification-Public Health Laboratory 195 Colchester Ave. P. O. Box 1125 Burlington, Vt 05402-1125	VT 87820	2/11/08		X			
Virginia	R. Tracy Hunter	Commonwealth of Virginia Division of Consolidated Laboratory Services 600 North 5th Street Richmond, Virginia 23219-3691	00046	6/08		X			Chemistry only
A2LA	Mr. Randy Querry	The Amercian Association for Laboratory Accreditation 5301 Buckeystown Pike, Suite 350 Frederick, MD 21704-8373	2371.01	10/31/08					Food Micro
U.S. Navy	Ms. Patricia Moreno	Department of the Navy Naval Facilities Eng. Service Center 1100 23rd Ave Port Hueneme, CA 93043-4370	N/A	08/30/08			X	X	

NEL PROFESSIONAL AFFILIATIONS/CERTIFICATIONS/COMPLIANCE TRAINING

Division	Registrations/Affiliations	Description	
Media	FDA	In-Vitro Diagnostic Device Manufacturer	
Media	USDA Approved	Manufacturing Facility	
Media	IBWA Membership		
Media	NACMID		
Media	Independent Laboratory Distributors Assoc. (ILDA)		
Microbiology	A2LA	Food Microbiology	
Microbiology	AIHA	Indoor Air Quality	

Division	Professional Affiliations/Certifications/Compliance Trained	Employee
Media QA/QC	Registered Medical Laboratory Technologist-American Medical Technology Assoc.	Lauraly LeBrasseur
Media QA/QC	Registered Medical Laboratory Technician – American Society of Clinical Pathologists	Cindy Kennard
Maintenance	State of Maine Licensed Stationary Steam Engine 4th Class	Andrew Davidson
Maintenance	State of Maine Licensed Plumber	Robert Leathers
Maintenance	State of Maine Licensed Boiler Operators Permit	Robert Leathers
Shipping/Receiving/Sampling	IATA HAZMAT Shipping Certified	Jeremy Boutin
Shipping/Receiving/Sampling	US DOT Shipping Certified	Jeremy Boutin
Shipping/Receiving/Sampling	Confined Space Entry Certified	Jeremy Boutin
Shipping/Receiving/Sampling	Forklift Safety Trained	Jeremy Boutin/Blaine Bouchard
Shipping/Receiving/Sampling	1910 General Industry Occupational Safety Compliant Trained	Dale Mullen
Shipping/Receiving/Sampling	RCRA Hazard Waste Trained	Dale Mullen
Shipping/Receiving/Sampling	Merc Trained	Dale Mullen
Chemistry	American Chemical Society Member #00817500	Dr. James Galasyn

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CHAPTER 2

STAFF RESPONSIBILITIES, ORGANIZATION AND TRAINING

2.1.0 Organization

This (LQAP) standard document provides information on management and staff responsibilities, organization and the laboratory training program. The owners of the laboratory are Mr. Rodney "Beau" Mears, President & CEO and Dr. Kenneth Eskelund, Consultant and Corporate Treasurer. The owners of the corporation oversee the financial stability of the company and make the final decision on company direction, major purchases, laboratory staffing and cost analysis. Additionally, the Laboratory Managers report to Mr. Mears regularly as does the Total Quality Manager. Mr. Mears is also responsible for all major contractual agreements with major clients and approves all project quotations.

Dr. James Galasyn is the Chemistry Laboratory Manager. Dr. Galasyn is responsible for the daily activities of the laboratory including QC, analyses, technical direction, employee assignments and training. All supervisors report directly to Dr. Galasyn. He is the primary contact for non-routine client questions and requests. Ms. Pamela Doughty is the deputy appointed in Dr. Galasyn's absence.

Ms. Pamela Doughty is the Total Quality Manager. Ms. Doughty is responsible for NEL's Total Quality Management program (TQM). Allison Hurlburt is the deputy appointed in Ms. Doughty's absence.

Mr. Paul Lynch is the Radiation Safety Manager and is the primary contact for client requests for sampling services.

Mr. Brett Goodrich is the Microbiology Manager. He is the primary contact for any food micro and IAQ Micro inquiries. Vanessa Vigue is the deputy appointed in Mr. Goodrich's absence.

For more information on job descriptions, please see the job descriptions provided in this document.

2.2.0 Environmental Laboratory Staff Responsibilities

2.2.1 Quality Assurance Department

The quality assurance functions conducted by the department managers and/or quality assurance officer occur at several levels.

The QA responsibilities are to include but may not be limited to the following:

- Update the Laboratory Quality Assurance Program Manuals (LQAPs).
- Coordinate SOP preparation with the appropriate Staff Technicians/Chemists.
- Coordinate reproduction/review and revision of all SOP's.
- Verify, monitor and assure compliance with SOP's.
- Define the critical job elements for employees with respect to quality and workmanship, quantity, and safety.
- Initiate the preparation of and/or updating of all staff resumes/training records ensuring that information is current and up-to-date.
- Validate data prior to submittal to clients as signified by initials on the raw data.
- Oversight of corrective actions.
- Oversight of PE analysis.
- Report to management.
- Internal audits.
- Procurement Q.A.

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2.2.2 Staff Responsibilities

2.2.2.1 Minimum Training Requirements

Laboratory Personnel shall be qualified to perform all analysis assigned. Staff who perform analytical demonstrations of capabilities must be on file prior to the employee being allowed to perform the task unsupervised. Ethical and legal training will be performed annually in conjunction with annual safety and sexual harassment training which includes a discussion of conflict of interest.

2.2.2.2 Data Integrity Training

Data integrity training shall be provided as a formal part of new employee orientation and must also be provided on an annual basis for all current employees. Topics covered shall be documented in writing and provided to all trainees. Key topics covered during training must include organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting, how and when to report data integrity issues, and record keeping. Training shall include discussion regarding all data integrity procedures, data integrity training documentation, in-depth data monitoring and data integrity procedure documentation. Employees are required to understand that any infractions of the laboratory data integrity procedures will result in a detailed investigation that could lead to very serious consequences including immediate termination, debarment or civil/criminal prosecution. The initial data integrity training and the annual refresher training shall have a signature attendance sheet or other form of documentation that demonstrates all staff have participated and understand their obligations related to data integrity. Laboratory managers acknowledge their support of these procedures by 1) upholding the spirit and intent of NEL's data integrity procedures and 2) effectively implementing the specific requirements of the procedures.

Specific examples of breaches of ethical behavior should be discussed including improper data manipulations, adjustments of instrument time clocks, and inappropriate changes in concentrations of standards. Data integrity training requires emphasis on the importance of proper written narration on the part of the analyst with respect to those cases where analytical data may be useful, but are in one sense or another partially deficient. The data integrity procedures may also include written ethics agreements, examples of improper practices, examples of improper chromatographic manipulations, requirements for external ethics program training, and any external resources available to employees. For a list of specific breaches of ethical behavior that are not tolerated at NEL please see Chapter 1, Section 1.3.1.

2.2.2.3 Preliminary Analyst Evaluation

Each new analyst must perform and document an initial one time demonstration of the ability to generate acceptable data (precise and accurate) as defined in the appropriate standard reference method through the replicate analysis of quality control check standards.

Microbiology staff shall be evaluated and approved for specific methods by performing such methods under the direct observation of the supervisor or designated training analyst. The trainer shall ensure that the analyst is capable of independently performing the analysis and QC protocols outlined in the Method SOP.

2.2.2.4 On-going QC Check

Periodically staff shall analyze QC check standards of known concentrations in order to verify calibrations/system control and proper analytical technique. The frequency requirements for QC check analyses shall be defined in the appropriate SOP's. Staff shall be required to participate in external proficiency samples provided by a NIST certified provider such as Environmental Resource Associates (ERA) for non-potable, potable and solid proficiency samples for their parameters of responsibility. Evidence is on file in each employee's training file that proficiencies are being performed.

2.3.0 Responsibility for Compliance

2.3.1 General QC Compliance

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The analytical Laboratory staff shall conduct their analytical tasks in accordance with the appropriate standard reference method and the Laboratory Quality Assurance Program Manual. Evidence is on file that each employee has read, understood and is using the latest version of the lab's quality manual and SOP which relates to his/her job responsibilities.

2.3.1.1 For microbiology laboratory, managers shall designate an analyst for data review in the event of managerial absence. This analyst shall be selected based on factors such as past experience/credentials and/or overall performance in their current position.

2.3.2 Proposal/Project Compliance

For proposals requiring site specific Quality Assurance Project Plans, the QA manager and laboratory manager shall review the original QAPP requirements and if necessary prepare or initiate QAPPs.

2.4.0 Training Techniques

Training techniques may include one or a combination of the following:

* Training courses/seminars conducted by an accredited university, technical school or by an instrument manufacturer.
* Internal/On-the-job training consisting of:

1. Reading and studying the Method and SOP.

2. Instruction from an experienced analyst with continued supervision/observation from an analyst experienced in the procedure.

3. Semi-independent performance of the analyses by the trainee using check samples or equivalent. This proficiency will be documented by performing acceptable MDL or precision/accuracy study.

4. Only after a period of time to be determined by the technical manager of the analysis, and documentation of proficiency shall the staff trainee then be allowed to perform the analysis on their own.

5. Microbiology staff will follow these guidelines except that proficiency is not based on the use of check samples. Instead, the analyst shall independently carry out the method under the direct observation of a qualified (trained) analyst. The trainee shall comply with all analytical and QC protocols defined in the Method SOP.

2.5.0 Documenting Staff Credentials

For projects having minimum experience and training requirements of staff handling the samples, the staff members involved shall provide certification, diplomas, or training certificates to the Total Quality Manager. Please see attached job descriptions and resumes of key personnel as well as a copy of NEL's signatures and initials identification page.

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Northeast Laboratory Services
Employee Signatures/Initials

Dept	Last Name	First Name	Signature
Chem	Anthony	Kelsey	
Sample Rm	Bawcum	Mary	
Chem	Bishop	Tim	
Shipping	Boudreau	Blaine	
Admin	Boudreau	Laurie	
Shipping	Boutin	Jeremy	
Chem	Cassin	Joseph	
Micro	Chodham	Thomas	
Chem	Detosby	Robert	
Sample Rm	Doughty	Pamela	
Micro	Dudley	Brooke	
Chem	Fenwick	Melissa	
Chem	Fitzheaset	Amy	
Shipping	Frost	Shelly	
Chem	Galassin	James	
Admin	Greenwood	Richard	
QA	Hurlburt	Allison	
Micro	Kyes	Amanda	
Chem	LaFlamme	Valentina	
Chem	Landry	Katrzyna	
Chem	Lynch	Paul	
Chem	McCaw	Andrew	
Admin	Mears	Sandy	
Chem	Mullen	Dale	
Chem	Mullen	Deborah	
Sample Rm	Nunnny	Angela	
Chem	Parker	Jacob	
Chem	Paternaude	Abram	
Admin	Sargent	William	
Micro	Schaeffer	Matthew	
Chem	Sheinnagan	Lanaki	
Micro	Vigue	Vanessa	

Chem & Micro Signatures

UNCONTROLLED

NEL PHONE LIST

Beau Mears	**331**
cell:	756-5678

Direct Phone Extensions	
Name	Ext
Adams, Amy	305
Bawcum, Mary	301
Bouchard, Laurie	325
Boutin, Gail	338
Brown, Annette	330
Cain, Rhonda	300
Casarin, Joe	307
Chase, Eva	318
Dodge, Tamara	316
Doughty, Pam	310
Fitzherbert, Amy	313
Galasyn, James	337
Goodrich, Brett	321
Hall, Tim	335
Higgins, Missy	336
Hurlburt, Allison	302
Kennard, Cindy	343
Landry, Kasia	315
LeBrasseur, Lauraly	317
Lewis, George	332
Lynch, Paul	341
Martin, Kathy	304
Massey, Vickie	311
McCaw, Andrew	324
Parlin, Carla	344
Quirion, Kathy	339
Reed, Francis	329
Stephenson, Fred	314
Vigue, Vanessa	342

Cell Phones	
Coombs, Shaun	861-1887
Doughty, Pam	592-6622
Galasyn, Jim	229-3603
Goodrich, Brett	441-0662
Gordon, Mike	314-1754
Lewis, George	314-8388
Lynch, Paul	649-4483
Reed, Francis	692-3136
Vigue, Vanessa	680-6896
Spare NEL Cell	314-4246

Voice Mail Only	
Ashey, Rhonda	483
Ashey, Ryan	-
Boyce, Vanessa	-
Butler, Regina	466
Coombs, Shaun	-
Courtney, Roberta	468
Davidson, Liliane	-
Derosby, Bob	-
Doane, Jay	467
Estabrook, Kim	-
Getchell, Carol	-
Gordon, Mike	488
Hartwell, Ashley	-
Hebert, Alan	-
Hreben, Crystal	474
Leathers, Jody	463
Leathers, Kalena	-
Noble, Misty	-
Price, Tiffany	461
Pullen, Debra	-
Zhang, Wei	485

Misc/Shared Extensions	
Name/Location	Ext
Conference Rm	323
Chemistry-Metals Lab	
Bishop, Tim	326*
Parker, Jacob	
Chemistry - Organics	
Fenwick, Melissa	327*
Patenaude, Abram	
Chemistry - Organics Prep	
Anthony, Kelsey	312*
Mullen, Dale	
Chemistry - Radon Lab	333
Chemistry - Wet Chem	
Laflamme, Valentina	334*
Mullen, Debbie	
Maintenance	
Davidson, Andrew	328*
Leathers, Robert	462 (messages)
Micro - Lab I (Main Lab)	
Dudley, Brooke	320*
Kyes, Amanda	
Micro - IAQ	
Cheethan, Tom	306*
Shaeffer, Matt	465 (messages)
Sample Test Kits	460 (messages)
Shipping/Receiving	
Boutin, Jeremy	303*
Bouchard, Blaine	
Frost, Shelly	
*Shared Extensions	

Portland Office	878-6481
Bill's cell:	415-7770

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NEL CURRICULUM VITAE

NAME OF EMPLOYEE (Last, first, middle)	TITLE	DATE OF HIRE
GOODRICH, BRETT A.	MICROBIOLOGY LABORATORY MANAGER	04/30/1999

EDUCATION

NAME OF HIGH SCHOOL, COLLEGE AND/OR UNIVERSITY	NO. OF YRS. COMPLETED	DATES ATTENDED		MAJOR SUBJECTS/ CREDIT HOURS	DEGREE	DATE CONFERRED
		FROM	TO			
Winslow High School	4	1990	1994		H.S. Diploma	June 1994
University of Maine – Orono	5	1994	1999	Chemistry – 16 Micro – 42	BS – Micro	May 1999

WORK EXPERIENCE

NAME AND ADDRESS OF ESTABLISHMENT	DATE EMPLOYED		TITLE OF POSITION HELD	RELATED WORK PERFORMED (Include specific analyses performed if applicable)
	FROM	TO		
NORTHEAST LABORATORY SERVICES	2004	Present	Microbiology Lab Manager	Spore trap analysis, fungal culture and ID, aerobic plate count, total coliform, salmonella, listeria.
	1999	2004	Microbiology Supervisor	As above
	1999	1999	Wet Chem Technician	Total suspended solids, total dissolved solids, grease & oil, pH, conductivity.

SIGNATURE OF EMPLOYEE	DATE

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NEL CURRICULUM VITAE

NAME OF EMPLOYEE (Last, first, middle)	TITLE	DATE OF HIRE
DOUGHTY, PAMELA M.	Total Quality MANAGER	09/18/1989

EDUCATION

NAME OF HIGH SCHOOL, COLLEGE AND/OR UNIVERSITY	NO. OF YRS. COMPLETED	DATES ATTENDED		MAJOR SUBJECTS/ CREDIT HOURS	DEGREE	DATE CONFERRED
		FROM	TO			
Bayview Academy	4	1979	1983	College Prep	H.S. Diploma	June 1983
University of Maine-Farmington	4	1983	1987	Chem – 19 Biology – 45 Micro – 8	BA - Biology	May 1987

WORK EXPERIENCE

NAME AND ADDRESS OF ESTABLISHMENT	DATE EMPLOYED		TITLE OF POSITION HELD	RELATED WORK PERFORMED (Include specific analyses performed if applicable)
	FROM	TO		
NORTHEAST LABORATORY SERVICES	2005	Present	QA Manager	Provide internal and external assistance of customers. Maintain NEL's analytical division quality system.
	2004	2005	Customer Service/Project Manager	Provide internal and external assistance of customers.
	1994	2004	Quality Assurance Manager	Quality System
	1989	1994	Sample Manager	Log-in

SIGNATURE OF EMPLOYEE	DATE

UNCONTROLLED

NEL CURRICULUM VITAE

NAME OF EMPLOYEE (Last, first, middle)	TITLE	DATE OF HIRE
HURLBURT, ALLISON L.	QC Officer/Analyst	06/01/2004

EDUCATION

NAME OF HIGH SCHOOL, COLLEGE AND/OR UNIVERSITY	NO. OF YRS. COMPLETED	DATES ATTENDED		MAJOR SUBJECTS/ CREDIT HOURS	DEGREE	DATE CONFERRED
		FROM	TO			
Winslow High School	4	1993	1997		H.S. Diploma	June 1997
St. Anselm College	4	1997	2001	Biology – 40 hrs	BA - Biology	May 2001
University of Maine @ Orono	2	2001	2003	Micro – 30 hrs	MS - Microbiology	December 2003

WORK EXPERIENCE

NAME AND ADDRESS OF ESTABLISHMENT	DATE EMPLOYED		TITLE OF POSITION HELD	RELATED WORK PERFORMED (Include specific analyses performed if applicable)
	FROM	TO		
NORTHEAST LABORATORY SERVICES	2004	Present	QC Officer/ Analyst	Performs routine & specialized bench procedures as required. Conducts and oversees QC/QA in food laboratory. Generates SOPs.
NORTHEAST LABORATORY SERVICES	2004	2004	QC Assistant – Media Div.	Assisting in growth challenges of products.
Harvard Medical School	2003	2004	Research Assistant II	Maintenance of *C. elegans* strain library, preparation of media and lab stock solutions, RNAi.
University of Maine @ Orono	2001	2003	Teaching Assistant	Preparation of reagents and materials used in classes, prepare and grade quizzes and exams, taught labs.
Bio-Concept Laboratories	2000	2000	Junior Scientist	Prepare media, growth challenges, assist in testing of articles for FDA submission.

SIGNATURE OF EMPLOYEE	DATE

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NEL CURRICULUM VITAE

NAME OF EMPLOYEE (Last, first, middle)	TITLE	DATE OF HIRE
VIGUE, VANESSA A.	Food Microbiology Technical and Project Manager	01/06/2003

EDUCATION

NAME OF HIGH SCHOOL, COLLEGE AND/OR UNIVERSITY	NO. OF YRS. COMPLETED	DATES ATTENDED		MAJOR SUBJECTS/ CREDIT HOURS	DEGREE	DATE CONFERRED
		FROM	TO			
East Windsor High School	1	1992	1993	College Prep		
Freeport High School	3	1993	1996	College Prep	H.S. Diploma	June 1996
Maine Maritime Academy	4	1996	2000	Bio – 22 hrs Chem – 22 hrs	B.S. Ocean Studies	May 2000
Temple University	1	2003	2003	QA/Reg. Affairs – 8 hrs	QA/RA	N/A

WORK EXPERIENCE

NAME AND ADDRESS OF ESTABLISHMENT	DATE EMPLOYED		TITLE OF POSITION HELD	RELATED WORK PERFORMED (Include specific analyses performed if applicable)
	FROM	TO		
NORTHEAST LABORATORY SERVICES	01/2006	Present	Food Microbiology Technical and Project Manager	Identify and secure new business, maintain and support existing business with additional testing and technical support, deliver sales presentations, coordinate and attend industry shows and events.
NORTHEAST LABORATORY SERVICES	2004	01/2006	Food Microbiology Manager	Perform technical, administrative and managerial functions for the daily operation of the food micro lab.
NORTHEAST LABORATORY SERVICES	2003	2004	Supervisor/QA Officer/Microbiologist	Microbiological assays (food, environ, pharmaceutical), pathogens, bioburden, indicator orgs., Chloramphenicol, ethanol, SOPs, histamine, PT studies, implement QA program, client services.
Wyeth Pharmaceuticals	2001	2002	Compliance Microbiologist	Thermometer, autoclave, oven, ref. Calibrations & verifications. Create SOPs. Ensure compliance to FDA & GMP specs, investigate discrepancies.
Accutest Environmental	2000	2001	Inorganic Chemist	Distillations, CN, PN, AMN, titrations, color, odor, TCLP, hexavalent chromium, Hg, TN, NO2, No3, pH, Cl, TDE, TOX, leachate analysis, chromatography.

SIGNATURE OF EMPLOYEE	DATE

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NEL CURRICULUM VITAE

NAME OF EMPLOYEE (Last, first, middle)	TITLE	DATE OF HIRE
LYNCH, PAUL R.	RADIATION HAZ MAT & FIELD SERVICES MANAGER	02/2001

EDUCATION

NAME OF HIGH SCHOOL, COLLEGE AND/OR UNIVERSITY	NO. OF YRS. COMPLETED	DATES ATTENDED		MAJOR SUBJECTS/ CREDIT HOURS	DEGREE	DATE CONFERRED
		FROM	TO			
Skowhegan Area High School	4	1985	1989		H.S. Diploma	June 1985
Allegheny College	4	1989	1993	Environmental Science	BS – Env. Science	May 1993
Thomas College	2	2000	2002	Information Technician	BS – Information Tech	May 2002

WORK EXPERIENCE

NAME AND ADDRESS OF ESTABLISHMENT	DATE EMPLOYED		TITLE OF POSITION HELD	RELATED WORK PERFORMED (Include specific analyses performed if applicable)
	FROM	TO		
NORTHEAST LABORATORY SERVICES	2001	Present	EHSO	Sampling, environmental management, RSO, customer service, safety officer
R.F. Weston	1998	2000	Project Manager	Air Emission Testing
Eastmount Analytical	1993	1998	Project Manager	Air Emission Testing

SIGNATURE OF EMPLOYEE	DATE

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NEL CURRICULUM VITAE

NAME OF EMPLOYEE (Last, first, middle)	TITLE	DATE OF HIRE
GALASYN, JAMES	CHEMISTRY LABORATORY MANAGER	02/19/2003

EDUCATION

NAME OF HIGH SCHOOL, COLLEGE AND/OR UNIVERSITY	NO. OF YRS. COMPLETED	DATES ATTENDED		MAJOR SUBJECTS/ CREDIT HOURS	DEGREE	DATE CONFERRED
		FROM	TO			
Newington (Ct) High School	4	1972	1976		H.S. Diploma	June 1976
University of Connecticut	4	1976	1980	Chemistry	BS - Chemistry	1980
Dartmouth College	4	1980	1984	Analytical/Env Chemistry	PhD – Analytical/Env Chemistry	1984
Colorado College	2	1984	1985	Atmospheric Chemistry	Postdoctoral Fellow-NSF	

WORK EXPERIENCE

NAME AND ADDRESS OF ESTABLISHMENT	DATE EMPLOYED		TITLE OF POSITION HELD	RELATED WORK PERFORMED (Include specific analyses performed if applicable)
	FROM	TO		
NORTHEAST LABORATORY SERVICES	2006	Present	Chemistry Laboratory Manager	Responsible for daily activities of the laboratory including qc, analyses, technical direction, employee assignments and training.
NORTHEAST LABORATORY SERVICES	02/2003	2006	Organics Supervisor	Extractions, 8081, 8082, 8151, 8260, 624, 524.2, 8270, 8270-SIM, TOC
Katahdin Analytical	1987	2003	GC/MS Supervisor	All GC/MS Methods
York Wastewater	1985	1987	SVOA Supervisor	SVOA-GC/MS

SIGNATURE OF EMPLOYEE	DATE

UNCONTROLLED

JOB DESCRIPTION

Northeast Laboratory Services	***Chemistry Laboratory Manager***
Department:	Analytical
Reports to:	Company President
FLSA Status:	Exempt – Professional Learned
Summary:	Directs the performance of the various laboratories to achieve safe, efficient operation that meets the data quality objectives of our clients. Responsible for an effective sales and marketing program to promote laboratory services.

Essential duties and responsibilities include the following:

- Coordinates quotes, schedules, analyses, report generation, billing, and client contact for laboratory functions.
- Supervises and oversees training of all laboratory staff.
- Manages personnel issues of all laboratory staff.
- Oversees scheduling of laboratory workload.
- Ensures that a preventative maintenance program maintained.
- Assures that all activities are performed in accordance with methods and protocols specified in SOPs.
- Approves changes in SOPs and quality control procedures.
- Complies with and requires compliance with the LQAP by employees.
- Ensures that a preventative maintenance program is maintained.
- Primary point of contact for laboratory clients.
- Reviews and approves data for compliance with data quality objectives.
- Enforces corporate policies.
- Performs additional duties as required.
- Performs all duties in accordance with established safety guidelines.

Supervisory responsibilities:

Supervises analytical chemistry division staff. Is responsible for the overall direction, coordination and evaluation of this unit. Carries out supervisory responsibilities in accordance with NEL's policies and applicable laws. Recommends and coordinates purchases pertaining to the analytical division. Negotiates contracts, price variations and maintains business relations with analytical clients. Responsibilities include interviewing, hiring and training employees; planning, assigning and directing work; appraising performance; addressing complaints and resolving problems.

Minimum Qualifications:

Education: Bachelor of Science degree in appropriate scientific discipline,

Experience: Eight (8) years environmental laboratory experience, three (3) years supervisory experience, three (3) years budgetary experience.

Other: Good communication, computer, and organizational skills.

UNCONTROLLED

JOB DESCRIPTION

Northeast Laboratory Services	***Microbiology Laboratory Manager***
Department:	Microbiology
Reports to:	Company President
FLSA Status:	Exempt – Professional Learned
Summary:	Responsible for the daily operation of the Microbiology Lab

Essential duties and responsibilities include the following:

- Maintains required documentation. Communicates with clients and regulatory agencies concerning results and analytical procedures.
- Recommends and processes department purchases. Establishes service fees and cost accounting for analytical procedures. Oversees department accounting and sales/marketing efforts.
- Serves as a technical resource to department members and clients. Oversees quality control of analytical procedures and reporting.
- Performs routine and some specialized bench procedures when required
- Oversees the development of new analytical procedures and standard operating procedures
- Monitors and verifies the quality of analytical result data entered into the laboratory information management system
- Supervises department staff and serves as technical resource for clients and staff
- Conducts performance evaluations of department staff
- Performs cost quotations for analytical work
- Oversees Microbiology Lab QC testing
- Conducts field sampling and client sales visits
- Manages budget for the Microbiology Lab
- Meets regularly with other company management staff regarding corporate and department affairs
- Performs preventative maintenance and repair of lab equipment
- Performs additional duties as required
- Performs all duties in accordance with established lab quality and safety guidelines

Supervisory responsibilities:

Supervises microbiology staff. Is responsible for the overall direction, coordination and evaluation of this division. Carries out supervisory responsibilities in accordance with NEL's policies and applicable laws. Recommends and coordinates purchases pertaining to the microbiology division. Negotiates contracts, price variations and maintains business relations with analytical clients. Responsibilities include interviewing, hiring and training employees; planning, assigning and directing work; appraising performance; addressing complaints and resolving problems.

Minimum Qualifications:

Education: Masters degree in Microbiology or a closely related science

Experience: 2 years related lab experience with some supervisory experience
Alternate: Bachelors Degree in Microbiology or a closely related science and 5 or more years closely related lab experience, including some supervisory experience.

Other: Ability to organize and communicate effectively with clients and co-workers. Strong math and computer skills. Ability to travel as necessary, including occasional overnight travel. Ability to lift 35 pounds.

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JOB DESCRIPTION

Northeast Laboratory Services	***Total Quality Manager***
Department:	Administration
Reports to:	President & CEO
FLSA Status:	Exempt – Professional Learned
Summary:	Manages all aspects of the QA/QC program for all divisions at NEL.

Essential duties and responsibilities include the following:

- Manages Certification/Accreditations for NEL
- Performs Method and Quality Audits with assistance from Supervisors and QA/QC Technicians
- Manages a program for reports to Management and Management Review to encompass all three divisions
- Manages a program for calibration of support equipment throughout the lab
- Addresses/manages the discrepancies/corrective actions program with the aid of the database (track trends, produce reports, etc.)
- Monitors a program for instrument maintenance/calibration for adherence to accreditation requirements
- Monitors the program for random audits of sample data from receipt to reporting
- Maintains and updates the LQAP and monitors the update of the IAQ training manual and all other controlled documents including SOPs
- Manages the proficiency testing program with the lab, tracking trends with inclusion into the report to management and management review
- Performs annual ethics/QA training for all employees

Supervisory responsibilities:

This job has no supervisory responsibilities.

Minimum Qualifications:

Education: Bachelors degree in appropriate scientific discipline

Experience: Two years experience in an environmental lab

Other: Good statistical, communication, computer, and organizational skills

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JOB DESCRIPTION

Northeast Laboratory Services	***Quality Assurance/Quality Control Officer***
Department:	Microbiology
Reports to:	Laboratory Manager – Microbiology
FLSA Status:	Exempt – Professional Learned
Summary:	Reviews analytical data to assure compliance with data quality objectives under the guidance of the Quality Manager

Essential duties and responsibilities to assist the Quality Manager include the following:

- Ensures that sub contract labs have the appropriate certifications.
- Performs additional duties as required Ensures that method validation information is documented and accessible.
- Ensures the QA/QC program meets certification requirements.
- Reviews written responses to corrective actions to assure resolution.
- Responsible for the certification program in the lab.
- Responsible for keeping lab in compliance with certification requirements.
- Monitors the training program within the lab.
- Complies with the LQAP.
- Monitors the self-audit program within the lab.
- Performs all duties in accordance with established safety guidelines.

Supervisory responsibilities:

This job has no supervisory responsibilities.

Minimum Qualifications:

Education: Bachelors degree in appropriate scientific discipline

Experience: Two years experience in an environmental lab

Other: Good statistical, communication, computer, and organizational skills

UNCONTROLLED

JOB DESCRIPTION

Northeast Laboratory Services	**Food Microbiology Technical & Project Manager**
Department:	Microbiology
Reports to:	Microbiology Laboratory Manager
FLSA Status:	Exempt – Professional Learned
Summary:	Responsible for performing technical, administrative & supervisory functions for the daily operation of the Microbiology Lab

Essential duties and responsibilities include the following:

- Assists with sales efforts via cold calling, client visits and trade show attendance.
- Assures that all activities are performed in accordance with the methods and protocols specified in SOPs.
- Assists in supervision of analytical staff with regard to data quality.
- Identifies quality control problems, takes appropriate corrective actions.
- Recommends changes in SOPs and quality control procedures to the Quality Assurance Officer.
- Recommends actions for non-compliance with established policies and procedures.
- Performs routine and some specialized bench procedures when required
- Monitors and verifies the quality of analytical result data entered into the laboratory information management system
- Supervises department staff and serves as technical resource for clients and staff
- Performs cost quotations for analytical work
- Performs preventative maintenance and repair of lab equipment
- Performs additional duties as required
- Performs all duties in accordance with established lab quality and safety guidelines

* Note- Sales effort will be coordinated with Sales/ Marketing Manager at all times.

Supervisory responsibilities:

Supervises analysts in food microbiology division. Carries out supervisory responsibilities in accordance with NEL's policies and applicable laws. Recommends and coordinates purchases pertaining to the section. Maintains business relations with analytical clients. Assists with interviewing, hiring and training employees; planning, assigning and directing work; appraising performance; addressing complaints and resolving problems. Cold calling, client visits and tradeshow attendance is also required.

Minimum Qualifications:

Education: Bachelors degree in appropriate scientific discipline

Experience: Two years experience in an environmental lab

Other: Ability to organize and communicate effectively with clients and co-workers. Strong math and computer skills. Ability to travel as necessary, including occasional overnight travel. Ability to lift 35 pounds.

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JOB DESCRIPTION

Northeast Laboratory Services	***Radiation Haz Mat & Field Services Manager***
Department:	Chemistry
Reports to:	Environmental Health and Safety Officer
FLSA Status:	Exempt – Professional
Summary:	Assists in all aspects of an Environmental Health and Safety Program including Radiation Safety and Environmental Sampling.

Essential duties and responsibilities include the following:

- Maintains current status of instrument calibrations, QC, SOP for radon analysis
- Assists in hazardous waste determination and disposal
- Primary point of contact for radiation clients for shipping and receiving purposes, as well as radon questions
- Performs radon air and water analyses and issues reports
- Assists in field sampling events
- Performs method specific analysis as required
- Conforms to all Laboratory Quality Assurance Program guidelines
- Performs any additional duties as required
- Performs all duties in accordance with established safety guidelines

Supervisory responsibilities:

This job has no supervisory responsibilities.

Minimum Qualifications:

Education: Associate's degree in chemistry, environmental science or related field and completion of the Radioactive Safety Course or the ability to complete the course within six months of hire.

Experience: Two year's experience in an environmental laboratory.

Other: Knowledge of radioactive rules and regulations. The ability to lift 40 lbs. Valid drivers license. Good written and communication skills.

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JOB DESCRIPTION

Northeast Laboratory Services	***Section Supervisor***
Department:	Analytical Chemistry
Reports to:	Laboratory Manager – Analytical Division
FLSA Status:	Exempt – Professional Learned
Summary:	Reviews analytical data to assure compliance with methodologies

Essential duties and responsibilities include the following:

- Reviews all data produced by section.
- Assists in the maintenance of an internal audit program.
- Assures that all activities are performed in accordance with the methods and protocols specified in SOPs.
- Assists in supervision of analytical staff with regard to data quality.
- Assists in instrument operations as needed
- Identifies quality control problems, takes appropriate corrective actions.
- Recommends changes in SOPs and quality control procedures to the Quality Assurance Officer.
- Complies with the LQAP.
- Recommends actions for non-compliance with established policies and procedures.
- Performs additional duties as required.
- Performs all duties in accordance with established safety guidelines.

Supervisory responsibilities:

Supervises analysts and technicians in analytical division. Is responsible for the direction, coordination and evaluation of these units. Carries out supervisory responsibilities in accordance with NEL's policies and applicable laws. Recommends and coordinates purchases pertaining to the section. Maintains business relations with analytical clients. Responsibilities include interviewing, hiring and training employees; planning, assigning and directing work; appraising performance; addressing complaints and resolving problems.

Minimum Qualifications:

Education: Bachelors degree in appropriate scientific discipline; five years of experience in the group of interest along with the appropriate manufacturer instrument training may be substituted for the bachelors degree.

Experience: Minimum of two years experience in an environmental lab

Other: Good statistical, communication, computer, and organizational skills.
Ability to analyze environmental samples using EPA Methodologies.
Ability to troubleshoot laboratory equipment (i.e. GC, GC/MS, IC, ICP)

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JOB DESCRIPTION

Northeast Laboratory Services	***Analyst***
Department:	Analytical Chemistry
Reports to:	Laboratory Manager – Analytical Division
FLSA Status:	Exempt – Professional Learned
Summary:	Responsible for the timely and accurate analysis of specific analytes and associated recording and transcription of results.

Essential duties and responsibilities include the following:

- Coordinates the work schedules and the priority of analysis.
- Trains assigned technical staff.
- Assures that identifying data has been properly transcribed into records.
- Performs method specific analysis as required.
- Assures that analyses have been completed within established hold and turn around times.
- Assures that all associated analytical calculations are correct and that analyses or procedures have been performed according to NEL Sops and method protocols.
- Identifies, documents and corrects any data quality problems related to analyses.
- Conforms to all Laboratory Quality Assurance Program guidelines.
- Recommends departmental purchases.
- Performs calibration and maintenance of equipment.
- Performs additional duties as required.
- Performs all duties in accordance with established safety guidelines.

Supervisory responsibilities:

This job has no supervisory responsibilities.

Minimum Qualifications:

Education: Bachelor's degree in appropriate science discipline

Experience: --

Other: Be able to work safely with chemicals, lift 40 lb. and differentiate titration colors. Good computer and mechanical skills

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JOB DESCRIPTION

Northeast Laboratory Services	***Laboratory Technician***
Department:	Analytical Chemistry
Reports to:	Section Supervisor - Analytical Division
FLSA Status:	Non-exempt - Hourly
Summary:	Performs timely and accurate analysis of specific analytes and associated recording and transcription of results

Essential duties and responsibilities include the following:

- Coordinates the work schedules and the priority of analysis.
- Performs calibration and maintenance of equipment.
- Assures that identifying data has been properly transcribed into records
- Performs method specific analysis as required
- Assures that analyses have been completed within established hold and turn around times
- Assures that all associated analytical calculations are correct and that analyses or procedures have been performed according to NEL Sops and method protocols
- Conforms to all Laboratory Quality Assurance Program guidelines
- Performs additional duties as required
- Performs all duties in accordance with established safety guidelines

Supervisory responsibilities:

This job has no supervisory responsibilities.

Minimum Qualifications:

Education: High School Diploma

Experience: None

Other: Be able to work safely with chemicals, lift 40 lb. and differentiate titration colors. Good computer and mechanical skills

UNCONTROLLED

JOB DESCRIPTION

Northeast Laboratory Services	***Bottle & Kit Prep Technician***
Department:	Analytical Chemistry
Reports to:	Laboratory Manager – Analytical Division
FLSA Status:	Non-exempt - Hourly
Summary:	Responsible for all aspects of analytical kit, glassware and sampling equipment preparation prior to use.

Essential duties and responsibilities include the following:

- Prepares glassware and sampling equipment for laboratory use by a variety of techniques
- Responsible for receiving, ordering, preserving and send out of sample kits for all analytical testing
- Performs routine maintenance of equipment
- Responds to daily voice mail requests from clients for kits
- Performs additional duties as assigned
- Performs all duties in accordance with established safety guidelines

Supervisory responsibilities:

This job has no supervisory responsibilities.

Minimum Qualifications:

Education: High school diploma or GED equivalent

Experience: None

Other: Ability to lift 40 lb., work with minimal supervision and work safely around chemicals.

UNCONTROLLED

JOB DESCRIPTION

Northeast Laboratory Services	***Analyst***
Department:	Microbiology
Reports to:	Microbiology Laboratory Manager
FLSA Status:	Exempt – Professional Learned
Summary:	Responsible for performing technical and administrative functions for the daily operation of the Microbiology Lab.

Essential duties and responsibilities include the following:

- Performs routine and some specialized bench procedures as required.
- Consults with team members to determine and prioritize daily workload.
- Assists in the development of new analytical procedures.
- Assists in writing standard operating procedures.
- Enters analytical result data into laboratory information management system.
- Serves as technical resource for clients and department technicians.
- Assists in performance evaluations of department staff.
- Oversees and carries out production of media and dilution water.
- Performs preventative maintenance of lab equipment.
- Performs additional duties as required.
- Performs all duties in accordance with established lab quality and safety guidelines.

Supervisory responsibilities:

This job has no supervisory responsibilities.

Minimum Qualifications:

Education: Bachelor's degree in Microbiology or a closely related science

Experience: Two or more years closely-related lab experience.

Alternate: Associates degree in Microbiology or a closely related science *and* four or more years closely related lab experience

Other: Ability to organize and communicate effectively with clients and co-workers. Ability to travel as necessary, including occasional overnight travel. Strong math and computer skills. Ability to lift 35 pounds.

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2.6.0 Organizational Chart & Schematics of Facility

The following page is the present structure of the Laboratory and a schematics of the entire facility.

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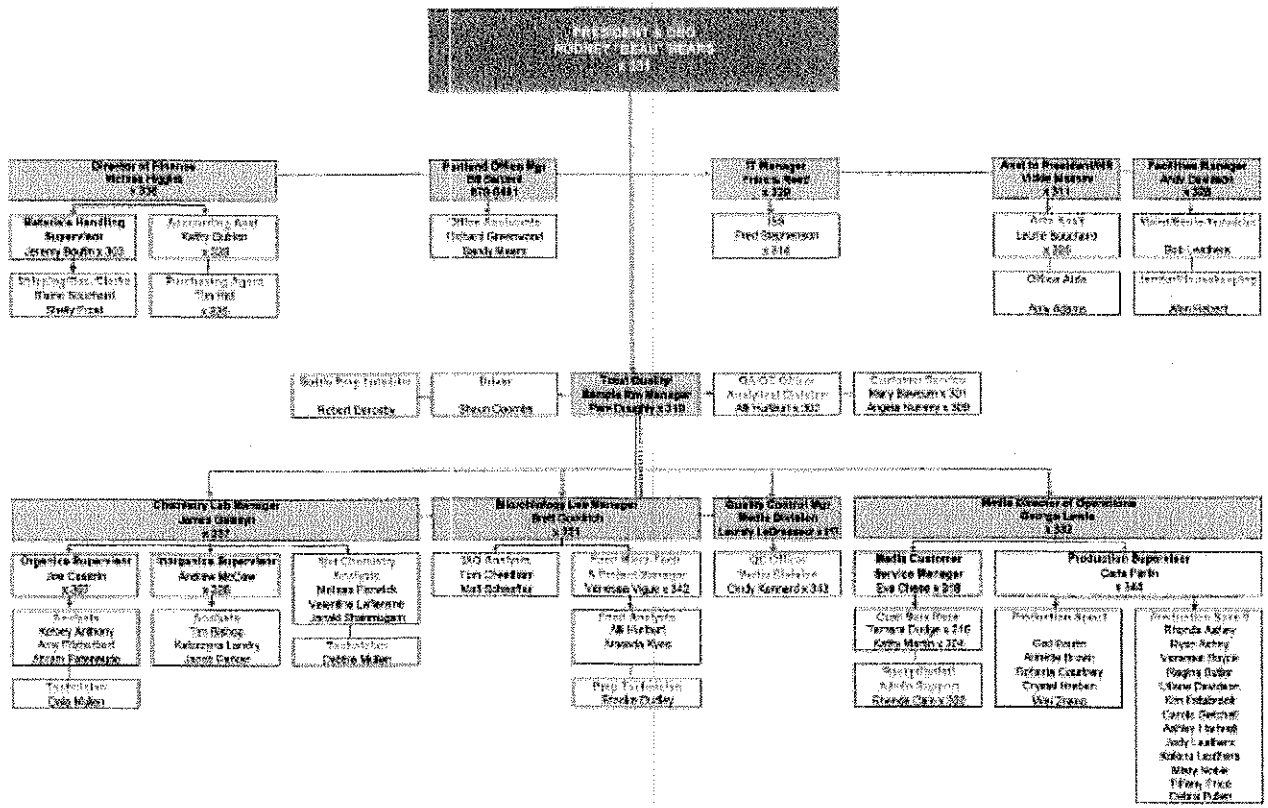
NORTHEAST LABORATORY SERVICES
ORGANIZATION CHART



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JOB DESCRIPTION

Northeast Laboratory Services	***Laboratory Technician***
Department:	Microbiology
Reports to:	Microbiology Laboratory Manager
FLSA Status:	Non-exempt - Hourly
Summary:	Responsible for performing technical procedures for the daily operation of the Microbiology Lab

Essential duties and responsibilities include the following:

- Performs routine and some specialized bench procedures as required
- Enters analytical result data into laboratory information management system
- Consults with team members to determine and prioritize daily workload
- Carries out production of media and dilution water
- Determines and prioritizes daily workload
- Assists in preventative maintenance of lab equipment
- Assists in general sanitation and housekeeping of the Microbiology Lab
- Performs additional duties as required
- Performs all duties in accordance with established safety guidelines

Supervisory responsibilities:

This job has no supervisory responsibilities.

Minimum Qualifications:

Education: Associates degree in Microbiology or a closely-related science field.

Experience: None

Alternate: High School Diploma or GED equivalent with one or more years experience working in a laboratory.

Other: Ability to organize and communicate effectively. Basic math skills and familiarity with computers. Ability to left 35 pounds.

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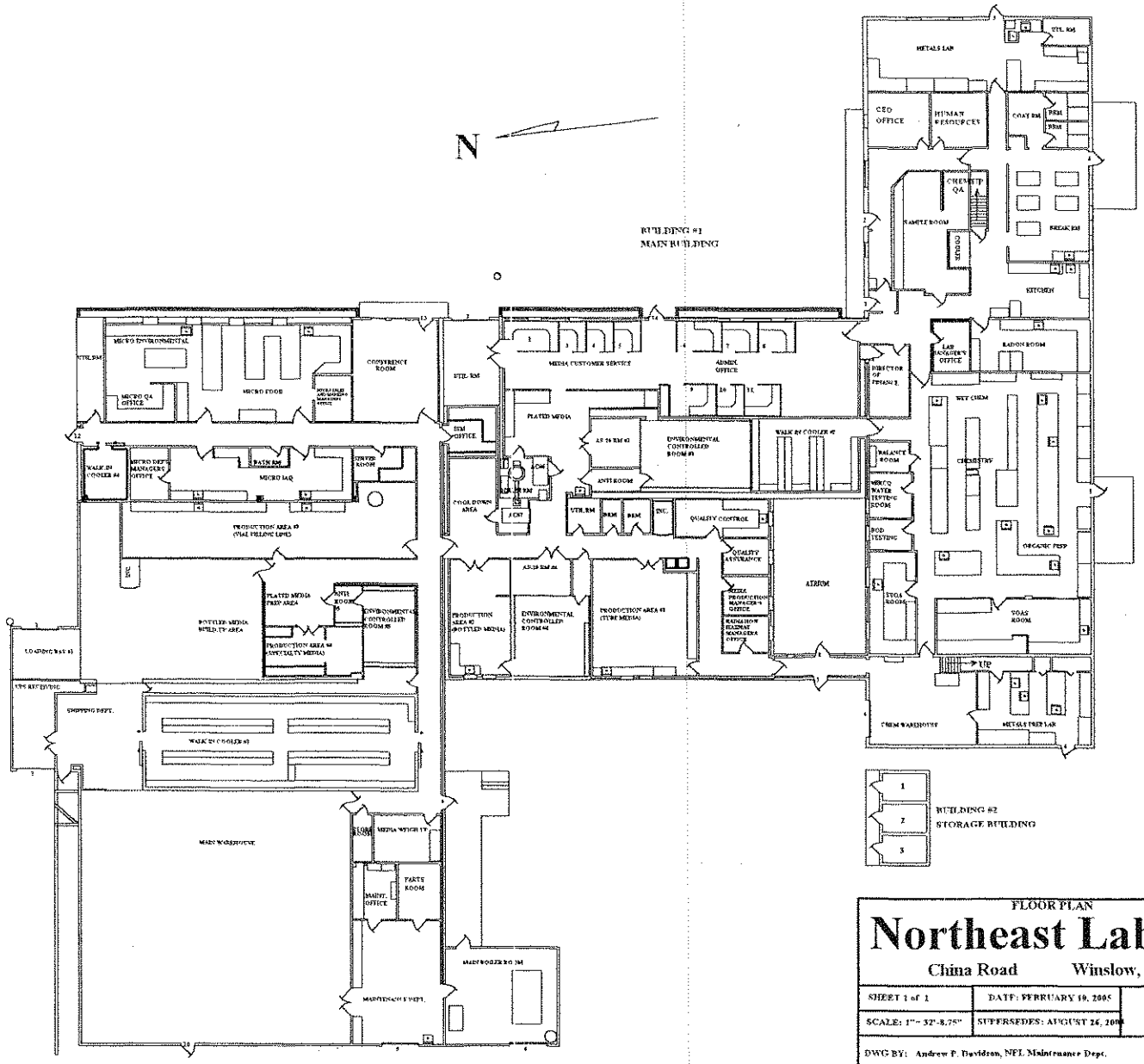

FLOOR PLAN

Northeast Laboratory

China Road Winslow, Maine

SHEET 1 of 1	DATE: FEBRUARY 10, 2005	
SCALE: 1"= 32'-8.75"	SUPERSEDES: AUGUST 26, 2004	
DWG BY: Andrew P. Davidson, NFL Maintenance Dept.		REF. MAP #

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CHAPTER 3

DATA QUALITY OBJECTIVES

3.1.0 Definitions

Data Quality Objectives (DQOs) are quantitative and qualitative statements describing the quality of data needed to support a specific environmental decision or action. DQOs include statements regarding precision, accuracy, representativeness, comparability and completeness (PARCC) needed to evaluate data. It is the responsibility of the client services staff, in conjunction with the laboratory manager and/or designee to provide assistance to clients in developing comprehensive and competitive bids that will satisfy the client's DQO's while also trying to educate clients and regulators as to the laboratory methods, instruments and other analytical requirements.

3.2.0 Project Management Capability and Capacity Review

Prior to bidding on an RFP (Request for Proposal) or RFQ (Request for Quotation) the laboratory manager and/or designee meets with the senior staff members of the laboratory to assess the laboratory capacity during the timing of the project. This only occurs when laboratory capacity or capabilities are in question and is determined by the laboratory manager. Otherwise, the project is accepted without question. The senior staff members must include:

- Client Service Manager
- Laboratory Manager
- QA Manager
- Organic Supervisor
- Inorganic Supervisor
- Food Microbiology Manager
- IAQ Microbiology Manager

If it is determined that the laboratory has the capacity and capability to perform the work, the supervisors are responsible for informing their employees of all details relating to the project. Details shall include: client name, number and matrix of samples, expected receipt date, turnaround time, analyses required and Quality Control (QC) level for data deliverables, and shall be maintained in a bound logbook or computer generated database.

3.3.0 Objective

3.3.1 The clients of the laboratory should be involved "up-front" in the development of DQO's. Some of the basic questions that need to be addressed at the outset of a given project include:

1) Name, address, telephone number of company
2) Contact person and when and how they wish to be contacted.
3) What is the purpose of the project? Project number, purchase order number?
4) What technical information/data is needed? Special reporting limits?
5) What is the use of data?
6) What resources are available?
7) What are the financial limitations of the project?
8) Are there any environmental variables the lab should be aware of?
9) When will the project arrive?
10) What turn-around-time is expected? Verbal and hardcopy?
11) Number of samples involved and matrix?
12) Electronic deliverables involved?

Initiated by: Total Quality Manager

Approved By: Pamela Doughty
Total Quality Manager

Approved By: [illegible]
Chemistry Lab Manager

Approved By: [illegible]
Microbiology Manager

13) Are there special certifications required?

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3.3.2 If the client is non-technical, the Laboratory's technical staff shall assist in identifying DQOs needed, usually over the phone, with a written quote or explanation of those objectives by the Laboratory, to follow by mail. However, the Laboratory may also assist in the development of a Quality Assurance Project Plan. Projects may proceed without DQOs; however, the resultant data may exceed or fall short of the clients' goals.

3.3.3 Once the basic tests are determined, the laboratory can provide a computer-generated price quote to the client that specifies the analytical methods and pricing. Upon review of the quote, the client will be afforded the opportunity to review any and all methods sited. If any of the tests requested cannot be performed at NEL, the price quote will reflect that subcontracted labs will be used. In the case of American Association of Laboratory Accreditation (A2LA) and American Industrial Hygiene Association (AIHA) scopes of accreditation, subcontracting is not permitted. All tests will be performed in house at NEL's Winslow, Maine Facility. If subcontracted labs are used for NELAC accreditation testing, the subcontractor will be identified in a separate column on the quote next to the test being subcontracted. All subcontracted labs used will be required to provide NEL with specific accreditations, SOPs and Quality Plans to support the work they are providing. All subcontractor lab information will be stored in the QA Manager's office. NEL will not be held responsible for client specified subcontractors if they have not been previously approved by NEL. The quote number should be referenced on all chain-of-custody forms for the sample to ensure correct pricing.

3.4.0 Standard DQO's

When special DQOs are not specified, the Laboratory's standard DQOs shall apply:

* Precision and accuracy requirements identified in the methods used to perform the analyses, shall apply. These requirements should be spelled out in all of the Standard Operating Procedures listed in Chapter 1. Precision and accuracy are defined on a method specific basis.

* Parameters required should be specified by the client up-front and may remain the same for specific monitoring projects (NPDES, RCRA groundwater, solids characterization, Baseline Studies). Alterations to the list of routine monitoring parameters must be communicated to the laboratory. Any alterations from the contract shall be communicated to the client.

* Standard Practical Quantitation limits/method detection limits shall apply unless prior notice to the laboratory as to their technical feasibility has been discussed. These limits are specified in the laboratory performance QC sections to follow or in the method SOP's.

* Significant figures differs by methodology but NEL's results are reported to 2 significant figures whenever possible. Some methods may only be accurate to one significant figure (i.e. total coliform [from 0 to 9 colonies]), while others may have up to 3 or 4 significant figures (i.e. pH). However, the majority of analytical methods employed here have an accuracy of two significant figures. NEL will not report non-significant figures unless required to do so by a regulating authority (i.e. proficiency samples to 3 significant figures).

* It is the laboratory's goal to provide 100% completeness in generating and reporting quantitatively usable data. The client service staff in conjunction with the Analytical Department Managers and/or designees shall be available to answer questions regarding analytical results and any other concerns that may arise after the project is complete. Data questions may involve re-running of samples. It is possible, depending on the outcome of the re-analyses, that additional charges may be necessary.

3.5.0 Client Calls

All client calls are returned on the same day or next business morning. If additional time and information is needed, the client will be notified and a time set for that additional information to be available.

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Client complaints will be handled as above. Regardless of where the fault lies, a corrective action report will be completed and all applicable staff will be notified so as to avoid the problem in the future. A record of the corrective action/customer complaint form/discrepancy report will be kept on file in the QA Manager's office and will be electronically stored in the Discrepancy Database.

Client calls with regard to billing will be routed to our Accounting Department. Calls relating to chain-of-custody questions, bottle orders or status of results will be handled by the customer service staff. Calls relating to quotes, data reports, analytical questions and any other matters are handled by the Customer Service Manager and technical staff. The Laboratory President is available to handle any particular complaints, difficulties or special requests that may arise in relation to laboratory services or client needs.

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CHAPTER 4

SAMPLE PRESERVATION PROCEDURES
HOLDING TIMES – LIQUIDS & SOLIDS

4.1.0 After the samples are taken, they should be sent to the laboratory for analysis as quickly as possible in order to ensure that the most reliable and accurate answers will be obtained as a result of the analysis. As a general rule, storage at low temperatures (4 degrees C) is the best way to preserve most samples, although the length of time the samples can be held at low temperatures varies with the method, analyte and matrix. Freezing samples to extend holding time IS NOT permitted. The bottles should be packaged for shipping in insulated containers, constructed to insure that the bottles will arrive at the laboratory intact.

When the samples are received at the laboratory, the time lapse between sample acquisition and analysis may not be such that the total holding time is exceeded.

4.2.0 Sample preservation should be performed immediately upon sample collection. For composite samples, each aliquot should be preserved at time of collection. When use of an automatic sampler makes it impossible to preserve each aliquot, samples may be preserved by keeping them at 4°C until compositing and sample splitting is completed. A sample preservation and holding timetable compiled from EPA sources is attached. For parameters and/or matrices not referenced standard EPA sample preservation requirements will be followed. The pHs are checked at the time of analyses for those samples that require acid or base preservation.

4.3.0 When any sample is to be shipped by common carrier or sent through the U.S. Mail, it must comply with the Department of Transportation's Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance.

4.4.0 Samples should be analyzed as soon as possible after collection. (The maximum holding times that samples may be stored before analysis and still be considered valid are listed in the attachment.)

4.5.0 For dissolved metals, samples should be filtered immediately on site before adding preservative.

4.6.0 When samples are received without adequate preservation, beyond holding times or are missing other pertinent information, the client is contacted by phone. If the client cannot be reached by phone, the attached FAX (Hold FAX.doc) will be sent to the Client defining the problem and requesting guidance from the client in writing before the sample will be admitted to the laboratory process. The discrepancy is also noted on the sample receipt condition report.

Initiated By: Total Quality Manager

Approved By: Pamela Doughty
Total Quality Manager

Approved By: R Halasy
Chemistry Lab Manager

Approved By: Brett Strobach
Microbiology Lab Manager

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COLLECTION AND PRESERVATION REQUIREMENTS**

General Chemistry

Chemistry

TEST	MATRIX*	ANALYTICAL METHOD	CONT. TYPE SIZE (ml)	MIN SAMPLE VOL (ml)	MAX HOLD TIME	PRESERVATION AMT----TYPE---TEMP	NOTES
Alkalinity, Total Bicarbonate	B D G S W	SM 2320 B/EPA 310.1/SM2320B	P / 250	250	14d	None------None------4C	
Carbonate	B D G S W	SM 2320 B	P / 250	250	14d	None------None------4C	
Asbestos	B D G S	Subcontract	G / 1000	1000	48h	None------None------4C	
BOD (Carbonaceous 5 Day)	D G S W	SM 5210B	P /500	500	24h	None------None------4C	
Bromide	B D G S	EPA 300.0/ EPA 9056	P /120	10	28d	None------None------4C	
Carbon Dioxide	B D G S	SM 4500-CO2B	See temperature,	Total dissolved	solids, pH and	alkalinity requirements	
Chloride	B D G S W	SM 4500 Cl D/EPA 300.0	P /120	10	28d	None------None------4C	
Chlorine, Residual	B D G S W	SM 4500-Cl D	P /500	50	Immediate	None------None------4C	(1)
COD	D G S W	SM 5220C/EPA 410.0	P /500	100	28d	2 ml-----H2SO4-------4C	
Color (Apparent & True)	B D G S	SM 2120	P /500	500	48hr	None------None-------4C	
Conductivity	B D G S	EPA 120.1/SM2510B	P /120	50	28d	None------None-------4C	
Quality Parameters							
Corrosivity (Langlier Index)	B D	EPA 9045/EPA 9040B	See total solids,	temperature,	total hardness,	pH and total alkalinity	Requirements
Cyanide, Total	B D G S W	EPA 300.0/EPA 9010B/SM4500 CN E	G / 1000	500	14d	1 pellet----NaOH-----4C	(17)
Fluoride	B D G S	EPA 300.0/EPA 9056	P / 120	50	28d	None-----None--------4C	
Foaming Agents (MBAS)	B D G S W	SM 5540C	P / 500	100	48h	None-----None--------4C	
Hardness, Calcium, Magnesium	B D G S	EPA 200.7 /6010B	P / 120	100	180d	5 ml/L---1:1 HNO3--4C	(17)
Nitrogen, Ammonia	G S	EPA 350.1/ SM4500 NH B3	P / 250	100	28d	4 ml/L-----1:1 H2SO4—4C	(17)
Nitrogen, Ammonia	W-Effluent	SM4500 NH B3	G / 1000	500	28d	4ml/L------1:1 H2SO4---4C	(17)
Nitrogen, Kjeldahl	G S W	SM4500 Norg C	P / 500	500	28d	4 ml/L-----H2SO4-------4C	(17)
Nitrogen, Nitrate	B D G S W	EPA 353.2/9056	P / 120	10	48h	None-------None----------4C	
Nitrogen, Nitrate-Nitrite	B D G S W	EPA 353.2/9056	P / 120	100	28d	4 ml/L-----1:1 H2SO4—4C	(17)
Nitrogen, Nitrite	D G S W	EPA 353.2/9056	P / 120	100	48h	None-------None----------4C	

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TEST	MATRIX*	ANALYTICAL METHOD	CONT. TYPE SIZE (ml)	MIN SAMPLE VOL (ml)	MAX HOLD TIME	PRESERVATION AMT----TYPE---TEMP	NOTES
Oil & Grease, Total or Petroleum Hydrocarbons	D G S W	EPA 1664	G / 500	1500	28d	2 ml/L-----H2SO4-------4C	(17)
	SO SE	Subcontract	G / 4 oz	20 g	28d	None-------None----------4C	
Oxygen, Dissolved	B D G S W	EPA 360.1	Test performed	in field	Immediate	None-------None----------	
pH	B D G S W	EPA 150.1/SM4500 H+ B	P / 120	25	Immediate	None-------None----------4C	
Phenols, Total	B D G S W	EPA 420.1/EPA 9056	G / 1000	1000	28d	2 ml/L-----H2SO4-------4C	
Phosphorus, Ortho	B D G S W	SM4500 P E/EPA 300.0/EPA 9056	P / 120	100	48h	None-------None----------4C	(2)
Phosphorus, Total	W	SM4500 P E	P / 120	100	28d	2 ml/L-----1:1 H2SO4---4C	(17)
Phosphorus, Total	BDGS	EPA 6010B	P / 120	100	28d	2ml/L-------1:1 HNO3----4C	(17)
Silica, as Si02	D G	EPA 200.7/EPA 6010B	P / 120	50	28d	None-------None----------4C	
Solids, Dissolved	B D G S W	EPA 160.1/SM2540C	P / 250	100	7d	None-------None----------4C	
Solids, Settleable	B D G S W	SM2540F	P / 250	100	48h	None-------None----------4C	
Solids, Suspended	B D G S W	SM2540D	P / 250	100	7d	None-------None----------4C	
Solids, Total	B D G S W	SM2540B	P / 250	100	7d	None-------None----------4C	
Solids, Volatile	B D G S W	EPA 160.4	P / 250	100	7d	None-------None----------4C	
Sulfate	B D G S W	EPA 9056/ EPA 300.0	P / 120	100	28d	None-------None----------4C	
Sulfide, Hydrogen	B D G S W	EPA 376.1	Test Performed	in field	Immediate	None-------None	
Sulfide, Total	W	SM4500 S2 F	P / 500	200	7d	4 dr/100 ml---2 N ZnAc---4C	(17)
Sulfite	W	EPA 377.1	P / 120	100	Not Listed	1 ml/100ml---2.5% EDTA-4C	(1) (17)
Temperature	B D G S W	EPA 170.1	Test performed	in field	Immediate	None-------None----------	
Total Organic Carbon (TOC)	B D G S W	EPA 415.1/9060/5310 C	Amber G / 250	100	28d	0.75 ml---1:1 H2SO4-----4C	(3) (17)
Total Organic Halogens (TOX)	B D G S W	EPA 9020	(non-CI) G / 250	100	14d	3 ml------1:1 HNO3-------4C	(4) (17)
			(CI) G / 250	100	14d	1 ml------10% Na2SO3---4C	(17)
Turbidity	B D G S W	EPA 180.1	P / 120	100	48h	None-------None----------4C	

* B=bottled water; D=drinking water; G=groundwater; S=surface water; W=wastewater; SO=solid; SE=sediment
(1) No headspace; (2) Filter immediately; (3) pH must be < 2, do not add excess H2SO4; (4) HNO3 must be added to pH = 2, NOT < 2; Protect from light; (7) upon receipt, HNO3 must be added to pH = 2, NOT < 2; (16) Preserved upon receipt; (17) Preserved when shipped
**References: Please see Chapter 20 of this document for references on the sources of this information.

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COLLECTION AND PRESERVATION REQUIREMENTS

General Chemistry – con't.

Radionuclides

TEST	MATRIX*	ANALYTICAL METHOD	CONT. TYPE SIZE (ml)	MIN SAMPLE VOL (ml)	MAX HOLD TIME	PRESERVATION AMT----TYPE---TEMP	NOTES
Gross Alpha & Beta	B D G S	Subcontract	P or G / 1000	1000	6m	3 ml/L---1:1N HNO3---4C	(17)
Gamma Spectral Anaylsis	B D G S	Subcontract	P or G / 1000	1000	6m	3 ml/L---1:1N HNO3---4C	(17)
Iodine-131	B D G S	Subcontract	P or G / 1000	1000	7d	3 ml/L---1:1N HNO3---4C	(17)
Radium-226	B D G S	Subcontract	P or G / 1000	1000	6m	3 ml/L---1:1N HNO3---4C	(17)
Radium-228	B D G S	Subcontract	P or G / 1000	1000	6m	3 ml/L---1:1N HNO3---4C	(17)
Radon-222	B D G S	Subcontract	G / 2 x 44.7	30	4d	None-------None---------4C	
Strontium-89 & 90	B D G S	Subcontract	P or G / 1000	1000	6m	3 ml/L---1:1N HNO3---4C	(17)
Tritium	B D G S	Subcontract	G / 1000	1000	6m	None-------None---------4C	
Uranium – 234, 235, & 238	B D G S	Subcontract	P or G / 1000	1000	6m	3 ml/L---1:1N HNO3---4C	(17)

Metals – By Analytical Method

200.7

TEST	MATRIX*	ANALYTICAL METHOD	CONT. TYPE SIZE (ml)	MIN SAMPLE VOL (ml)	MAX HOLD TIME	PRESERVATION AMT----TYPE---TEMP	NOTES
Mercury	B D G S W	EPA 245.1	P / 500	100	28d	3 ml/L-1:1 HNO3-Ambient	(17)
All Other Metals		EPA 200.7 / 6010	P / 500	500	6m	3 ml/L-1:1 HNO3-Ambient	(5) (17)

SM 3500-Cr D

TEST	MATRIX*	ANALYTICAL METHOD	CONT. TYPE SIZE (ml)	MIN SAMPLE VOL (ml)	MAX HOLD TIME	PRESERVATION AMT----TYPE---TEMP	NOTES
Chromium, Hexavalent	B D G S W	SM 3500-Cr D	P / 500	100	24h	None-----None----Ambient	

SM 3111B

TEST	MATRIX*	ANALYTICAL METHOD	CONT. TYPE SIZE (ml)	MIN SAMPLE VOL (ml)	MAX HOLD TIME	PRESERVATION AMT----TYPE---TEMP	NOTES
Mercury	SO SE	7000 Series	G / 4 oz	20 g	28d	None--------None-------4C	
Chromium, Hexavalent	SO SE	SW 7196	G / 4 oz	20 g	24h	None--------None-------4C	
All Other Metals					6m		

*B=bottled water; D=drinking water; G=groundwater; S=surface water; W=wastewater; SO=solid; SE=sediment

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COLLECTION AND PRESERVATION REQUIREMENTS

Microbiology

TEST	MATRIX*	ANALYTICAL METHOD	CONT. TYPE SIZE (ml)	MIN SAMPLE VOL	MAX HOLD TIME	PRESERVATION AMT----TYPE---TEMP	NOTES
Total Coliform/E. coli - MF	BD	SM9223B	G or P/120 ml	100 ml	30h	0.1 ml--10% Na2S203-4C	
Total Coliform/E. coli - MF	BDGSW	SM9222B	G or P/120 ml	100 ml	30h	0.1 ml--10% Na2S203-4C	
Total Coliform - RCRA	GS	EPA 9132	G or P/120 ml	100 ml	24h	0.1 ml--10% Na2S203-4C	
Toal Coliform - RCRA	SO SE	EPA 9131	G or P/120 ml	25 g	24h	0.1 ml--10% Na2S203-4C	(17)
Total Coliform - MPN	W	SM92221B	G or P/120 ml	25 g or ml	24h	0.1 ml--10% Na2S203-4C	(17)
Fecal Coliform - MF	BDSW	SM9222D	G or P/120 ml	100 ml	24h	0.1 ml--10% Na2S203-4C	
Fecal Coliform - MPN	BDW SO SE	SM9221E	G or P/120 ml	25 g or ml	24h	0.1 ml--10% Na2S203-4C	(17)
Fecal Strep - MF	W	SM9230C	G or P/120 ml	100 ml	24h	0.1 ml--10% Na2S203-4C	
Fecal Strep - MPN	W SO SE	SM9230B	G or P/120 ml	25 g or ml	24h	0.1 ml--10% Na2S203-4C	(17)
Salmonella - MPN	W SO SE	SM9260D	G or P/120 ml	25 g or ml	24h	0.1 ml--10% Na2S203-4C	(17)
HPC – Pour Plate	D	SM9215B	P/120ml	100 ml	24h	0.1 ml—10% Na2S203-4C	
HPC – Pour Plate	B	SM9215B	Gallon	100 ml	24h	None –4C	

* B=bottled water; D=drinking water; G=groundwater; S=surface water; W=wastewater; SO=solid; SE=sediment
(17) Preserved when shipped

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COLLECTION AND PRESERVATION REQUIREMENTS

Semi-Volatile Organic Chemicals

TEST	MATRIX*	ANALYTICAL METHOD	CONT. TYPE SIZE (ml)	MIN SAMPLE VOL (ml)	MAX HOLD TIME	PRESERVATION AMT----TYPE---TEMP	NOTES
Acetanalide Screen	B D G S	Envirogard	Amber G / 3x 40	40	14d	None--------None-------4C	
Acetochlor Screen	B D G S	Envirogard	Amber G / 3x 40	40	14d	None--------None-------4C	
Acid Extractables (Phenols)	G S W	EPA 8270	(non-CL) G / 2x 1000	1000	7d / 40d(ext)	None--------None-------4C	
	SO	EPA 8270	G / 4 oz	20 g	14d	None--------None-------4C	
Acrylamide	B D G S	EPA 8316	(non-CL) AG/ 2x 40	40	14d	None--------None-------4C	
			(CL) AG/ 2x 40	40	14d	0.1 ml--3% Na2S2O3---4C	(17)
Alcohols	B D G S	EHL	G / 2x 40	1 ml	28d	None--------None-------4C	
Aldehydes	B D G S	SM 6252	(non-CL) G / 2x 40	20	48h	None--------None-------4C	
			(CL) G / 2x 40	20	48h	0.1 ml--20% NH4CI---4C	(17)
			(O3) G / 2x 40	20	48h	0.1 ml--3% Na2S2O3---4C	(17)
Base/Neutral Extractables	G S	EPA 8270	(non-CL) G /2x 1000	1000	7d / 40d(ext)	None--------None-------4C	
	SO	EPA 8270	G / 4 oz	20 g	14d	None--------None-------4C	
Carbamates – Method List (Herbicides)	B D G S	EPA 531.1 Subcontract	G / 3x 40	40	28d	1.5 ml-------MC---------4C	(17)
- Phase II & V						and 0.1 ml---3% Na2S2O3	(15)
Chlorinated Acids – Method List	B D G S	EPA 515.1	(non-CL) G/1000	1000	14d / 28d(ext)	4 ml----1:1 HCI---------4C	(17)
- Phase II & V							
Chloral Hydrate	B D G S	EPA 551.1	G / 3x 40	40	14d	See Note---See Note----4C	(8)(17)
Dioxin	B D G S	Subcontract	G / 2x 1000	2000	30d	None--------None-------4C	
	B D G S	Subcontract	(non-CL) P / 500	2000	45d	None--------None-------4C	
	B D G S	Subcontract	(CL) P / 500	2000	45d	1 ml 10% Na2S2O3----4C	(17)
Diquat – Diquat/Paraquat	B D G S	EPA 549.1	(non-CL) P / 500	250	7d / 21d(ext)		(6)(12)(17)
			(CL) P / 500	250	7d / 21d(ext)	0.5 ml-10% Na2S2O3--4C	(6)(17)
EDB/DBCP	B D G S	EPA 504.1	G / 44.7	44.7	14d	0.1 ml--3% Na2S2O3---4C	(1)(17)
						and 0.2 ml 1:1 HCI	(17)
ETU	B D G S	EPA 509	Amber G / 2x 44.7	20	14d	None--------None-------4C	
Endothall	B D G S	EPA 548.1	G / 2x 44.7	10	7d / 14d(ext)	0.1 ml--3% Na2S2O3---4C	(17)

* B=bottled water; D=drinking water; G=groundwater; S=surface water; W=wastewater; SO=solid; SE=sediment
(1) No headspace; (6) Protect from light; (8) Addition of 0.45 g Phosphate Buffer with Na2SO3
(12) pH must be 2, do not add excess H2SO4; (15) MCA and Na2S2O3 can be mixed before shipment; (17) Preserved when shipped

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COLLECTION AND PRESERVATION REQUIREMENTS

Semi-Volatile Org. Chem. con't.

TEST	MATRIX*	ANALYTICAL METHOD	CONT. TYPE SIZE (ml)	MIN SAMPLE VOL (ml)	MAX HOLD TIME	PRESERVATION AMT----TYPE---TEMP	NOTES
Glyphosate – Glyphosate/AMPA	B D G S	EPA 547	G / 3x 40	40	14d	0.1 ml--3% Na2S2O3----4C	(17)
Haloacetic Acids	B D G S	SM 6251B	Amber G / 3x 40	40	9d / 21d(ext)	0.5 ml-----1% NH4CI----4C	(6)**(17)
Haloacetonitrites/Haloketones	B D G S	EPA 551.1	G / 3x 40	40	14d	See note-----See note----4C	(9) (17)
Chloropicrin							
Other Miscellaneous Pesticides	B D G S	Various	See requirements for	the method	that pesticides	are added to----------4C	
PAH, Total	SO	EPA 8270	G / 4 oz	20 g	14d	None--------None--------4C	
PAH, Total	GS	EPA 8270	G/2x 1000	1000	7d/ 40d(ext)	None--------None--------4C	
PCB	B D	EPA 505	G / 2x 1000	1000	7d / 40d(ext)	0.1 ml--3% Na2S2O3---4C	(17)
PCB, Total	G S	EPA 8082	G/ 2x 1000	1000	7d/ 40d(ext)	None---------None--------4C	
PCB, Total	SO	EPA 8082	G / 4 oz	20 g	14d/ 30d(ext)	None--------None--------4C	
Pesticides	G S	EPA 8081	G / 2x 1000	1000	7d / 14d(ext)	None---------None---4C	
- Phase II & V							
Pesticides	B D	EPA 525.2	(non-CL) G / 1000	1000	14d / 40d(ext)	4 ml ---1:1 HCI----------4C	(17)
Phase II & V			(CL) G / 1000	1000	14d / 40d(ext)	0.5 ml-10% Na2S2O3---4C	(17)
525.1 Method List							
Broad Spectrum							
Industrial Chemicals							
Priority Pollutants	W	EPA 625	(non-CL) G / 1000	1000	7d / 40d(ext)	None--------None--------4C	
			(CL) G / 1000	1000	7d / 40d(ext)	3 ml—10% Na2S2O3---4C	(17)
Triazine Screen	B D G S	Envirogard	Amber G / 1000	1000	14d	None--------None--------4C	
EHL-L131	B D G S	EHL/L131	Amber G / 500 ml	100 ml	14d	0.5 ml-10% Na2S2O3---4C	
EHL-HPLC Phenols	B D G S	EHL/HPLC Ph	Amber G / 250 ml	100 ml	7d	0.5 ml-10% Na2S2O3---4C	

* B=bottled water; D=drinking water; G=groundwater; S=surface water; W=wastewater; SO=solid; SE=sediment
** ICR Holding Time – 14 Days to extraction
(6) Protect from light; (9) Addition of 0.45 g Phosphate Buffer with NH4CI; (17) Preserved when shipped

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COLLECTION AND PRESERVATION REQUIREMENTS

Volatile Organic Chemicals

Petroleum Organics

TEST	MATRIX*	ANALYTICAL METHOD	CONT. TYPE SIZE (ml)	MIN SAMPLE VOL (ml)	MAX HOLD TIME	PRESERVATION AMT----TYPE---TEMP	NOTES
BTEX	G S	SW 8260	G / 3x 40	40	14d	0.2 ml------1:1 HCI------4C	(1) (17)
	SO	SW 8260	G / 4 oz	20 g	14d	None--------None--------4C	
Total Petroleum Hydrocarbons	D G S W	ME DEP 4.1.25	G / 2x 1000 ml	1000 ml	14d	3 ml------1:1 HCI-------4C	(1) (17)
--TPH as Diesel	SO	ME DEP 4.1.25	G / 4 oz	30 g	14d	None--------None--------4C	
TPH as Gasoline, Volatile	G S W	ME DEP 4.2.17	G / 3x 40	40	14d	0.2 ml------1:1 HCI-------4C	(1) (17)
	SO	ME DEP 4.2.17	G / 40	10 g	14d	10 ml------Methanol-----4C	
TPH as Waste or Heavy Oil,	G S W	ME DEP 4.1.25	G / 2x 1000 ml	1000 ml	14d	3 ml------1:1 HCI-------4C	(1) (17)
--Semi-Volatile	SO	EPA 8270	G / 4 oz	20 g	14d	None--------None--------4C	

Volatile Organics

TEST	MATRIX*	ANALYTICAL METHOD	CONT. TYPE SIZE (ml)	MIN SAMPLE VOL (ml)	MAX HOLD TIME	PRESERVATION AMT----TYPE---TEMP	NOTES
EHL-MIMS-THMs	B D G S	EHL	(non-CL) G / 2x 40	40	14d	0.2 ml------1:1 HCI------4C	(1) (17)
			(CL) G / 2x 40	40	14d	0.5 ml-5% Ascorbic acid-4C	(1) (17)
Phase I, II & V, Trihalomethane	B D G S	EPA 524.2	(non-CL) G / 3x 40	40	14d	0.2 ml------1:1 HCI------4C	(1) (17)
			(CL) G / 2x 40	40	14d	0.5 ml-5% Ascorbic acid-4C	(1) (17)
Maximum Total Trihalomethane	B D G S	EPA 510.1	G / 2x 250	500	14d	None--------None--------4C	(1) (6)
--Formation Potention							
Max Trihalomethane Potential	B D G S	44FR68672Part3	G / 4x 40	120	14d	None--------None--------4C	(1) (6)
Geosmin & MIB	B D G S	SM 6040C	G / 4x 40 ml	40	3d	None--------None--------4C	(1)
Other Miscellaneous Volatiles	B D G S	EPA 524.2/624	(non-CL) G / 3x 40	40	14d	0.2 ml------1:1 HCI------4C	(1) (17)
			(CL) G / 3x 40	40	14d	0.5 ml-5% Ascorbic acid-4C	(1) (17)
	G S W	EPA 8260 / 624	G / 3x 40	40	14d	0.2 ml------1:1 HCI------4C	(1) (17)
	SO	EPA 8260	G / 4 oz	20 g	14d	None--------None--------4C	(1)
Priority Pollutants	G S W	EPA 624 / 8260	G / 3x 40	40	14d	0.2 ml------1:1 HCI------4C	(1) (17)
Septic System	W	EPA 624	G / 3x 40	40	14d	0.2 ml------1:1 HCI------4C	(1) (17)
Trihalomethane	B D	EPA 5242	(non-CL) G / 3x 40	40	14d	0.2 ml------1:1 HCI------4C	(1) (17)
	GS	EPA 8260	(non-CL) G / 3x 40	40	14d	0.2ml------1:1 HCL------4C	(1) (17)

* B=bottled water; D=drinking water; G=groundwater; S=surface water; W=wastewater; SO=solid; SE=sediment
(1) No headspace; (6) Protect from light; (17) Preserved when shipped

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CHAPTER 5

SAMPLE HANDLING AND TRANSMITTAL PROCEDURES

5.1.0 Introduction

This Sample Handling Transmittal Procedure addresses sample handling from the time of sample receipt at the Laboratory until final disposition. This LQAP is divided into several key sections:

- Sample shipment, delivery, receipt and log-in.
- Chain of Custody Procedures.
- Internal Sample tracking, archiving and Sample disposal.

5.2.0 Sample Shipment and Delivery

All samples will be received by the Sample Specialist/Support Staff or a Designee.

5.2.1 The Sample Specialist or Designee shall have primary responsibility for the receipt and to log-in samples as follows:

- Potable Waters to be tested for the Express Test profile, FHA real-estate tests, Safety profiles as well as Radon Air and Water measurements.
- Wastewater/Groundwater/Surface water
- Solid waste/soils/ash
- Oils
- All other Chemistry and Microbiological samples (except radioactive samples).

5.2.2 Radioactive samples will be initially checked in by the Radiation Safety Officer (RSO) or designee.

5.2.3 All deliveries must be made between the hours of 0800 through 1700 Monday through Friday. There should be no receipt of samples at any other time than those listed above unless prior arrangements have been made or unless specifically required by contract. Samples can also be dropped off at night and during weekends in the drop-off box.

5.2.4 Samples may be shipped via NEL courier, common courier, U.S. Mail, and received through direct deliveries/walk-ins.

5.3.0 Sample Receipt

5.3.1 The Client Service Staff or Designee shall receive and log-in all samples, please see SOP 3.4.0.

5.3.2 The Staff must follow the procedures listed below when receiving samples:

5.3.3 Inspect documentation accompanying shipping container, verifying all are dated and signed where required.

Initiated By: Total Quality Manager

Approved By: Pamela Dayley
Total Quality Manager

Approved By: H Halay
Chemistry Lab Manager

Approved By: Brett Hardesh
Microbiology Lab Manager

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5.3.4 When handling chain-of-custody samples, sign for after verifying that the number of packages received agrees with airbill/shipping documents.

5.3.5 When handling chain-of-custody documents, sign and date the airbill/shipping documents upon receipt of samples.

5.3.6 Open the shipping container (i.e. Box, cooler, icechest) unless the samples are radioactive. If samples are radioactive contact the Radiation Safety Officer, who will then proceed to receive the samples in the radon room. Check condition of samples. Check for presence of any other forms or notes with the samples. Verify if the samples are custody sealed, if so verify if the seal is intact. If a temperature blank is included, record the temperature on the COC. If samples are in an insulated container but no temperature blank is included, measure and record the internal temp of the insulated container. Compile sample required log-in information and billing information. If samples have any discrepancies, a phone call is made to the client or if unavailable by phone, the attached Hold Fax form is used to notify the client. The sample receipt form is to be completed, followed by samples being held under refrigeration, if necessary, until the issue is resolved. Any samples suspected of being radioactive please see the following SOPS: SOP 3.6.3 Radioactive Sample Receipt, SOP 3.6.4 Radioactive Sample Return, SOP 3.6.5 Radiological Survey Meter Source Check, SOP 3.6.6 Radioactive Material Handling and SOP 3.6.7 Radioactive Surveys and Wipe Tests.

It should also be noted whom the report should be sent to if different than submitter. Check that the client sample identification(s) agrees with sample(s) submitted for analysis. Note any discrepancies in the client's file, as well as on the sample receipt form (see attached examples) which accompanies all other paperwork and is sent to the client with the final report and chain of custody form (see attached examples), resolve those discrepancies with the client and send follow-up documentation to the client.

5.4.0 Sample Log-in

Sample log-in involves the documentation of sample receipt by client services/designee prior to processing/analysis. The first step of this documentation is the proper entry of information into the laboratory information management system (LIMS), please see SOP 3.4.0.

5.4.1 The following LIMS entries shall be made for all samples received at the Laboratory.

Date Date sample received by laboratory and if required, the time received.

Sample ID# Internal sequentially generated unique sample number.

Sample Description/ID Whenever applicable, a literal identification of the location of sampling, or well number. [i.e. Monitoring Well 23, (MW 23)]

Study Groups/Test Required Study Groups should be established prior to sample submittal, under special quoted conditions which agree with the client's data quality objectives (DQOs). The use of work orders will automatically prompt which analyses are required on a routine recurring basis/routine wastewater monitoring parameters or a specific one time project. Tests required shall be identified when non-routine parameters have been requested. (i.e. As, Ba, Cr, 8260 VOA)

5.4.2 The sample log-in step involving data entry into the LIMS system shall include the following:

5.4.2.1 Entered into the LIMS Log-In Screen:

SBT NUMBER - Unique NEL account number.

MATRIX - Matrix refers to the sample type (i.e.: W-Water, G-Water, Solid, Oil, Soil, Leachate)

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DATE/TIME COLLECTED - This information should be obtained from the Chain of Custody Form.

DATE/TIME RECEIVED - This information should be obtained from the Chain of Custody Form.

SAMPLE DESCRIPTION - The submitter's sample identification goes here. It is the unique number or name the submitter assigns to each sample at the time of sampling and will be used by the customer to clearly identify the sample when reviewing the results.

STUDY GROUP # - Name for a collection of tests predefined by the user.

REPORT TO INFORMATION – Name, phone number, fax number and address of the person results are to be sent to.

Electronic Data Deliverable (EDD) REQUIREMENTS – Data format and delivery information entered in the comments field.

BILLING INFORMATION – Name, phone number, and address of the person who is to receive the invoice.

REPORTING INFORMATION (including QC level) QC level will appear on the analyst's worklists as individual codes and will be "x"'d out when completed by the analyst.

For chemistry samples, once the initial log in information is entered, the prices and specific tests have been assigned and the computer generated laboratory number has been assigned, the sample labels are checked to verify preservation and a member of the sample log-in staff will verify, through the use of pH paper and pipettes, that the pH of the sample is where it should be. If pH is not what it should be, log-in will request an analyst add the appropriate preservative and then samples are stored in coolers. The analysts will begin work on that sample in a timely manner, such that holding times are not exceeded.

For microbiology samples, samples are taken to the microbiology laboratory and separated into the food or IAQ lab. Each lab logs in the sample into their Internal Chain-of-Custody Logbook and all appropriate fields are completed in the logbook. Samples in the food micro lab with multiple containers per sample will have a unique identifier applied to each container, a sticker applied to reflect this identity, and storage condition of each, including discarding date will be recorded. For multiple container samples that are to be treated as one representative sample, these containers will be homogenized in the laboratory in such a way to ensure that a representative test portion is used for analysis.

If the food samples are sent out for contract laboratory analysis or to another department at NEL, this is also documented in the logbook. A disposal date for microbiology will also be assigned to the sample in the designated Internal Chain of Custody Book by the receiving analyst. The assigned disposal date will be 30 days from the completion of testing.

5.5.0 Sample Chain of Custody Procedures

A sample is physical evidence from a facility, an official, or from the environment. An essential part of monitoring activities is that samples collected by our clients and data may be used as evidence in legal proceedings. To satisfy evidentiary uses of data, the following sample chain-of-custody procedures have been established.

5.5.1 Sample Identification

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To ensure traceability of a sample while in the possession of the Laboratory, the following cross reference procedures have been established:

* Each sample or sample preparation container shall be labeled with a unique number identifier (Sample Number). The identifier shall be cross-referenced to the client sample ID# as documented in LIMS. This identification system is also applied to any sample blanks received with a batch of samples. Field blanks aid in identifying sampling and/or environmental contamination.

* The Sample Specialist/Support Staff shall verify through inspection that no transcription errors have been made in labeling. This cross-checking of labeling shall occur while the samples are in the possession of the above-mentioned staff and prior to securing the samples in the appropriate refrigerator at the time of sample receipt/log-in.

* The samples shall be labeled with a preprinted adhesive label bearing the sample number. This pre-printed label will also contain an additional number to indicate the number of bottles pertaining to the sample (i.e. AH01327.1)

5.5.2 A sample is under custody if:

* It is in your actual possession.
* It is in your view after being in your physical possession.
* It was in your possession and then you locked or sealed it up to prevent tampering, or
* If it is in a secure area. It is NEL's policy that the Chemistry and Microbiology sections of the laboratory are considered secure areas and that only authorized personnel are allowed to come in contact with the samples.

5.5.3 Upon receipt of the sample in custody, the Sample Specialist/Support Staff shall inspect the shipping container/cooler and sample bottles and shall document the following receiving information on the Sample Receipt Condition Report (see attached):

* Presence or absence of correct date(s) where necessary
* Presence or absence of signature(s) where necessary
* Presence or absence of a chain-of-custody form (if it had to be generated at the Laboratory)
* Presence or absence of air bills, noting any numbers
* Presence or absence of custody seals on shipping and/or sample containers and their condition (intact vs. broken)
* Presence or absence of sample tags, noting any ID numbers
* Sample tags with client identification numbers if not recorded on the chain-of-custody records
* Condition of the shipping container and temperature at the time of receipt
* Condition and type of the sample containers (see 5.5.3.1)
* Verification that the correct sample containers were used for the tests specified
* Presence or absence of a sufficient amount of sample for the tests specified
* Verification of agreement of information on receiving documents
* Resolution of problems or discrepancies with the client or the Laboratory Field Sampler.

5.5.3.1 Sample containers shall not be considered to be received in good condition if they are:

* Leaking
* Inadequately preserved or not at 4 degrees C (if applicable)
* Broken
* Past holding times

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* Insufficiently filled

5.5.4 The Laboratory's Sample Specialist/Support Staff shall sign and date all appropriate receiving documents at the time of receipt (i.e. Chain-of-Custody Forms, Shipping lists, air bills, etc.). The client shall be contacted if documentation is absent; if information does not agree, if custody seals are not intact, or if the sample is not in good condition with the Hold FAX form and/or telephone call.

The laboratory Sample Specialist/Support Staff shall document resolution of any discrepancies and this documentation shall become a part of the client's permanent file. This information should be noted on the FAX transmittal form and/or sample receipt form that accompanies all reports.

5.5.5 For potable waters, samples may be rejected if all pertinent information is not present. For example, the sample must be less than 30 hours old when it is received by the laboratory, the sufficient amount of money for the test must be enclosed with the sample (unless the client has an established credit account), the date and time the sample was taken must be recorded on the sheet accompanying the sample and the sample must not contain residual chlorine. If a sample is rejected for any of these reasons, the client will be contacted.

For samples requiring Heterotrophic Plate Count (HPC) analysis, refrigerate at 4°C if the analysis occurs more than 6 hours after collection. Maximum elapsed time between collection and analysis is 6 hours.

5.5.6 Sample Storage and Security

5.5.6.1 All samples shall be placed in the appropriate refrigerators, accessible to authorized personnel only, from time of receipt until sample preparation or analysis is completed.

5.5.6.2 Refrigerated storage areas are located in the Radioactive Walk-in and the Chemistry Section Walk-in. A refrigerator dedicated to volatile organic samples is located in the chemistry warehouse. Separate refrigerators for organic sample extracts are located in the organic section.

5.5.6.3 Samples will be stored in the original containers unless damaged.

5.5.6.4 All transfers of samples into and out of storage must be documented on the Internal tracking record. (See examples for chemistry and microbiology attached.)

5.5.6.5 When the analyst/technician removes the sample(s) from storage, the analyst becomes responsible for maintaining custody of the sample and shall date and initial the Internal Tracking Record.

5.5.6.6 Authorized personnel at the Laboratory shall consist of the Chemistry and Microbiology personnel only. No other personnel shall enter locked storage areas without prior authorization by the above parties.

5.5.6.7 Refrigerators for samples specifically requesting special security will be locked at all times.

5.5.6.8 A holding blank is placed in the volatiles refrigerator and the RAD refrigerator and analyzed once a week to ensure no cross contamination is occurring. A freezer blank for low level soil analysis is placed in with those samples in the freezer outside of the Sample Room. A water holding blank is a 40 ml VOA vial of distilled and organic free water. The freezer blank contains distilled and organic free water as well but does contain air space to prevent breakage. A report is generated and stored in the Total Quality Manager's office. If corrective action is necessary a

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discrepancy report is initiated by QA and followed through by the Chemistry Lab Manager.

5.5.7 Samples and standards must not be stored in the same refrigerators.

5.6.0 Policy for Disposal of Laboratory Samples

5.6.1 Water samples requiring BOD and Micro analyses will be disposed of 7 days after the completion of the analysis.

5.6.2 For all other analyses, after issuance of the report, the sample will be retained in a specified area for 30 days. After 30 days (unless other arrangements have been made), the sample will be disposed of in accordance with the state and federal regulations or if found to be hazardous returned to respective clients. For microbiology samples, the assigned 30 day disposal date is found in the Internal Chain of Custody Logbook.

5.6.3 For solid samples not known to contain a hazardous or regulated (RCRA) characteristic, TSCA, PCB, or State listed contaminant, disposal through a local municipal solid waste collection system as appropriate.

5.6.4 Regulated (RCRA) characteristic, TSCA, PCB, or state listed contaminant samples shall be inventoried by the Safety Officer and disposed of through an approved waste management company.

5.6.5 No criminal investigation samples or sample containers will be disposed of until disposal has been authorized by the appropriate officer of the court.

5.7.0 Subcontracting of Environmental Tests (for complete procedure see SOP 3.4.1).

5.7.1 Samples that arrive at NEL but cannot be analyzed by NEL will immediately be shipped overnight in a cooler packed with ice to a subcontracted laboratory.

5.7.2 Samples will be preserved according to the analyses they are to be analyzed for. See LQAP Chapter 4 for proper preservation of samples.

5.7.3 A copy of the chain of custody record and the corresponding purchase order number will be maintained in the subcontract logbook.

5.7.4 A copy of each subcontractor's quality manual will be obtained and stored in the Total Quality Manager's office.

5.7.5 For Navy samples, a copy of the Subcontractor Lab's Navy Acceptance letter will be kept on file with that lab's Quality Manual.

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Northeast Laboratory SERVICES
P.O. Box 788 • Waterville, ME 04903-0788
207-873-7711 / Fax: 207-873-7022
Shipping: Rt. 137, China Road
Winslow, Maine 04901

CHAIN OF CUSTODY RECORD

Company Name:

Reporting Address:

Client Contact: Phone Fax

Billing Name:

Billing Address:

PO #:
Quote #
Project Name

Lab Comments:

Preservation: 1 H_2SO_4, 2=HNO_3, 3=HCL, 4=NaOH, 5=Thio, 6=4°C, 7=None

Matrix Code Choose One: GW = Ground Water, WW = Waste Water, DW = Drinking Water, SW = Surface Water, O = Oil, WP = Wipe, S = Soil, F = Food, Other (Please List)

Sample Type Code: G = Grab, C = Composite

Sample Designation	Sample Date/Time	Preservation	Container Size, Type	QTY	Sample Type Code	Matrix Code	Analyses Requested	Lab Sample ID

Notes:

Known hazards associated with samples:

Sampled By	Date/Time	Received By	Date/Time	pH	The recommended temperature of the internal container is 2-6°C. Do you authorize analysis if the temp is out of range?
Relinquished By	Date/Time	Received By	Date/Time	Rush Yes 5 days 50% surcharge 2 days 100% surcharge	
Relinquished By	Date/Time	Received By	Date/Time	Field Filtered Y / N	Yes No

Chain of Custody must be filled out completely. This includes signature, sample description, analysis requested, date and time sampled.

If not completed, samples will be rejected.

Rev. 5/01 Form #NEL 100

If a sample is hazardous or radioactive, the sample will be returned to the client at the client's expense.
Standard turn-around-time is 10 business days - depending on season.

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NORTHEAST LABORATORY SERVICES
SAMPLE RECEIPT CONDITION REPORT

Client:_____ Date/Time Received:_____ Intials:_____

Project:_____ Rush? Yes No TAT

NEL Sample Numbers:_____

Delivered by:_____ Tracking Number:_____

Cooler _____of_____

	Yes	No	Comment
1. Are custody seals present?	___	___	_____
2. Is the COC present in cooler?	___	___	_____
3. Is the COC properly filled out?	___	___	_____ { } Completed at NEL
4. Is a temp blank present?	___	___	_____
5. Is the temperature of the cooler 4^0C +/- 2^0C?	___	___	Temp:_____ { } NA Collected <2Hr ago
6. Are ice pacs/ice present in cooler?	___	___	_____
7. Do sample labels match the COC?	___	___	_____
8. Are samples in proper sample containers with proper preservation?*	___	___	_____
9. Are samples within the analytical hold time?	___	___	_____
10. Is there sufficient volume to perform tests?	___	___	_____
11. Are samples in good condition?	___	___	_____
12.Are aqueous VOA/GRO vials free of headspace?	___	___	_____
13. If necessary, has the cooler been screened for radiological activity?	___	___	_____

*See internal COC for preservation verification

Comments:_____

_____

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Northeast Laboratory SERVICES

FAX TRANSMITTAL

To:		**From:**	Client Service Representative
Fax:		**Pages:**	1
Re:	Problem w/sample(s) received	**Date:**	
Project Name or Number:			

YOU MUST REPLY TO THIS FAX WITH THE NECESSARY INFORMATION TO ASSURE THE PROCESSING OF YOUR SAMPLE(S).

We are holding your samples pending the resolution of the following:

	Past Hold Time		Not Preserved		Not Labeled
	Not at Appropriate Temperature		Analysis Not Specified		Insufficient Paperwork
	Incomplete Paperwork		Other		

We will hold your sample(s) and wait for instructions on whether to proceed with the analyses. If hold time appears likely to expire before you can respond, we will proceed with analyses.

Please check one of the following and fax to 207/873-7022:

	I am aware of the discrepancy – please proceed with the analyses.
	Discard or return the sample(s) – we will resubmit or resolve the issue in another manner.

Name:	Date:

This message is intended only for the use of the individual or entity to which it is addressed and may contain information that is privileged, confidential and exempt from disclosure under applicable law. If the reader of this message is not the intended recipient or the employee or agent responsible for delivering the message to the intended recipient, you are hereby notified that any dissemination, distribution or copying of the communication is strictly prohibited. If you receive this communication in error, please notify us by telephone immediately (toll free 1-800-244-8378) to arrange for return of the original document(s) to NEL.

PO BOX 788, WATERVILLE, ME 04903-0788
CHINA ROAD, RT 137, WINSLOW, ME 04901
207/873-7711 1-800-244-8378

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NORTHEAST LABORATORY SERVICES
SAMPLE RECEIPT CONDITION REPORT

Client:_____
Date/Time Received:_____ **Intials:**_____

Project:_____
Rush? Yes No TAT

NEL Sample Numbers:_____

Delivered by:_____
Tracking Number:_____

FOOD AND WATER

	Yes	No
1. Are custody seals present?	____	____
2. Is the COC properly filled out?	____	____
3. Do sample labels match the COC?	____	____
4. Are samples within the analytical hold time?	____	____
5. Is there sufficient volume to perform tests?	____	____
6. Are samples in good condition?	____	____

Comments:_____

INDOOR AIR QUALITY

	Yes	No
1. Is the COC properly filled out?	____	____
2. Do sample labels match the COC?	____	____
3. Are slides broken or damaged?	____	____

Comments:_____

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NEL INTERNAL CHAIN OF CUSTODY FORM

Date Received: _____ Customer Name: _____ Project: _____ Immediate: _____ Initials: _____

		CONTAINER				LOCATION		IN	OUT	IN	OUT	IN	OUT
LAB ID #	# of Bottles	Type (a)	Size (b)	Preserva-tive (c)	pH	Analyst	Shelf (d)	Date/Time	Date/Time	Date/Time	Date/Time	Date/Time	Date/Time

(a) TYPE OF CONTAINERS			(b) CONTAINER SIZE				(c) PRESERVATIVE				(d) SHELF	
P = Plastic	G = Glass	Bg = Bag	1 = 1L	3 = 125L	5 = 4oz	7 = 100ml	1 = H_2SO_4	3 = HCL	5 = Thio	7 = None	M = Metals	W = Wet Chem
			2 = 5L	4 = 40ml	6 = 8oz	8 = 5 ml	2 = HNO_3	4 = NaOH	6 = 4°C	8 = Zn/Ac	O = Organics	

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Northeast Laboratory SERVICES

P.O. Box 748
Waterville, Maine 04903-0748

Rt. 137, Cline Road
Winslow, Maine 04901

Administrative Offices:
Phone: 207-873-7711
Fax: 207-873-7022
Customer Service
Phone: 800-244-8378
Fax: 207-873-7022

SUBJECT:	Sample Acceptance	EFFECTIVE DATE:	June 2007
		APPROVED BY:	Total Quality Manager

NEL SAMPLE ACCEPTANCE POLICY

- **All samples should arrive with proper, full and complete documentation** which includes:
  - Sample identification
  - Location of sample collection
  - Date & time of collection
  - Collector's name
  - Preservation type
  - Sample type
  - Any special remarks concerning the sample
- **Proper sample labeling** must include unique identification on the sample bottle(s)/containers(s) printed with indelible ink or other form of permanent marking.
- **Appropriate sample containers** must be used and obtained from NEL only.
- **Adequate sample volume** must be submitted to perform the necessary tests. All bottles/containers must be filled to the rim except in the case of 40 ml glass VOA vials which should be filled to over-flowing with no air space. If **soil samples** are submitted, please fill all bottles/containers to the rim except in the case of Volatile Organic or Gasoline Range Organics samples where a plug of soil is inserted into a 40 ml VOA vial. Please see attached Terra Core sampler instructions for additional instructions if this sampling is necessary.
- **All samples must be shipped on ice** so that temperature taken at the laboratory, through the use of an enclosed temperature blank, ranges between 0 and 6 degrees Celsius.
- **Frozen samples will not be accepted.** Clients will be called and the lab will proceed with the analyses requested if the client gives his/her permission to do so. In this case, reports and applicable documentation will be noted appropriately.

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CHAPTER 6

ANALYTICAL PROCEDURES

6.1.0 Introduction

Analytical procedures for the analysis of samples include the following:

6.1.1 Prescribed methods for sample preparation, including observance of stated sample holding times (as shown in LQAP 8) and necessary extractions, dilutions, etc.

6.1.2 Instrument standardization, including calibration and preventive maintenance.

6.1.3 Analytical techniques to be used in processing the sample.

6.1.4 Prescribed format on prepared data sheets for recording raw data which includes:

- Identification of project(s)
- Identification of sample number(s)
- Method Identification
- Identification of Analyst(s) by initialing and dating the "prepared by" line
- Identification of Analyst performing data validation by initialing and dating the "reviewed by" line
- Dates for sample analysis and data validation
- Raw data resulting from the analysis with appropriate calibration standards and blanks to include any necessary tag #s or unique NEL #s assigned for traceability to the original standard
- Unique date and method code on the subsample, digestate or extraction for the analysis
- Document all analytical and operational activities for the test in question (i.e. sulfide interference checks for cyanide, 30 minute reaction time for Waste Reactivity and sulfate ignition temperature (EPA 375.3) of 800°C.

6.1.5 Method for computation of analytical results, which will be included on the data sheet.

6.2.0 Documentation

Documentation of the above analytical procedure is dependent upon the specific instrumentation and data collection and reduction methods used within the laboratory. The requirements for activities such as calibration, preventive maintenance and quality control sample analysis shall be incorporated into the analytical laboratory specific standard operating manuals.

6.3.0 Analytical Methods

Whenever possible, the Laboratory utilizes industry recognized analytical methods from source documents published by agencies such as the U.S. Environmental Protection Agency (USEPA), American Public Health Association (APHA), American Society for Testing and Materials (ASTM) and the Official Methods of Analysis of the Association of Official Analytical Chemists (AOAC). The list of methods is attached.

6.4.0 Procedures for Departures from Documented Policies, Procedures or Standard Specifications

Analyses will be performed in accordance with the methods cited herein unless specific project requirements or needs dictate adoption of an alternate method or modification of the cited methods.

Initiated By: Total Quality Manager

Approved By: Pamela Dougherty
Total Quality Manager

Approved By: H Haley
Chemistry Lab Manager

Approved By: Brett Rothrock
Microbiology Lab Manager

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NEL handles nonconforming work via NEL SOP 3.1.4, which addresses complaints and corrective actions. Nonconforming work would result in an internal complaint and subsequent investigation and corrective action.

If analysis is performed in an alternate manner, the method shall be documented on the final report as well as in the analytical notebooks.

There will be occasions when time, sample matrix, or method constraints require a deviation from the normal quality control elements. When deviations occur, the following steps must be taken to ensure communication and authorization protocols are followed. It is very important that data is not misrepresented as meeting a certain protocol, when it does not.

A. It is the responsibility of all personnel to report nonconforming work to the area supervisor, laboratory director or quality manager.
B. The supervisory staff assesses the significance of the nonconforming work.
C. Corrective actions are taken immediately, together with any decision about the acceptability of the nonconforming work. Document the deviation on a Discrepancy Form.
D. Obtain permission from the Laboratory Manager and/or QA Manager to submit the data with the deviations.
E. Make certain, through sample comments or project narratives, that the client is aware of the deviation and what effect that deviation has on their data. Or if the data quality is or may be impacted, the client will be notified by the Laboratory Manager and/or Quality Manager within 7 business days after the nonconformance is identified. If necessary, an amended report is issued.
F. Work is resumed with the supervisory staff and quality manager's authorization only.

Any data submitted with undocumented deviations that do not meet NEL's minimum QC requirements could be considered fraudulent and may lead to termination.

If the assessment of the nonconforming work indicates that the deviation could reoccur or that there is doubt about the compliance of the laboratory's operations with its own policies and procedures, the correction action procedures given above will be followed immediately.

6.5.0 Anytime there is a change in personnel, a significant change made to the instrument or instrument type, or a change in test methods, an **initial demonstration of capabilities** will be completed (See Chapter 15). These demonstrations will be fully documented with the appropriate forms and raw data.

6.6.0 Microbiology Method Evaluation

6.6.1 Accepted Methods

Official test methods, methods from recognized national or international standards organizations, or commercial test kits may not require a specific validation. The laboratory will nonetheless demonstrate proficiency with all test methods prior to their first use. This demonstration may take several forms, including (but not limited to) analysis of validated proficiency test samples, comparison of results to those from a similar, validated method, or by simultaneous side-by-side analysis by several analysts. Please see Chapter 15 of this manual for additional information regarding spiked and non-spiked approaches to initial demonstration of capabilities.

6.6.2 Validation of Methods

Validation of microbiological test methods shall be performed under the same test conditions as those used for routine sample analysis. This can be achieved by using either reference suspensions made in the laboratory (or purchased from outside sources), naturally contaminated product and/or spiked product. Results should be capable of being statistically analyzed to demonstrate that the test meets its intended purpose. Qualitative (Presence/Absence) Methods may be validated in similar ways, except that statistical analysis may not be possible. If possible, specificity and reproducibility should be estimated. Measurement of uncertainty in all test methods is estimated using the Guidelines for Measurement of Uncertainty for all governing bodies.

6.6.3 Record Keeping

All validation data shall be recorded and stored at least as long as the test method is in use, or if withdrawn from active use, for at least five (5) years past the date of last use.

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6.6.4 Proficiency Test Programs

As a means of validating methods, demonstrating proficiency and maintaining accreditations, the laboratory shall participate in the Proficiency Test Programs identified by NELAC 5.4.2 or 5.5.34, or by appropriate sections amending or superceding these sections.

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American Association for Laboratory Accreditation

SCOPE OF ACCREDITATION TO ISO/IEC 17025:2005

NORTHEAST LABORATORY SERVICES
227 China Road
Winslow, ME 04901
Pamela M. Doughty Phone: 207 873 7711 Ext. 310

BIOLOGICAL

Valid to: October 31, 2009 Certificate Number: 2371.01

In recognition of the successful completion of the A2LA evaluation process (including an assessment of the laboratory's compliance with the A2LA Food Testing Program Requirements, containing the "AOAC *International Guidelines for Laboratories Performing Microbiological and Chemical Analyses of Food and Pharmaceuticals*"), accreditation is granted to this laboratory to perform the following tests on food, water, beverages and environmental wipes:

Test	SOP Reference	Test Method(s)
Quantitative		
Plate Counts (aerobic)	4.3.30	FDA BAM 8th Edition
Staphylococcus aureus	4.3.70	FDA BAM 8th Edition
Yeast and Mold	4.3.58	FDA BAM 8th Edition
MPN		
Coliforms	4.3.75	FDA BAM 8th Edition
Escherichia coli	4.3.43	FDA BAM 8th Edition
S. aureus	4.3.71	FDA BAM 8th Edition
Qualitative		
Salmonella sp.	4.3.65	FDA BAM 8th Edition
E. coli 0157:H7	4.3.45	BinaxNow, Meridian Immunocard STAT
Listeria sp. and *Listeria monocytogenes*	4.3.54	Oxoid Rapid Test

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Peter Mayer

5301 Buckeystown Pike, Suite 350 • Frederick, MD 21704-8373 • Phone: 301-644 3248 • Fax: 301-662 2974

LABORATORY QUALITY
ASSURANCE PROGRAMS

AIHA
Your Essential Connection. Advancing Occupational and Environmental Health and Safety Globally
2700 Prosperity Ave., Suite 250, Fairfax, VA 22031 U.S.A.
(703) 849-8888; Fax (703) 207-3561; www.aiha.org

AIHA Laboratory Quality Assurance Programs

SCOPE OF ACCREDITATION

Northeast Laboratory Services
227 China Road, Winslow, ME 04901

Laboratory ID: **102960**
Issue Date: 11/04/2005

The laboratory is approved for those specific field(s) of testing/methods listed in the table below. Clients are urged to verify the laboratory's current accreditation status for the particular field(s) of testing/Methods, since these can change due to proficiency status, suspension and/or revocation. A complete listing of currently accredited Industrial Hygiene laboratories is available on the AIHA website at:
http://www.aiha.org/LaboratoryServices/html/lists.htm

Environmental Microbiology Laboratory Accreditation Program (EMLAP)

Initial Accreditation Date: 10/01/2005

EMLAP Category	Field of Testing (FoT)	Method	Method Description (*for internal methods only*)
Fungal	Air – Direct Examination	SOP 4.3.24	Spore Trap Analysis: Direct Micrscopic Exam, Fungal, Spore Enumeration and ID
	Bulk – Direct Examination	SOP 4.3.27	Bulk Sample, Direct Microscopic Exam, Fungi, ID and Qualitative Assessment
	Surface – Direct Examination	SOP 4.3.25	Surface Tape Sample, Direct Micrscopic Exam, Fungi, ID and Qualitative Assessment
		SOP 4.3.26	Surface Swab Sample, Direct Microscopic Exam, Fungi, ID and Qualitative Assessment

The laboratory participates in the following AIHA proficiency testing programs:

- ✓ Fungal Culturable
- ✓ Bacterial Culturable
- ✓ Fungal Direct Examination

Effective: April 11, 2005
102960_Scope_EMLAP_2005_11_04
Author: Kris Heinbaugh
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NORTHEAST LABORATORY SERVICES
P.O. Box 788
Waterville, ME 04903-0788
Updated: 4/9/08

	SOP Number	Revision Code	SOP Title	Date	Compliant	NELAC AIHA A2LA Compliant Status
1	General Lab SOP # 3.7.5	L	Chemical Inventory and Traceability	1/17/08	Yes	Complete
2	General Lab SOP # 3.7.1	I	Washing of Glassware and Preservation of Sample Bottles	1/10/08	Yes	Complete
3	General Lab SOP # 3.7.2	O	QC of Refrigerators, Hoods, Balances, Incubators, Waterbaths, Petri Dishes and Calibration of Automatic Pipetters in the Analytical Divisions	4/2/08	Yes	Complete
4	General Lab SOP # 3.7.4	G	Daily Inspection of Hazardous Waste Building and Satellite Accum.	1/17/08	Yes	Complete
5	General Lab SOP # 3.7.3	G	Determination of Hazardous Constituents for Sample Disposal	1/17/08	Yes	Complete
6	General Lab SOP # 3.7.6	A	Procedure for Archiving Data for the Analytical Division	2/15/07	Yes	Complete
7	General Lab SOP # 3.7.7	F	Procedure for Rinsing Amber Bottles with Methylene Chloride	01/17/08	Yes	Complete
8	General Lab SOP # 3.7.8	A	Procedure for Formatting and Delivering Data Electronically to Clients	2/15/07	Yes	Complete
9	General Lab SOP # 3.7.9	D	Standard Operating Procedure for the Backup and Retrieval of Lab Data	2/15/07	Yes	Complete
10	General Lab SOP # 3.7.10	F	Standard Operating Procedure for Data Contained in Maintenance, Standards, Prep and Instrument Logs	1/10/08	Yes	Complete
11	General Lab SOP # 3.7.11	F	Standard Operating Procedure for the Proper Disposal of Samples	3/14/08	Yes	Complete
12	General Lab SOP # 3.7.12	A	Standard Operating Procedure for the Maintenance of the Final Report Generation System	2/15/07	Yes	Complete
13	General Lab SOP # 3.7.13	D	Standard Operating Procedure for the Determination of Method Detection Limits and Reporting Limits in the Chemistry Laboratory	2/27/08	Yes	Complete
14	General Lab SOP # 3.7.14	A	Standard Operating Procedure for the Proper Protocol used in the Event of Equipment Failure, Subsequent Client Contact and Corrective/Preventive Action	5/24/07	Yes	Complete
15	General Lab SOP # 3.7.15	A	Standard Operating Procedure for Auditing Computer System	11/8/07	Yes	Complete
16	General Lab SOP # 3.7.16	A	Standard Operating Procedure for Software Validation	11/8/07	Yes	Complete
17	Sample Log-In SOP # 3.4.0	N	Procedure for Proper Sample Receipt and Log-In of Analytical Samples	1/17/08	Yes	Complete
18	Sample Log-In SOP # 3.4.1	F	Procedure for Subcontracting Laboratory Analysis	1/11/08	Yes	Complete
19	Sample Log-In SOP # 3.4.2	E	Procedure for Proper Sample Receipt and Log-In of Potability Samples	01/17/08	Yes	Complete
20	General Lab SOP # 3.5.1	G	Procedure for the Review of Analytical Chemistry Reports to Client	1/23/08	Yes	Complete
21	General Lab SOP# 3.1.1	G	Standard Operating Procedure for Writing a Standard Operating Procedure	1/10/08	Yes	Complete
22	General Lab SOP # 3.1.4	K	Process Improvement Goals Program	1/10/08	Yes	Complete
23	General Lab SOP # 3.1.3	F	System Audits	1/10/08	Yes	Complete
24	General Lab SOP # 3.1.2	H	Procedure for Updating of Controlled Documents	1/10/08	Yes	Complete

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25	General Lab SOP # 3.1.5	D	Standard Operating Procedure for the Approval of all Documents Issued to the Laboratory	3/26/08	Yes	Complete
26	General Lab SOP # 3.1.6	F	Standard Operating Procedure for Management Review-Analytical Division	1/10/08	Yes	Complete
27	General Lab SOP # 3.1.7	E	Standard Operating Procedure for Proper Documentation Regarding Manual Integration of Chromatographic Peaks	2/27/08	Yes	Complete
28	General Lab SOP # 3.1.8	-	Data Review	3/16/08	Yes	Complete
29	Metals SOP # 3.2.1	I	Metals Analysis EPA 6010B	12/14/06	Yes	Complete
30	Metals SOP # 3.2.34	E	Mercury in Liquid Waste EPA 245.1	1/30/08	Yes	Complete
31	Metals SOP # 3.2.3	H	Mercury in Liquid Waste EPA 7470A	2/28/08	Yes	Complete
32	Metals SOP # 3.2.4	G	Mercury in Solid or Semisolid Waste EPA 7471A	1/28/08	Yes	Complete
33	Metals SOP #3.2.5	E	Acid Digestion of Aqueous Sample & Extracts for Total metals for Analysis by FLAA or ICP Spectroscopy EPA 3010A	1/23/08	Yes	Complete
34	Metals SOP # 3.2.6	I	Microwave Assisted Acid Digestion of Sediments, Sludges, Soils and Oils EPA 3051	4/18/02 **Obsolete**	No longer using	No longer using
35	Metals SOP # 3.2.28	C	Determination of Metals & Trace Elements in Water & Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry Method 200.7	6/6/07	Yes	Complete
36	Metals SOP # 3.2.8	D	Toxicity Characteristic Leaching Procedure Method 1311	2/25/08	Yes	Complete
37	Metals SOP # 3.2.74	A	Synthetic Precipitation Leaching Procedure Method 1312	8/8/06 **Obsolete**	Yes	Complete
38	Metals SOP # 3.2.10	E	Sample Preparation Procedure for Spectrochemical Determination of Total Recoverable Elements Method 200.2	3/26/08	Yes	Complete
39	Metals SOP # 3.2.12	B	Hardness by Calculation SM 2340B and Trivalent Chromium by Calculation-Lab Method	6/6/07	Yes	Complete
40	Metals SOP # 3.2.11	E	Determination of Total Solids in Solid and Semi-Solid Samples (SM2540G)	1/28/08	Yes	Complete
41	Metals SOP # 3.2.26	D	Determination of Trace Metals by Selective Temperature Graphite Furnace Atomic Absorption Spectrometry (200.9)	1/28/08	Yes	Complete
42	Wet Chemistry SOP # 3.2.27	A	Total Reactivity-Reactive Cyanide and Sulfide (SM 4500 CN E)	3/21/06	Yes	Complete
43	Wet Chemistry SOP # 3.2.38	C	Chromium, Hexavalent (7196A and 3500)	3/21/06	Yes	Complete
44	Metals SOP # 3.2.56	I	Odor EPA method 140.1	1/22/02 **Obsolete**	Yes	In process
45	Metals SOP # 3.2.57	A	Color EPA method SM2120B	1/28/08	Yes	Complete
46	Metals SOP # 3.2.59	D	Method 3005A Metals Digestion Waters	1/28/08	Yes	Complete
47	Metals SOP # 3.2.60	I	ILM 04 Metals CLP	2/4/02 **Obsolete**	Yes	Complete
48	Wet Chem SOP # 3.2.61	A	Total Sulfide EPA method 9034 for Groundwater and Solid Waste	7/3/06	Yes	Complete
49	Wet Chem SOP # 3.2.62	C	Calcium Carbonate Equivalence by AOAC	7/16/07	Yes	Complete
50	Metals SOP # 3.2.63	D	EPA 7060A Furnace Solids	3/3/08	Yes	Complete
51	Metals SOP # 3.2.64	F	Acid Digestion of Soils, Sediments and Sludges for metalsEPA 3050B	1/28/08	Yes	Complete
52	Metals SOP # 3.2.65	B	Paint Filter Liquids Test	1/28/08	Yes	Complete

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53	Metals SOP # 3.2.71	E	Thallium by EPA Method 7841 and Lead by EPA Method 7421 and Antimony by EPA Method 7041analysis by Graphite Furnace Atomic Absorption Spectrometry	3/3/08	Yes	Complete
54	Wet Chemistry SOP # 3.2.72	A	Alkaline Digestion of Solids for Hexavalent Chromium by EPA Method 3060A	3/21/06	Yes	Complete
55	Metals SOP # 3.2.73	B	EPA method 3020-Acid Digestion of Metals for Furnace Analysis	8/8/06 **Obsolete**	Yes	Complete
56	Metals SOP # 3.2.75	-	EPA method 206.2 Arsenic Groundwater	3/7/03 **Obsolete**	No	In process
57	Metals SOP # 3.2.76	-	AOAC 25.031-34-Cadmium and Lead in Earthenware	3/7/03 **Obsolete**	No	In process
58	Wet Chem SOP # 3.2.77	-	Standard Operating Procedure for Ash and Ash (Acid-Insoluble)	2/6/06	No	In process
59	Metals SOP # 3.2.79	B	Standard Operating Procedure for Selenium by EPA Method 7740A analysis by Graphite Furnace Atomic Absorption Spectrometry	3/3/08	Yes	Complete
60	Microbiology SOP # 4.1.1	D	Estimating Measurement Uncertainty	3/4/08	Yes	Complete
61	Microbiology SOP # 4.1.2	E	Standard Operating Procedure for the Review of Requests, Tenders and Contracts	1/17/08	Yes	Complete
62	Microbiology SOP # 4.1.3	F	Standard Operating Procedure for the Competency and Training of Analytical Personnel	1/17/08	Yes	Complete
63	Microbiology SOP # 4.1.4	E	Standard Operating Procedure for the Purchasing of Service and Supplies	3/08	Yes	Complete
64	Microbiology SOP # 4.1.5	D	Microbiology Department Procedure for Rush Samples	1/17/08	Yes	Complete
65	Microbiology SOP # 4.1.6	E	Microbiology Laboratory Environmental Monitoring Program	1/17/08	Yes	Complete
66	Microbiology SOP # 4.1.7	C	Validation of Methods Developed in Microbiology Laboratory	1/17/08	Yes	Complete
67	Microbiology SOP # 4.1.8	D	Standard Operating Procedure Control of Records in the Analytical Divisions	1/17/08	Yes	Complete
68	Microbiology SOP # 4.1.9	A	Ocular Micrometer Calibration	1/12/07	Yes	Complete
69	Microbiology SOP # 4.1.10	C	Procedure for the Handling, Transport, Storage, Use and Planned Maintenance of Measuring Equipment	1/17/08	Yes	Complete
70	Microbiology SOP # 4.1.11	C	Procedure for Safe Handling, Transport, Storage and Use of Reference Standards	1/17/08	Yes	Complete
71	Microbiology SOP # 4.1.12	D	Procedure for Obtaining Sub-Samples and Food Homogenates	1/17/08	Yes	Complete
72	Microbiology SOP # 4.1.13	F	Procedure to Determine Suitability and Disposal of Media	1/23/08	Yes	Complete
73	Microbiology SOP # 4.1.14	D	Approved Supplier List	1/17/08	Yes	Complete
74	Microbiology SOP # 4.1.15	B	Standard Operating Procedure for the Solicitation of Feedback from Customers	1/17/08	Yes	Complete

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75	Microbiology SOP # 4.1.16	B	Stability and Uniformity of the Temperature Distribution of Temperature Controlled Equipment (Thermal Mapping)	3/4/08	Yes	complete
76	Microbiology SOP # 4.1.17	A	Procedure for the Handling, Transport, Storage, Use and Safety of Biosafety Level II Infectious Microorganisms in the Workplace.	1/17/08	Yes	Complete
77	Microbiology SOP # 4.1.18	A	Procedure for Calibrating Working Thermometers	3/4/08	Yes	Complete
78	Microbiology SOP # 4.3.1	H	HPC SM 9215B	3/08	Yes	Complete
79	Microbiology SOP # 4.3.2	D	MI Agar for Total Coliforms & *Escherichia coli* in Drinking Water	1/19/07 **Obsolete**	Yes	Complete
80	Microbiology SOP # 4.3.3	E	Total Coliforms & *Escherichia coli* in Drinking Water and *E. coli* in WW SM 9223Bcolilert	3/26/08	Yes	Complete
81	Microbiology SOP # 4.3.4	F	Total Coliforms and *Escherichia coli* in DW and WW(m-Endo) SM9222B	3/08	Yes	Complete
82	Microbiology SOP # 4.3.5	E	Membrane Filtration Technique for Fecal Coliform (SM9222D)	3/08	Yes	Complete
83	Microbiology SOP # 4.3.9	C	Fecal Coliforms in Wastewater	1/05/06 **Obsolete**	Yes	Complete
84	Microbiology SOP # 4.3.10	A	Multiple Tube Technique for Fecal Streptoccus SM9230B	1/05/06 **Obsolete**	Yes	Complete
85	Microbiology SOP # 4.3.11	E	Membrane Filtration Technique for Fecal Strep SM9230C.3.b (m-ent.)	3/4/08	Yes	Complete
86	Microbiology SOP # 4.3.12	B	Total Coliforms – Multiple Tube Technique EPA 9131	1/05/06 **Obsolete**	Yes	Complete
87	Microbiology SOP # 4.3.13	B	Total Coliforms-Membrane Filter Technique EPA 9132	1/05/06 **Obsolete**	Yes	Complete
88	Microbiology SOP # 4.3.14	D	Salmonella Enumeration by MPN-SM 9260D	3/4/08	Yes	Complete
89	Microbiology SOP # 4.3.15	D	E.coli in Recreational Water by MF EPA 1103.1	3/08	Yes	Complete
90	Microbiology SOP # 4.3.20	C	MPN for Fecal Coliforms in Water and Wastewater SM 9221E	3/4/08	Yes	Complete
91	Microbiology SOP # 4.3.21	C	MPN for Total Coliforms in Water and Wastewater SM 9221B	3/08	Yes	Complete
92	Microbiology SOP # 4.3.22	I	SDWA EC MUG	6/28/02 **Obsolete**	No	In Process
93	Microbiology SOP # 4.3.23	A	Enterococci in Water by MF using mE-EIA Agar EPA 1106.1	1/19/06 **Obsolete**	Yes	Complete
94	Microbiology SOP # 4.3.24	F	Spore Trap Analysis: Direct Microscopic Exam, Fungal Spore Enumeration and ID	12/17/07	Yes	Complete
95	Microbiology SOP # 4.3.25	C	Surface Tape Sample, Direct Microscopic Exam, Fungi, Identification and Semi-Quantitative Assessment	10/30/07	Yes	Complete

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96	Microbiology SOP # 4.3.26	C	Surface Swab Sample, Direct Microscopic Exam, Fungi, Identification and Semi-Quantitative Assessment	10/31/07	Yes	Complete
97	Microbiology SOP # 4.3.27	C	Bulk Sample, Direct Microscopic Exam, Fungi, Identification and Semi-Quantitative Assessment	10/31/07	Yes	Complete
98	Microbiology SOP # 4.3.30	C	Aerobic Plate Count – SMA Pour Plate (FDA)	3/08	Yes	Complete
99	Microbiology SOP # 4.3.43	C	*E. coli* – MPN (FDA)	3/08	Yes	Complete
100	Microbiology SOP # 4.3.45	C	*E. coli* O157:H7 FDA	3/08	Yes	Complete
101	Microbiology SOP # 4.3.48	A	Fecal Coliforms – MPN	4/5/05 **Obsolete**	Yes	Complete
102	Microbiology SOP # 4.3.49	I	Fecal Shellfish MPN	4/27/05 **Obsolete**	Yes	Complete
103	Microbiology SOP # 4.3.50	I	Flat Sour Sporeformer	5/10/05 **Obsolete**	Yes	Complete
104	Microbiology SOP # 4.3.54	C	Listeria AOAC (Oxoid)	3/08	Yes	Complete
105	Microbiology SOP # 4.3.58	C	Mold and Yeast (FDA)	3/08	Yes	Complete
106	Microbiology SOP # 4.3.65	C	Salmonella FDA	3/4/08	Yes	Complete
107	Microbiology SOP # 4.3.68	I	SPC Dairy	4/29/05 **Obsolete**	Yes	Complete
108	Microbiology SOP # 4.3.70	C	*Staphylococcus aureus* (FDA)	3/08	Yes	Complete
109	Microbiology SOP # 4.3.71	C	*Staphylococcus aureus* MPN	3/08	Yes	Complete
110	Microbiology SOP # 4.3.74	I	Total Coliforms-Dairy SMEDP	**Obsolete**	No	Incomplete
111	Microbiology SOP # 4.3.75	A	Total Coliforms MPN (FDA)	3/4/08	Yes	Complete
112	Microbiology SOP # 4.3.76	A	Viscosity-Brookfield DV-II+ Pro	9/06 **Obsolete**	Yes	Complete
113	Microbiology SOP # 4.3.77	A	Brix	9/06 **Obsolete**	Yes	Complete
114	Microbiology SOP # 4.3.78	B	Moisture/Solids-CEM Microwave Analyzer	3/08	Yes	Complete
115	Microbiology SOP # 4.3.79	A	Color-Spectrophotometric	9/06 **Obsolete**	Yes	Complete
116	Microbiology SOP # 4.3.80	A	Specific Gravity – Toledo *Densito*	9/06 **Obsolete**	Yes	Complete

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117	Microbiology SOP # 4.3.81	A	Titratable Acidity – SCHOTT Titronic Potentiometric to 8.3	9/06 **Obsolete**	Yes	Complete
118	Microbiology SOP # 4.3.82	A	Calibration of New Microscopes	1/12/07	Yes	Complete
119	Microbiology SOP # 4.3.83	A	Procedure for the Preparation of Indoor Air Quality Reports	1/12/07	Yes	Complete
120	Microbiology SOP # 4.3.84	A	Aerobic Plate Count – Petrifilm (AOAC)	3/4/08	Yes	Complete
121	Microbiology SOP # 4.3.85	A	Total Coliforms – Petrifilm (AOAC)	3/4/08	Yes	Complete
122	Microbiology SOP 4.3.86	A	Mold and Yeast – Petrifilm (AOAC)	3/4/08	Yes	Complete
123	Microbiology SOP # 4.3.87	A	Enterobacteriaceae – Petrifilm (AOAC)	3/4/08	Yes	Complete
124	Microbiology SOP # 4.3.88	A	Bioburden Testing for Tampons	3/4/08	Yes	Complete
125	Microbiology SOP # 4.3.89	A	Total Coliforms and *E. coli* – Simplate (AOAC)	3/4/08	Yes	Complete
126	Microbiology SOP # 4.3.90	A	Lactic Acid Bacteria – MRS Agar	3/4/08	Yes	Complete
127	Microbiology SOP # 4.3.94	B	Biological Indicator Testing Service	3/4/08	Yes	Complete
128	Microbiology SOP # 4.3.95	A	Container and Closure Testing for Microbiological Contaminants SM9222B and SM9215D	11/5/07	Yes	Complete
129	Microbiology SOP # 4.3.96	A	Pseudomonas, Salmonella, E. coli and S. aureus USP	3/18/08	Yes	Complete
130	Organics SOP # 3.3.5	G	Determination of Chlorinated Acids in Water (8151A)	1/23/08	Yes	Complete
131	Organics SOP # 3.3.13	K	Volatile Organic Compounds (8260B)	1/23/08	Yes	Complete
132	Organics SOP # 3.3.14	I	Organochlorine Pesticides (8081)	1/23/08	Yes	Complete
133	Organics SOP # 3.3.15	L	Semivolatile Organic Compounds EPA 8270C	1/23/08	Yes	Complete
134	Organics SOP # 3.3.16	F	PCBs as Arochlors (8082)	1/23/08	Yes	Complete
135	Organics SOP # 3.3.17	I	Gasoline Range Organics (GRO) (8015B)	1/23/08	Yes	Complete
136	Organics SOP # 3.3.18	J	Diesel Range Organic Compounds (DRO)	1/23/08	Yes	Complete
137	Organics SOP # 3.3.29	F	Separatory Funnel Liquid-Semi-Volatile Organics	1/23/08	Yes	Complete
138	Organics SOP # 3.3.30	E	Separatory Funnel Liquid-Liquid Extraction SW-846 Method 3510C-Pesticides and PCBs	1/23/08	Yes	Complete
139	Organics SOP # 3.3.23	D	Sulfuric Acid/Permanganate Cleanup for Semi-Volatile Extraction SW-846 Method 3665A	1/23/08	Yes	Complete
140	Organics SOP # 3.3.25	D	Flashpoints (EPA 1010)	1/23/08	Yes	Complete
141	Organics SOP # 3.3.26	H	Volatile Organic Compounds in Drinking Water by Purge and Trap (524.2)	1/23/08	Yes	Complete
142	Organics SOP # 3.3.27	-	AOX	3/19/02	Yes	Complete
143	Organics SOP # 3.3.28	-	TOX (9020B)	3/18/02	Yes	Complete
144	Organics SOP # 3.3.31	H	Ultrasonic Extraction for Semi Volatile Organics	1/23/08	Yes	Complete

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145	Organics SOP # 3.3.32	E	Volatile Organic Compounds (624)	1/23/08	Yes	Complete
146	Organics SOP # 3.3.33	D	EPA method 3540C Soxhlet Extraction for Semi Volatile Organics	1/23/08	Yes	Complete
147	Organics SOP # 3.3.34	C	EPA method 3580 Extraction for Semi Volatile Organics by Waste Dilution	1/23/08	Yes	Complete
148	Organics SOP # 3.3.35	E	EPA method Ultrasonic Extraction for PCBs and Pesticides EPA method 3550B	1/23/08	Yes	Complete
149	Organics SOP # 3.3.36	I	EPA method 3540C Soxhlet Extraction for PCBs and Pesticides	1/23/08	Yes	Complete
150	Organics SOP # 3.3.37	A	EPA method 3660B Sulfur cleanup for Semi volatile Organic Extraction	1/23/08	Yes	Complete
151	Organics SOP # 3.3.38	[illegible]	EPA method 3620B Florisil Clean up for Semi volatile Organic Extraction	1/17/02 **Obsolete**	No	In process
152	Organics SOP # 3.3.39	C	Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) Capillary Column Technique- EPA method 625	1/23/08	Yes	Complete
153	Organics SOP # 3.3.40	F	Purge and Trap Aqueous samples --Prep for volatiles-EPA method 5030B	1/23/08	Yes	Complete
154	Organics SOP # 3.3.41	A	CLP Semi Volatile GC/MS water analysis	2/11/05 **Obsolete**	Yes	Complete
155	Organics SOP # 3.3.42	Removed 2/11/05	CLP Volatile GC/MS water analysis	2/19/02 **Obsolete**	Yes	Need major revision 2/11/05
156	Organics SOP # 3.3.43	C	EPA 608 Pesticides & PCBs in Wastewater	1/23/08	Yes	Complete
157	Organics SOP # 3.3.44	-	TOX by SM 5320B	3/25/02	Yes	Complete
158	Organics SOP # 3.3.45	E	EPA 5035 Volatiles	1/23/08	Yes	Complete
159	Organics SOP # 3.3.46	B	EPA method 8270C (SIM)	12/14/06	Yes	Complete
160	Organics SOP # 3.3.47	B	Extractable Total Petroleum Hydrocarbons	1/23/08	Yes	Complete
161	Organics SOP # 3.3.48	C	Determination Petroleum Range Organics (FL-PRO)	1/23/08	Yes	Complete
162	Organics SOP # 3.3.49	B	1,4-Dioxane 8270 (SIM)	1/23/08	Yes	Complete
163	Organics SOP # 3.3.50	B	1,4-Dioxane 8260	1/23/08	Yes	Complete
164	Organics SOP # 3.3.51	-	Total Halogens in Oil Products by Oxidative Combustion and Microcoulometry-EPA Method 9076	3/23/06	No	In process
165	Radon SOP # 3.6.1	F	Radon Analysis air	5/29/07	Yes	Complete
166	Radon SOP # 3.6.2	F	Radon Analysis Water	5/29/07	Yes	Complete
167	Radon SOP # 3.6.3	F	Radioactive Sample Receipt	5/29/07	Yes	Complete
168	Radon SOP # 3.6.4	F	Radioactive Sample Return	5/29/07	Yes	Complete
169	Radon SOP # 3.6.5	F	Radiological Survey Meter Source Check	5/29/07	Yes	Complete
170	Radon SOP # 3.6.6	D	Radioactive Material Handling	5/29/07	Yes	Complete
171	Radon SOP # 3.6.7	B	Radioactive Surveys and Wipe Tests	5/29/07	Yes	Complete
172	Wet Chemistry SOP # 3.2.13	K	Total Dissolved Solids (SM 2540C/EPA 160.1)	1/23/08	Yes	Complete
173	Wet Chemistry SOP # 3.2.2	C	Alkalinity (SM2320B/310.1) Drinking Water	6/10/02 **Obsolete**	Combined with 3.2.46	Combined with 3.2.46
174	Wet Chemistry SOP # 3.2.22	C	Total & Amenable Cyanide (Distillation)	3/21/06	Yes	Complete
175	Wet Chemistry SOP # 3.2.35	D	Total & Amenable Cyanide (Manual Spectrophotometric Determination)Method 9014	10/24/06	Yes	Complete
176	Wet Chemistry SOP # 3.2.43	Removed 5/3/05	Silica Standard Methods 4500 Si F	6/6/02 **Obsolete**	Yes	No longer on line 5/3/05

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177	Wet Chemistry SOP # 3.2.36	I	Total Suspended Solids- Residue, Non-Filterable EPA 160.2/SM 2540D	4/7/08	Yes	Complete
178	Wet Chemistry SOP # 3.2.25	G	Chemical Oxygen Demand (410.1/SM5220C)	2/25/08	Yes	Complete
179	Wet Chemistry SOP # 3.2.29	D	Ammonia Nitrogen EPA 350.2/SM4500 NH3B	3/16/06	Yes	Complete
180	Wet Chemistry SOP # 3.2.7	H	Determination of Inorganic Anions by Ion Chromatography Method 9056/300.0	4/7/08	Yes	Complete
181	Wet Chemistry SOP # 3.2.37	E	Acidity Method 2310B	3/27/06	Yes	Complete
182	Wet Chemistry SOP # 3.2.9	Q	Biochemical Oxygen Demand (SM5210B)	4/9/08	Yes	Complete
183	Wet Chemistry SOP # 3.2.39	B	Total Phenols	4/6/06	Yes	Complete
184	Wet Chemistry SOP # 3.2.40	E	Nitrogen, Total Kjeldahl (SM 4500 Norg C)	2/25/08	Yes	Complete
185	Organics SOP # 3.2.23	E	Oil and Grease (EPA 1664A)	4/8/08	Yes	Complete
186	Wet Chemistry SOP # 3.2.41	F	pH Value (150.1/SM4500 H+ B)	1/23/08	Yes	Complete
187	Wet Chemistry SOP # 3.2.42	F	Soil & Waste pH (EPA 9045/EPA 9040B)	1/23/08	Yes	Complete
188	Wet Chemistry SOP # 3.2.30	H	Phosphorus (SM 4500 P E)	2/25/08	Yes	Complete
189	Wet Chemistry SOP # 3.2.33	D	Sulfide (SM 4500 S2 F)	6/25/07	Yes	Complete
190	Metals SOP # 3.2.18	E	Turbidity (180.1)	1/28/08	Yes	Complete
191	Wet Chemistry SOP # 3.2.24	E	Total Nitrate and Nitrite Nitrogen-Cadmium Reduction (353.2)	1/31/08	Yes	Complete
192	Wet Chemistry SOP # 3.2.19	B	Bromide	6/10/02 **Obsolete**	Yes	Complete
193	Wet Chemistry SOP # 3.2.31	D	Turbidimetric Sulfate (375.4)	6/9/06 **Obsolete**	Yes	Complete
194	Wet Chemistry SOP # 3.2.32	B	Gravimetric Sulfate	6/10/02 **Obsolete**	No longer using	No longer using
195	Wet Chemistry SOP # 3.2.44	A	Fluoride	2/9/05 **Obsolete**	Yes	Complete
196	Wet Chemistry SOP # 3.2.45	E	Ammonia Nitrogen (350.1)	1/23/08	Yes	Complete
197	Wet Chemistry SOP # 3.2.46	F	Alkalinity Standard Methods (EPA 310.1)	3/11/08	Yes	Complete
198	Wet Chemistry SOP # 3.2.47	B	Ferrous Iron	7/13/07	Yes	Complete

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199	Wet Chemistry SOP # 3.2.48	-	Salinity SM 2520B	1/15/08	No	In process
200	Wet Chemistry SOP # 3.2.49	B	Dissolved Oxygen SM 4500 G	6/19/07	Yes	Complete
201	Wet Chemistry SOP # 3.2.50	D	Tannin SM 5550B	6/18/07	Yes	Complete
202	Wet Chemistry SOP # 3.2.51	F	Total Solids (SM2540B/ EPA 160.3)	1/23/08	Yes	Complete
203	Wet Chemistry SOP # 3.2.52	F	Volatile Solids (SM2540E/EPA 160.4)	1/23/08	Yes	Complete
204	Wet Chemistry SOP # 3.2.53	F	Settleable Solids (SM2540F/ EPA 160.5)	1/23/08	Yes	Complete
205	Wet Chemistry SOP # 3.2.54	C	Specific Conductance (120.1/SM2510B)	6/19/07	Yes	Complete
206	Wet Chemistry SOP # 3.2.55	A	Surfactants Standard Methods (SM5540C)	1/22/02	No	In process
207	Wet Chemistry SOP # 3.2.58	B	Total Hardness	3/10/05 **Obsolete**	Yes	Complete
208	Wet Chemistry SOP # 3.2.70	B	Total Chloride (300.0 and 325.2)	1/23/08	Yes	Complete
209	Wet Chemistry SOP # 3.2.66	[illegible]	Temperature	3/25/02 **Obsolete**	No	In process
210	Wet Chemistry SOP # 3.2.67	A	Total Chlorine (SM4500 Cl D)	3/31/05	Yes	Complete
211	Wet Chemistry SOP # 3.2.68	B	Oil & Grease	7/3/06	Yes	Complete
212	Wet Chemistry SOP # 3.2.69	B	Total Chloride	3/22/06	Yes	Complete
213	Wet Chemistry SOP # 3.2.21	F	Total Organic Carbon 415.1/9060/SM5310C	2/25/08	Yes	Complete
214	Wet Chemistry SOP # 3.2.78	A	Salt in Food with Calculations for Water Phase Salt in Fish	1/23/08	Yes	Complete
215	Wet Chemistry SOP # 3.2.80	-	Sulfite (376.1)	8/2/06	Yes	Complete
216	Wet Chemistry SOP # 3.2.81	A	Volatile Suspended Solids	1/23/08	Yes	Complete
217	Wet Chemistry SOP # 3.2.82	A	Orthophosphorous in Wastewater -- EPA Method 365.2/SM 4500 P E	02/25/08	Yes	Complete
218	Wet Chemistry SOP # 3.2.83	-	TOC in Soil EPA 9060/Kahn 1988	1/15/08	Yes	Complete
218	LQAP	19	Laboratory Quality Assurance Program Manual	11/07	Yes	Complete
219	Micro Air Training Manual	4	IAQ Training Manual	11/07		

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220	Microbiology Quality Policies	7	Microbiology Quality Policies according to ISO/IEC 17025	8/07		
221	Facility SOP # 1.303	B	Housekeeping Duties and Schedule for Areas A (Analytical) & B (Media/Micro/Admin/Shipping)	1/07		
222	Facility SOP # 1.304	-	Rodent Prevention and Entrapment	08/01		
223	Facility SOP # 2.100	-	Building Evacuation Procedures	10/01		

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NELAP RECOGNIZED ACCREDITING AUTHORITY nelap

FLORIDA DEPARTMENT OF HEALTH

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State of Florida

Department of Health, Bureau of Laboratories

This is to certify that

E87820
NORTHEAST LABORATORY SERVICES, INC.
227 CHINA ROAD
WINSLOW, ME 04901

has complied with Florida Administrative Code 64E-1,
for the examination of Environmental samples in the following categories

DRINKING WATER - GROUP II UNREGULATED CONTAMINANTS, DRINKING WATER - MICROBIOLOGY, DRINKING WATER - OTHER REGULATED CONTAMINANTS, DRINKING WATER - PRIMARY INORGANIC CONTAMINANTS, DRINKING WATER - SECONDARY INORGANIC CONTAMINANTS, NON-POTABLE WATER - EXTRACTABLE ORGANICS, NON-POTABLE WATER - GENERAL CHEMISTRY, NON-POTABLE WATER - METALS, NON-POTABLE WATER - MICROBIOLOGY, NON-POTABLE WATER - PESTICIDES-HERBICIDES-PCB'S, NON-POTABLE WATER - VOLATILE ORGANICS, SOLID AND CHEMICAL MATERIALS - EXTRACTABLE ORGANICS, SOLID AND CHEMICAL MATERIALS - PESTICIDES-HERBICIDES-PCB'S, SOLID AND CHEMICAL MATERIALS - GENERAL CHEMISTRY, SOLID AND CHEMICAL MATERIALS - METALS, SOLID AND CHEMICAL MATERIALS - MICROBIOLOGY, SOLID AND CHEMICAL MATERIALS - VOLATILE ORGANICS

Continued certification is contingent upon successful on-going compliance with the NELAC Standards and FAC Rule 64E-1 regulations. Specific methods and analytes certified are cited on the Laboratory Scope of Accreditation for this laboratory and are on file at the Bureau of Laboratories, P. O. Box 210, Jacksonville, Florida 32231. Clients and customers are urged to verify with this agency the laboratory's certification status in Florida for particular methods and analytes.

EFFECTIVE July 01, 2007 THROUGH June 30, 2008

GREAT SEAL OF THE STATE OF FLORIDA — IN GOD WE TRUST

Max Salfinger, M.D.
Chief, Bureau of Laboratories
Florida Department of Health
DH Form 1697, 7/04
NON-TRANSFERABLE E87820-18-7/1/2007
Supersedes all previously issued certificates

Laboratory Scope of Accreditation

Attachment to Certificate #: E87820-18, expiration date June 30, 2008. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87820 **EPA Lab Code: ME00009** **(207) 873-7711**

E87820
Northeast Laboratory Services, Inc.
227 China Road
Winslow, ME 04901

Matrix: Drinking Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1-Trichloroethane	EPA 524.2	Other Regulated Contaminants	NELAP	4/24/2002
1,1,2,2-Tetrachloroethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	5/31/2007
1,1,2-Trichloroethane	EPA 524.2	Other Regulated Contaminants	NELAP	4/24/2002
1,1-Dichloroethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	5/31/2007
1,1-Dichloroethylene	EPA 524.2	Other Regulated Contaminants	NELAP	12/17/2004
1,1-Dichloropropene	EPA 524.2	Group II Unregulated Contaminants	NELAP	8/21/2002
1,2,3-Trichloropropane	EPA 524.2	Group II Unregulated Contaminants	NELAP	5/31/2007
1,2,4-Trichlorobenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	4/24/2002
1,2,4-Trimethylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	12/17/2004
1,2-Dibromo-3-chloropropane (DBCP)	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/28/2004
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/28/2004
1,2-Dichlorobenzene	EPA 524.2	Other Regulated Contaminants	NELAP	12/17/2004
1,2-Dichloroethane	EPA 524.2	Other Regulated Contaminants	NELAP	4/24/2002
1,2-Dichloropropane	EPA 524.2	Other Regulated Contaminants	NELAP	4/24/2002
1,3,5-Trimethylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	12/17/2004
1,3-Dichlorobenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	12/17/2004
1,3-Dichloropropane	EPA 524.2	Group II Unregulated Contaminants	NELAP	4/24/2002
1,4-Dichlorobenzene	EPA 524.2	Other Regulated Contaminants	NELAP	4/24/2002
1-Chlorobutane	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/28/2004
2,2-Dichloropropane	EPA 524.2	Group II Unregulated Contaminants	NELAP	4/24/2002
2-Butanone (Methyl ethyl ketone, MEK)	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/28/2004
2-Hexanone	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/28/2004
2-Nitropropane	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/28/2004
4-Chlorotoluene	EPA 524.2	Group II Unregulated Contaminants	NELAP	4/24/2002
4-Isopropyltoluene	EPA 524.2	Group II Unregulated Contaminants	NELAP	12/17/2004
4-Methyl-2-pentanone (MIBK)	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/28/2004
Acetone	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/28/2004
Acrylonitrile	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/28/2004
Alkalinity as CaCO3	SM 2320 B	Primary Inorganic Contaminants	NELAP	4/24/2002
Allyl chloride (3-Chloropropene)	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/28/2004
Aluminum	EPA 200.7	Secondary Inorganic Contaminants	NELAP	12/17/2004
Antimony	EPA 200.9	Primary Inorganic Contaminants	NELAP	6/24/2002
Antimony	SM 3113 B	Primary Inorganic Contaminants	NELAP	4/24/2002
Arsenic	EPA 200.9	Primary Inorganic Contaminants	NELAP	6/24/2002
Arsenic	SM 3113 B	Primary Inorganic Contaminants	NELAP	4/24/2002
Barium	EPA 200.7	Primary Inorganic Contaminants	NELAP	4/24/2002

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Laboratory Scope of Accreditation

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State Laboratory ID: E87820 **EPA Lab Code: ME00009** **(207) 873-7711**

E87820
Northeast Laboratory Services, Inc.
227 China Road
Winslow, ME 04901

Matrix: Drinking Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Boron	EPA 200.7	Secondary Inorganic Contaminants	NELAP	12/15/2006
Bromide	EPA 300.0	Primary Inorganic Contaminants	NELAP	12/15/2006
Bromobenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	12/17/2004
Bromochloromethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	4/24/2002
Bromodichloromethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	1/19/2005
Bromoform	EPA 524.2	Group II Unregulated Contaminants	NELAP	4/24/2002
Cadmium	EPA 200.7	Primary Inorganic Contaminants	NELAP	4/24/2002
Calcium	EPA 200.7	Primary Inorganic Contaminants	NELAP	4/24/2002
Carbon disulfide	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/28/2004
Carbon tetrachloride	EPA 524.2	Other Regulated Contaminants	NELAP	5/31/2007
Chloride	EPA 300.0	Secondary Inorganic Contaminants	NELAP	3/25/2005
Chloride	EPA 325.2	Secondary Inorganic Contaminants	NELAP	7/28/2004
Chlorobenzene	EPA 524.2	Other Regulated Contaminants	NELAP	12/17/2004
Chloroethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	4/24/2002
Chloroform	EPA 524.2	Group II Unregulated Contaminants	NELAP	12/17/2004
Chromium	EPA 200.7	Primary Inorganic Contaminants	NELAP	4/24/2002
cis-1,3-Dichloropropene	EPA 524.2	Group II Unregulated Contaminants	NELAP	8/21/2002
Color	EPA 110.2	Secondary Inorganic Contaminants	NELAP	4/24/2002
Conductivity	SM 2510 B	Primary Inorganic Contaminants	NELAP	4/24/2002
Copper	EPA 200.7	Primary Inorganic Contaminants,Secondary Inorganic Contaminants	NELAP	4/24/2002
Dibromochloromethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	4/24/2002
Dibromomethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	12/17/2004
Dichlorodifluoromethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	4/24/2002
Dichloromethane (DCM, Methylene chloride)	EPA 524.2	Other Regulated Contaminants	NELAP	12/17/2004
Diethyl ether	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/28/2004
Di-isopropylether (DIPE)	NEL SOP 3.3.26, Rev. D/GC-MS	Group II Unregulated Contaminants	NELAP	12/15/2006
Ethyl methacrylate	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/28/2004
Ethylbenzene	EPA 524.2	Other Regulated Contaminants	NELAP	4/24/2002
Ethyl-t-butylether (ETBE)	NEL SOP 3.3.26, Rev. D/GC-MS	Group II Unregulated Contaminants	NELAP	12/15/2006
Fecal coliforms	SM 9222 D	Microbiology	NELAP	7/28/2004
Fluoride	EPA 300.0	Primary Inorganic Contaminants,Secondary Inorganic Contaminants	NELAP	4/24/2002
Hardness	SM 2340 B	Secondary Inorganic Contaminants	NELAP	12/15/2006
Heterotrophic plate count	SM 9215 B	Microbiology	NELAP	5/16/2006

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Laboratory Scope of Accreditation

Attachment to Certificate #: E87820-18, expiration date June 30, 2008. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87820 **EPA Lab Code: ME00009** **(207) 873-7711**

E87820
Northeast Laboratory Services, Inc.
227 China Road
Winslow, ME 04901

Matrix: Drinking Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Hexachlorobutadiene	EPA 524.2	Group II Unregulated Contaminants	NELAP	4/24/2002
Hexachloroethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/28/2004
Iodomethane (Methyl iodide)	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/28/2004
Iron	EPA 200.7	Secondary Inorganic Contaminants	NELAP	4/24/2002
Isopropylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	4/24/2002
Lead	EPA 200.9	Primary Inorganic Contaminants	NELAP	6/24/2002
Lead	SM 3113 B	Primary Inorganic Contaminants	NELAP	4/24/2002
m/p-Xylenes	EPA 524.2	Group II Unregulated Contaminants	NELAP	12/15/2006
Magnesium	EPA 200.7	Primary Inorganic Contaminants	NELAP	4/24/2002
Manganese	EPA 200.7	Secondary Inorganic Contaminants	NELAP	5/31/2007
Mercury	EPA 245.1	Primary Inorganic Contaminants	NELAP	4/24/2002
Methacrylonitrile	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/28/2004
Methyl bromide (Bromomethane)	EPA 524.2	Group II Unregulated Contaminants	NELAP	12/15/2006
Methyl chloride (Chloromethane)	EPA 524.2	Group II Unregulated Contaminants	NELAP	12/15/2006
Methyl methacrylate	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/28/2004
Methyl tert-butyl ether (MTBE)	EPA 524.2	Group II Unregulated Contaminants	NELAP	4/24/2002
Naphthalene	EPA 524.2	Group II Unregulated Contaminants	NELAP	4/24/2002
n-Butylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	12/17/2004
Nickel	EPA 200.7	Primary Inorganic Contaminants	NELAP	4/24/2002
Nitrate as N	EPA 300.0	Primary Inorganic Contaminants	NELAP	4/24/2002
Nitrate as N	EPA 353.2	Primary Inorganic Contaminants	NELAP	3/20/2006
Nitrite as N	EPA 300.0	Primary Inorganic Contaminants	NELAP	6/1/2007
Nitrite as N	EPA 353.2	Primary Inorganic Contaminants	NELAP	4/24/2002
Nitrobenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	4/24/2002
Orthophosphate as P	SM 4500-P E	Primary Inorganic Contaminants	NELAP	4/24/2002
o-Xylene	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/28/2004
Pentachloroethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/28/2004
pH	EPA 150.1	Primary Inorganic Contaminants,Secondary Inorganic Contaminants	NELAP	4/24/2002
Potassium	EPA 200.7	Secondary Inorganic Contaminants	NELAP	7/28/2004
Propionitrile (Ethyl cyanide)	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/28/2004
Residue-filterable (TDS)	EPA 160.1	Secondary Inorganic Contaminants	NELAP	7/28/2004
Residue-filterable (TDS)	SM 2540 C	Secondary Inorganic Contaminants	NELAP	4/24/2002
Selenium	EPA 200.9	Primary Inorganic Contaminants	NELAP	6/24/2002
Selenium	SM 3113 B	Primary Inorganic Contaminants	NELAP	4/24/2002
Silica as SiO2	EPA 200.7	Primary Inorganic Contaminants	NELAP	7/28/2004

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Laboratory Scope of Accreditation

Attachment to Certificate #: E87820-18, expiration date June 30, 2008. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87820 **EPA Lab Code: ME00009** **(207) 873-7711**

E87820
Northeast Laboratory Services, Inc.
227 China Road
Winslow, ME 04901

Matrix: Drinking Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Silver	EPA 200.7	Secondary Inorganic Contaminants	NELAP	12/15/2006
Sodium	EPA 200.7	Primary Inorganic Contaminants	NELAP	7/28/2004
Styrene	EPA 524.2	Other Regulated Contaminants	NELAP	12/17/2004
Sulfate	EPA 300.0	Primary Inorganic Contaminants,Secondary Inorganic Contaminants	NELAP	4/24/2002
Sulfate	EPA 375.4	Secondary Inorganic Contaminants	NELAP	12/15/2006
T-amylmethylether (TAME)	NEL SOP 3.3.26, Rev. D/GC-MS	Group II Unregulated Contaminants	NELAP	12/15/2006
tert-Butyl alcohol	NEL SOP 3.3.26, Rev. D/GC-MS	Group II Unregulated Contaminants	NELAP	12/15/2006
tert-Butylbenzene	EPA 524.2	Group II Unregulated Contaminants	NELAP	12/17/2004
Tetrachloroethylene (Perchloroethylene)	EPA 524.2	Other Regulated Contaminants	NELAP	12/17/2004
Tetrahydrofuran (THF)	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/28/2004
Thallium	EPA 200.9	Primary Inorganic Contaminants	NELAP	4/24/2002
Toluene	EPA 524.2	Other Regulated Contaminants	NELAP	4/24/2002
Total coliforms	SM 9221 B	Microbiology	NELAP	7/28/2004
Total coliforms	SM 9222 B	Microbiology	NELAP	6/24/2002
Total coliforms & E. coli	MI Agar	Microbiology	NELAP	4/24/2002
Total coliforms & E. coli	SM 9223 B	Microbiology	NELAP	12/15/2006
Total nitrate-nitrite	EPA 353.2	Primary Inorganic Contaminants	NELAP	4/24/2002
Total trihalomethanes	EPA 524.2	Other Regulated Contaminants	NELAP	12/17/2004
trans-1,2-Dichloroethylene	EPA 524.2	Other Regulated Contaminants	NELAP	5/31/2007
trans-1,3-Dichloropropylene	EPA 524.2	Group II Unregulated Contaminants	NELAP	4/24/2002
trans-1,4-Dichloro-2-butene	EPA 524.2	Group II Unregulated Contaminants	NELAP	7/28/2004
Trichloroethene (Trichloroethylene)	EPA 524.2	Other Regulated Contaminants	NELAP	4/24/2002
Trichlorofluoromethane	EPA 524.2	Group II Unregulated Contaminants	NELAP	4/24/2002
Turbidity	EPA 180.1	Secondary Inorganic Contaminants	NELAP	4/24/2002
Vanadium	EPA 200.7	Secondary Inorganic Contaminants	NELAP	12/15/2006
Xylene (total)	EPA 524.2	Other Regulated Contaminants	NELAP	12/17/2004
Zinc	EPA 200.7	Secondary Inorganic Contaminants	NELAP	12/17/2004

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State Laboratory ID: E87820 **EPA Lab Code: ME00009** **(207) 873-7711**

E87820
Northeast Laboratory Services, Inc.
227 China Road
Winslow, ME 04901

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	7/28/2004
1,1,1-Trichloroethane	EPA 624	Volatile Organics	NELAP	4/24/2002
1,1,1-Trichloroethane	EPA 8260	Volatile Organics	NELAP	7/28/2004
1,1,2,2-Tetrachloroethane	EPA 624	Volatile Organics	NELAP	4/24/2002
1,1,2,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	7/28/2004
1,1,2-Trichloroethane	EPA 624	Volatile Organics	NELAP	4/24/2002
1,1,2-Trichloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1-Dichloroethane	EPA 624	Volatile Organics	NELAP	4/24/2002
1,1-Dichloroethane	EPA 8260	Volatile Organics	NELAP	7/28/2004
1,1-Dichloroethylene	EPA 624	Volatile Organics	NELAP	4/24/2002
1,1-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,1-Dichloropropene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2,3-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2,3-Trichloropropane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2,4-Trichlorobenzene	EPA 625	Extractable Organics	NELAP	8/31/2006
1,2,4-Trichlorobenzene	EPA 8270	Extractable Organics	NELAP	8/31/2006
1,2,4-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	4/24/2002
1,2-Dichlorobenzene	EPA 625	Extractable Organics	NELAP	4/24/2002
1,2-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/28/2004
1,2-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/28/2004
1,2-Dichloroethane	EPA 624	Volatile Organics	NELAP	4/24/2002
1,2-Dichloroethane	EPA 8260	Volatile Organics	NELAP	7/28/2004
1,2-Dichloropropane	EPA 624	Volatile Organics	NELAP	4/24/2002
1,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,2-Diphenylhydrazine	EPA 8270	Extractable Organics	NELAP	7/1/2003
1,3,5-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,3-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	4/24/2002
1,3-Dichlorobenzene	EPA 625	Extractable Organics	NELAP	4/24/2002
1,3-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/28/2004
1,3-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/28/2004
1,3-Dichloropropane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1,4-Dichlorobenzene	EPA 624	Volatile Organics	NELAP	6/9/2005
1,4-Dichlorobenzene	EPA 625	Extractable Organics	NELAP	4/24/2002

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Laboratory Scope of Accreditation

Attachment to Certificate #: E87820-18, expiration date June 30, 2008. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87820 **EPA Lab Code: ME00009** **(207) 873-7711**

E87820
Northeast Laboratory Services, Inc.
227 China Road
Winslow, ME 04901

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,4-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/28/2004
1,4-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/28/2004
1-Chlorobutane	EPA 8260	Volatile Organics	NELAP	7/1/2003
1-Chloronaphthalene	EPA 8270	Extractable Organics	NELAP	7/28/2004
2,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	7/1/2003
2,4,5-T	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,4,5-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4,6-Trichlorophenol	EPA 625	Extractable Organics	NELAP	4/24/2002
2,4,6-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-D	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,4-DB	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
2,4-Dichlorophenol	EPA 625	Extractable Organics	NELAP	4/24/2002
2,4-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dimethylphenol	EPA 625	Extractable Organics	NELAP	4/24/2002
2,4-Dimethylphenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dinitrophenol	EPA 625	Extractable Organics	NELAP	4/24/2002
2,4-Dinitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,4-Dinitrotoluene (2,4-DNT)	EPA 625	Extractable Organics	NELAP	4/24/2002
2,4-Dinitrotoluene (2,4-DNT)	EPA 8270	Extractable Organics	NELAP	7/1/2003
2,6-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	7/28/2004
2,6-Dinitrotoluene (2,6-DNT)	EPA 625	Extractable Organics	NELAP	3/23/2006
2,6-Dinitrotoluene (2,6-DNT)	EPA 8270	Extractable Organics	NELAP	3/23/2006
2-Butanone (Methyl ethyl ketone, MEK)	EPA 8260	Volatile Organics	NELAP	7/1/2003
2-Butanone (Methyl ethyl ketone, MEK)	NE SOP 3.3.32 Rev. A	Volatile Organics	NELAP	7/26/2002
2-Chloroethyl vinyl ether	EPA 624	Volatile Organics	NELAP	4/24/2002
2-Chloroethyl vinyl ether	EPA 8260	Volatile Organics	NELAP	7/1/2003
2-Chloronaphthalene	EPA 625	Extractable Organics	NELAP	3/23/2006
2-Chloronaphthalene	EPA 8270	Extractable Organics	NELAP	3/23/2006
2-Chlorophenol	EPA 625	Extractable Organics	NELAP	4/24/2002
2-Chlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	7/1/2003
2-Hexanone	EPA 8260	Volatile Organics	NELAP	7/1/2003
2-Methyl-4,6-dinitrophenol	EPA 625	Extractable Organics	NELAP	4/24/2002
2-Methyl-4,6-dinitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Methylnaphthalene	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Methylphenol (o-Cresol)	EPA 8270	Extractable Organics	NELAP	7/1/2003

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State Laboratory ID: E87820 **EPA Lab Code: ME00009** **(207) 873-7711**

E87820
Northeast Laboratory Services, Inc.
227 China Road
Winslow, ME 04901

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
2-Nitroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Nitrophenol	EPA 625	Extractable Organics	NELAP	4/24/2002
2-Nitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
2-Nitropropane	EPA 8260	Volatile Organics	NELAP	7/1/2003
3,3'-Dichlorobenzidine	EPA 625	Extractable Organics	NELAP	4/24/2002
3,3'-Dichlorobenzidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
3/4-Methylphenols (m/p-Cresols)	EPA 8270	Extractable Organics	NELAP	12/15/2006
3-Nitroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
4,4'-DDD	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/14/2005
4,4'-DDD	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/14/2005
4,4'-DDE	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/14/2005
4,4'-DDE	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/14/2005
4,4'-DDT	EPA 608	Pesticides-Herbicides-PCB's	NELAP	12/15/2006
4,4'-DDT	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	12/15/2006
4-Bromophenyl phenyl ether	EPA 625	Extractable Organics	NELAP	3/23/2006
4-Bromophenyl phenyl ether	EPA 8270	Extractable Organics	NELAP	3/23/2006
4-Chloro-3-methylphenol	EPA 625	Extractable Organics	NELAP	4/24/2002
4-Chloro-3-methylphenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Chloroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Chlorophenyl phenylether	EPA 625	Extractable Organics	NELAP	4/24/2002
4-Chlorophenyl phenylether	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	7/1/2003
4-Methyl-2-pentanone (MIBK)	EPA 8260	Volatile Organics	NELAP	7/28/2004
4-Methyl-2-pentanone (MIBK)	NE SOP 3.3.32 Rev. A	Volatile Organics	NELAP	7/26/2002
4-Nitroaniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
4-Nitrophenol	EPA 625	Extractable Organics	NELAP	4/24/2002
4-Nitrophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
Acenaphthene	EPA 625	Extractable Organics	NELAP	4/24/2002
Acenaphthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Acenaphthylene	EPA 625	Extractable Organics	NELAP	4/24/2002
Acenaphthylene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Acetone	EPA 8260	Volatile Organics	NELAP	7/1/2003
Acidity, as CaCO3	SM 2310 B (4A)	General Chemistry	NELAP	4/24/2002
Acrolein (Propenal)	EPA 624	Volatile Organics	NELAP	4/24/2002
Acrolein (Propenal)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Acrylonitrile	EPA 624	Volatile Organics	NELAP	4/24/2002

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Laboratory Scope of Accreditation

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State Laboratory ID: E87820 **EPA Lab Code: ME00009** **(207) 873-7711**

E87820
Northeast Laboratory Services, Inc.
227 China Road
Winslow, ME 04901

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Acrylonitrile	EPA 8260	Volatile Organics	NELAP	7/1/2003
Adsorbable organic halogens (AOX)	EPA 1650	General Chemistry	NELAP	4/24/2002
Aldrin	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/14/2005
Aldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/14/2005
Alkalinity as CaCO3	EPA 310.1	General Chemistry	NELAP	4/24/2002
Alkalinity as CaCO3	SM 2320 B	General Chemistry	NELAP	4/24/2002
Allyl chloride (3-Chloropropene)	EPA 8260	Volatile Organics	NELAP	7/1/2003
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/24/2002
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
alpha-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aluminum	EPA 200.7	Metals	NELAP	4/24/2002
Aluminum	EPA 6010	Metals	NELAP	7/1/2003
Ammonia as N	EPA 350.1	General Chemistry	NELAP	4/24/2002
Ammonia as N	EPA 350.2	General Chemistry	NELAP	4/24/2002
Ammonia as N	SM 4500-NH3C	General Chemistry	NELAP	5/2/2007
Aniline	EPA 625	Extractable Organics	NELAP	12/15/2006
Aniline	EPA 8270	Extractable Organics	NELAP	7/1/2003
Anthracene	EPA 625	Extractable Organics	NELAP	4/24/2002
Anthracene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Antimony	EPA 200.7	Metals	NELAP	4/24/2002
Antimony	EPA 6010	Metals	NELAP	7/1/2003
Antimony	EPA 7041	Metals	NELAP	7/28/2004
Antimony	SM 3113 B	Metals	NELAP	12/15/2006
Aroclor-1016 (PCB-1016)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	3/23/2006
Aroclor-1016 (PCB-1016)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	3/23/2006
Aroclor-1221 (PCB-1221)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/24/2002
Aroclor-1221 (PCB-1221)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1232 (PCB-1232)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	1/10/2005
Aroclor-1232 (PCB-1232)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	1/10/2005
Aroclor-1242 (PCB-1242)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	3/23/2006
Aroclor-1242 (PCB-1242)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	3/23/2006
Aroclor-1248 (PCB-1248)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/24/2002
Aroclor-1248 (PCB-1248)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1254 (PCB-1254)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/24/2002
Aroclor-1254 (PCB-1254)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Aroclor-1260 (PCB-1260)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/24/2002

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Laboratory Scope of Accreditation

Attachment to Certificate #: E87820-18, expiration date June 30, 2008. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87820 **EPA Lab Code: ME00009** **(207) 873-7711**

E87820
Northeast Laboratory Services, Inc.
227 China Road
Winslow, ME 04901

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Aroclor-1260 (PCB-1260)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Arsenic	EPA 200.7	Metals	NELAP	4/24/2002
Arsenic	EPA 6010	Metals	NELAP	4/24/2002
Arsenic	EPA 7060	Metals	NELAP	7/28/2004
Arsenic	SM 3113 B	Metals	NELAP	7/28/2004
Barium	EPA 200.7	Metals	NELAP	4/24/2002
Barium	EPA 6010	Metals	NELAP	7/1/2003
Benzene	EPA 624	Volatile Organics	NELAP	4/24/2002
Benzene	EPA 8260	Volatile Organics	NELAP	7/28/2004
Benzidine	EPA 625	Extractable Organics	NELAP	4/24/2002
Benzidine	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(a)anthracene	EPA 625	Extractable Organics	NELAP	4/24/2002
Benzo(a)anthracene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(a)pyrene	EPA 625	Extractable Organics	NELAP	4/24/2002
Benzo(a)pyrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(b)fluoranthene	EPA 625	Extractable Organics	NELAP	4/24/2002
Benzo(b)fluoranthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(g,h,i)perylene	EPA 625	Extractable Organics	NELAP	4/24/2002
Benzo(g,h,i)perylene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzo(k)fluoranthene	EPA 625	Extractable Organics	NELAP	4/24/2002
Benzo(k)fluoranthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzoic acid	EPA 8270	Extractable Organics	NELAP	7/1/2003
Benzyl alcohol	EPA 8270	Extractable Organics	NELAP	7/1/2003
Beryllium	EPA 200.7	Metals	NELAP	4/24/2002
Beryllium	EPA 6010	Metals	NELAP	7/1/2003
beta-BHC (beta-Hexachlorocyclohexane)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	1/10/2005
beta-BHC (beta-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	1/10/2005
Biochemical oxygen demand	EPA 405.1	General Chemistry	NELAP	7/28/2004
Biochemical oxygen demand	SM 5210 B	General Chemistry	NELAP	4/24/2002
bis(2-Chloroethoxy)methane	EPA 625	Extractable Organics	NELAP	3/23/2006
bis(2-Chloroethoxy)methane	EPA 8270	Extractable Organics	NELAP	3/23/2006
bis(2-Chloroethyl) ether	EPA 625	Extractable Organics	NELAP	4/24/2002
bis(2-Chloroethyl) ether	EPA 8270	Extractable Organics	NELAP	7/1/2003
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 625	Extractable Organics	NELAP	4/24/2002
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 8270	Extractable Organics	NELAP	7/1/2003

Laboratory Scope of Accreditation

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State Laboratory ID: E87820 **EPA Lab Code: ME00009** **(207) 873-7711**

E87820
Northeast Laboratory Services, Inc.
227 China Road
Winslow, ME 04901

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 625	Extractable Organics	NELAP	4/24/2002
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 8270	Extractable Organics	NELAP	7/1/2003
Boron	EPA 6010	Metals	NELAP	7/1/2003
Bromide	EPA 300.0	General Chemistry	NELAP	4/24/2002
Bromide	EPA 9056	General Chemistry	NELAP	7/1/2003
Bromobenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Bromochloromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Bromodichloromethane	EPA 624	Volatile Organics	NELAP	4/24/2002
Bromodichloromethane	EPA 8260	Volatile Organics	NELAP	7/28/2004
Bromoform	EPA 624	Volatile Organics	NELAP	4/24/2002
Bromoform	EPA 8260	Volatile Organics	NELAP	7/28/2004
Butyl benzyl phthalate	EPA 625	Extractable Organics	NELAP	4/24/2002
Butyl benzyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Cadmium	EPA 200.7	Metals	NELAP	4/24/2002
Cadmium	EPA 6010	Metals	NELAP	4/24/2002
Calcium	EPA 200.7	Metals	NELAP	4/24/2002
Calcium	EPA 6010	Metals	NELAP	7/1/2003
Carbazole	EPA 625	Extractable Organics	NELAP	12/15/2006
Carbazole	EPA 8270	Extractable Organics	NELAP	7/1/2003
Carbon disulfide	EPA 8260	Volatile Organics	NELAP	7/1/2003
Carbon tetrachloride	EPA 624	Volatile Organics	NELAP	4/24/2002
Carbon tetrachloride	EPA 8260	Volatile Organics	NELAP	7/28/2004
Carbonaceous BOD (CBOD)	SM 5210 B	General Chemistry	NELAP	4/24/2002
Chemical oxygen demand	EPA 410.1	General Chemistry	NELAP	7/28/2004
Chemical oxygen demand	SM 5220 C	General Chemistry	NELAP	4/24/2002
Chlordane (tech.)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/24/2002
Chlordane (tech.)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Chloride	EPA 300.0	General Chemistry	NELAP	1/10/2005
Chloride	EPA 325.2	General Chemistry	NELAP	4/24/2002
Chloride	EPA 9056	General Chemistry	NELAP	1/10/2005
Chlorobenzene	EPA 624	Volatile Organics	NELAP	4/24/2002
Chlorobenzene	EPA 8260	Volatile Organics	NELAP	7/28/2004
Chloroethane	EPA 624	Volatile Organics	NELAP	6/9/2005
Chloroethane	EPA 8260	Volatile Organics	NELAP	7/28/2004
Chloroform	EPA 624	Volatile Organics	NELAP	4/24/2002
Chloroform	EPA 8260	Volatile Organics	NELAP	7/28/2004

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Laboratory Scope of Accreditation

Attachment to Certificate #: E87820-18, expiration date June 30, 2008. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87820 **EPA Lab Code: ME00009** **(207) 873-7711**

E87820
Northeast Laboratory Services, Inc.
227 China Road
Winslow, ME 04901

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Chromium	EPA 200.7	Metals	NELAP	4/24/2002
Chromium	EPA 6010	Metals	NELAP	4/24/2002
Chromium VI	EPA 7196	General Chemistry	NELAP	12/15/2006
Chrysene	EPA 625	Extractable Organics	NELAP	4/24/2002
Chrysene	EPA 8270	Extractable Organics	NELAP	7/1/2003
cis-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	7/1/2003
cis-1,3-Dichloropropene	EPA 624	Volatile Organics	NELAP	4/24/2002
cis-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Cobalt	EPA 200.7	Metals	NELAP	4/24/2002
Cobalt	EPA 6010	Metals	NELAP	7/1/2003
Color	EPA 110.2	General Chemistry	NELAP	6/24/2002
Color	SM 2120 B	General Chemistry	NELAP	5/2/2007
Conductivity	EPA 120.1	General Chemistry	NELAP	4/24/2002
Conductivity	SM 2510 B	General Chemistry	NELAP	7/28/2004
Copper	EPA 200.7	Metals	NELAP	4/24/2002
Copper	EPA 6010	Metals	NELAP	4/24/2002
Cyanide	SM 4500-CN E	General Chemistry	NELAP	12/15/2006
Dalapon	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
delta-BHC	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/24/2002
delta-BHC	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dibenz(a,h) anthracene	EPA 625	Extractable Organics	NELAP	4/24/2002
Dibenz(a,h) anthracene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Dibenzofuran	EPA 8270	Extractable Organics	NELAP	7/1/2003
Dibromochloromethane	EPA 624	Volatile Organics	NELAP	4/24/2002
Dibromochloromethane	EPA 8260	Volatile Organics	NELAP	7/28/2004
Dibromomethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Dicamba	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dichlorodifluoromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Dichloroprop (Dichlorprop)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Dieldrin	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/14/2005
Dieldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/14/2005
Diesel range organics (DRO)	CT-ETPH	Extractable Organics	NELAP	7/28/2004
Diesel range organics (DRO)	EPA 8015	Extractable Organics	NELAP	7/28/2004
Diesel range organics (DRO)	MEDEP 4.1.25	Extractable Organics	NELAP	7/1/2003
Diethyl ether	EPA 8260	Volatile Organics	NELAP	7/1/2003
Diethyl phthalate	EPA 625	Extractable Organics	NELAP	4/24/2002

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State Laboratory ID: E87820 **EPA Lab Code: ME00009** **(207) 873-7711**

E87820
Northeast Laboratory Services, Inc.
227 China Road
Winslow, ME 04901

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Diethyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Di-isopropylether (DIPE)	NEL SOP 3.3.13, Rev. I/GC-MS	Volatile Organics	NELAP	12/15/2006
Dimethyl phthalate	EPA 625	Extractable Organics	NELAP	4/24/2002
Dimethyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Di-n-butyl phthalate	EPA 625	Extractable Organics	NELAP	4/24/2002
Di-n-butyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Di-n-octyl phthalate	EPA 625	Extractable Organics	NELAP	4/24/2002
Di-n-octyl phthalate	EPA 8270	Extractable Organics	NELAP	7/1/2003
Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endosulfan I	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/24/2002
Endosulfan I	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endosulfan II	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/24/2002
Endosulfan II	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endosulfan sulfate	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/24/2002
Endosulfan sulfate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Endrin	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/14/2005
Endrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/14/2005
Endrin aldehyde	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/14/2005
Endrin aldehyde	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/14/2005
Endrin ketone	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Escherichia coli	EPA 1103.1	Microbiology	NELAP	7/26/2002
Ethyl methacrylate	EPA 8260	Volatile Organics	NELAP	7/1/2003
Ethylbenzene	EPA 624	Volatile Organics	NELAP	4/24/2002
Ethylbenzene	EPA 8260	Volatile Organics	NELAP	7/28/2004
Ethyl-t-butylether (ETBE)	NEL SOP 3.3.13, Rev. I/GC-MS	Volatile Organics	NELAP	12/15/2006
Fecal coliforms	SM 9221 E	Microbiology	NELAP	4/26/2006
Fecal coliforms	SM 9222 D	Microbiology	NELAP	4/26/2006
Fecal streptococci	SM 9230 C	Microbiology	NELAP	4/24/2002
Fluoranthene	EPA 625	Extractable Organics	NELAP	4/24/2002
Fluoranthene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Fluorene	EPA 625	Extractable Organics	NELAP	4/24/2002
Fluorene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Fluoride	EPA 300.0	General Chemistry	NELAP	6/14/2002
Fluoride	EPA 9056	General Chemistry	NELAP	7/1/2003
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/24/2002

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E87820
Northeast Laboratory Services, Inc.
227 China Road
Winslow, ME 04901

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
gamma-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Gasoline range organics (GRO)	EPA 8015	Volatile Organics	NELAP	7/28/2004
Gasoline range organics (GRO)	MEDEP 4.2.17	Extractable Organics	NELAP	7/1/2003
Hardness	SM 2340 B	General Chemistry	NELAP	7/28/2004
Hardness (calc.)	EPA 200.7	Metals	NELAP	4/24/2002
Heptachlor	EPA 608	Pesticides-Herbicides-PCB's	NELAP	12/15/2006
Heptachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	12/15/2006
Heptachlor epoxide	EPA 608	Pesticides-Herbicides-PCB's	NELAP	2/14/2005
Heptachlor epoxide	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	2/14/2005
Heterotrophic plate count	SIMPLATE	Microbiology	NELAP	12/15/2006
Heterotrophic plate count	SM 9215 B	Microbiology	NELAP	12/15/2006
Hexachlorobenzene	EPA 625	Extractable Organics	NELAP	4/24/2002
Hexachlorobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachlorobutadiene	EPA 625	Extractable Organics	NELAP	4/24/2002
Hexachlorobutadiene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Hexachlorobutadiene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Hexachlorocyclopentadiene	EPA 625	Extractable Organics	NELAP	3/23/2006
Hexachlorocyclopentadiene	EPA 8270	Extractable Organics	NELAP	3/23/2006
Hexachloroethane	EPA 625	Extractable Organics	NELAP	4/24/2002
Hexachloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Hexachloroethane	EPA 8270	Extractable Organics	NELAP	7/1/2003
Ignitability	EPA 1010	General Chemistry	NELAP	7/1/2003
Indeno(1,2,3-cd)pyrene	EPA 625	Extractable Organics	NELAP	4/24/2002
Indeno(1,2,3-cd)pyrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Iodomethane (Methyl iodide)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Iron	EPA 200.7	Metals	NELAP	8/31/2006
Iron	EPA 6010	Metals	NELAP	8/31/2006
Isophorone	EPA 625	Extractable Organics	NELAP	4/24/2002
Isophorone	EPA 8270	Extractable Organics	NELAP	7/1/2003
Isopropylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Kjeldahl nitrogen - total	EPA 351.3	General Chemistry	NELAP	4/24/2002
Kjeldahl nitrogen - total	SM 4500-NH3C	General Chemistry	NELAP	5/2/2007
Lead	EPA 200.7	Metals	NELAP	4/24/2002
Lead	EPA 6010	Metals	NELAP	4/24/2002
Lead	EPA 7421	Metals	NELAP	7/28/2004

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E87820
Northeast Laboratory Services, Inc.
227 China Road
Winslow, ME 04901

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Lead	SM 3113 B	Metals	NELAP	7/28/2004
Lithium	EPA 200.7	Metals	NELAP	7/28/2004
Lithium	EPA 6010	Metals	NELAP	7/1/2003
m/p-Xylenes	EPA 8260		NELAP	12/15/2006
Magnesium	EPA 200.7	Metals	NELAP	4/24/2002
Magnesium	EPA 6010	Metals	NELAP	7/1/2003
Manganese	EPA 200.7	Metals	NELAP	4/24/2002
Manganese	EPA 6010	Metals	NELAP	7/1/2003
Mercury	EPA 245.1	Metals	NELAP	4/24/2002
Mercury	EPA 7470	Metals	NELAP	4/24/2002
Methacrylonitrile	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methoxychlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
Methyl bromide (Bromomethane)	EPA 624	Volatile Organics	NELAP	4/24/2002
Methyl bromide (Bromomethane)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methyl chloride (Chloromethane)	EPA 624	Volatile Organics	NELAP	4/24/2002
Methyl chloride (Chloromethane)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methyl methacrylate	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methyl tert-butyl ether (MTBE)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Methyl tert-butyl ether (MTBE)	NE SOP 3.3.32 Rev. A	Volatile Organics	NELAP	7/26/2002
Methylene chloride	EPA 624	Volatile Organics	NELAP	4/24/2002
Methylene chloride	EPA 8260	Volatile Organics	NELAP	7/28/2004
Molybdenum	EPA 200.7	Metals	NELAP	4/24/2002
Molybdenum	EPA 6010	Metals	NELAP	4/24/2002
Naphthalene	EPA 625	Extractable Organics	NELAP	4/24/2002
Naphthalene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Naphthalene	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Butylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Nickel	EPA 200.7	Metals	NELAP	4/24/2002
Nickel	EPA 6010	Metals	NELAP	4/24/2002
Nitrate	EPA 9056	General Chemistry	NELAP	7/1/2003
Nitrate as N	EPA 300.0	General Chemistry	NELAP	4/24/2002
Nitrate as N	EPA 353.2	General Chemistry	NELAP	4/24/2002
Nitrite	EPA 9056	General Chemistry	NELAP	7/1/2003
Nitrite as N	EPA 300.0	General Chemistry	NELAP	4/24/2002
Nitrite as N	EPA 353.2	General Chemistry	NELAP	4/24/2002
Nitrobenzene	EPA 625	Extractable Organics	NELAP	4/24/2002

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State Laboratory ID: E87820 **EPA Lab Code: ME00009** **(207) 873-7711**

E87820
Northeast Laboratory Services, Inc.
227 China Road
Winslow, ME 04901

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Nitrobenzene	EPA 8260	Volatile Organics	NELAP	7/28/2004
Nitrobenzene	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosodimethylamine	EPA 625	Extractable Organics	NELAP	4/24/2002
n-Nitrosodimethylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosodi-n-propylamine	EPA 625	Extractable Organics	NELAP	4/24/2002
n-Nitrosodi-n-propylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Nitrosodiphenylamine	EPA 625	Extractable Organics	NELAP	4/24/2002
n-Nitrosodiphenylamine	EPA 8270	Extractable Organics	NELAP	7/1/2003
n-Propylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Oil & Grease	EPA 1664	General Chemistry	NELAP	4/24/2002
Organic nitrogen	TKN minus AMMONIA	General Chemistry	NELAP	4/24/2002
Orthophosphate as P	EPA 365.2	General Chemistry	NELAP	7/28/2004
Orthophosphate as P	EPA 9056	General Chemistry	NELAP	12/15/2006
Orthophosphate as P	SM 4500-P E	General Chemistry	NELAP	4/24/2002
o-Xylene	EPA 8260	Volatile Organics	NELAP	12/15/2006
Pentachloroethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Pentachlorophenol	EPA 625	Extractable Organics	NELAP	4/24/2002
Pentachlorophenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
pH	EPA 150.1	General Chemistry	NELAP	4/24/2002
pH	EPA 9040	General Chemistry	NELAP	7/1/2003
pH	SM 4500-H+-B	General Chemistry	NELAP	5/2/2007
Phenanthrene	EPA 625	Extractable Organics	NELAP	4/24/2002
Phenanthrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Phenol	EPA 625	Extractable Organics	NELAP	4/24/2002
Phenol	EPA 8270	Extractable Organics	NELAP	7/1/2003
Phosphorus, total	EPA 200.7	Metals	NELAP	7/28/2004
Phosphorus, total	EPA 365.2	General Chemistry	NELAP	7/28/2004
Phosphorus, total	EPA 6010	Metals	NELAP	7/28/2004
Phosphorus, total	SM 4500-P E	General Chemistry	NELAP	4/24/2002
p-Isopropyltoluene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Potassium	EPA 200.7	Metals	NELAP	4/24/2002
Propionitrile (Ethyl cyanide)	EPA 8260	Volatile Organics	NELAP	7/1/2003
Pyrene	EPA 625	Extractable Organics	NELAP	4/24/2002
Pyrene	EPA 8270	Extractable Organics	NELAP	7/1/2003
Pyridine	EPA 625	Extractable Organics	NELAP	12/15/2006
Pyridine	EPA 8270	Extractable Organics	NELAP	7/1/2003

Laboratory Scope of Accreditation

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State Laboratory ID: E87820 **EPA Lab Code: ME00009** **(207) 873-7711**

E87820
Northeast Laboratory Services, Inc.
227 China Road
Winslow, ME 04901

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
Residue-filterable (TDS)	EPA 160.1	General Chemistry	NELAP	4/24/2002
Residue-filterable (TDS)	SM 2540 C	General Chemistry	NELAP	12/15/2006
Residue-nonfilterable (TSS)	EPA 160.2	General Chemistry	NELAP	4/24/2002
Residue-nonfilterable (TSS)	SM 2540 D	General Chemistry	NELAP	12/7/2006
Residue-settleable	EPA 160.5	General Chemistry	NELAP	4/24/2002
Residue-settleable	SM 2540 F	General Chemistry	NELAP	12/15/2006
Residue-total	EPA 160.3	General Chemistry	NELAP	6/14/2002
Residue-total	SM 2540 B	General Chemistry	NELAP	12/15/2006
Residue-volatile	EPA 160.4	General Chemistry	NELAP	4/24/2002
Residue-volatile	SM 2540 E	General Chemistry	NELAP	12/15/2006
Salmonella	SM 9260 D	Microbiology	NELAP	4/24/2002
sec-Butylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Selenium	EPA 200.7	Metals	NELAP	4/24/2002
Selenium	EPA 6010	Metals	NELAP	4/24/2002
Selenium	EPA 7740	Metals	NELAP	7/28/2004
Selenium	SM 3113 B	Metals	NELAP	7/28/2004
Silica as SiO2	EPA 200.7	Metals	NELAP	7/28/2004
Silicon	EPA 6010	Metals	NELAP	7/28/2004
Silver	EPA 200.7	Metals	NELAP	8/31/2006
Silver	EPA 6010	Metals	NELAP	7/28/2004
Silvex (2,4,5-TP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	2/14/2005
Sodium	EPA 200.7	Metals	NELAP	4/24/2002
Sodium	EPA 6010	Metals	NELAP	7/1/2003
Strontium	EPA 200.7	Metals	NELAP	7/28/2004
Styrene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Styrene	NEL SOP 3.3.32, Rev. C/GC-MS	Volatile Organics	NELAP	12/15/2006
Sulfate	EPA 300.0	General Chemistry	NELAP	4/24/2002
Sulfate	EPA 375.4	General Chemistry	NELAP	12/15/2006
Sulfate	EPA 9056	General Chemistry	NELAP	7/1/2003
Sulfide	EPA 376.1	General Chemistry	NELAP	4/24/2002
Sulfide	SM 4500-S F (20th Ed.)	General Chemistry	NELAP	5/2/2007
T-amylmethylether (TAME)	NEL SOP 3.3.13, Rev. I/GC-MS	Volatile Organics	NELAP	12/15/2006
Tannin & Lignin	SM 5550 B	General Chemistry	NELAP	4/24/2002
tert-Butyl alcohol	EPA 8260	Volatile Organics	NELAP	7/28/2004
tert-Butyl alcohol	NEL SOP 3.3.32, Rev. C/GC-MS	Volatile Organics	NELAP	12/15/2006

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2007** **Expiration Date: 6/30/2008**

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Attachment to Certificate #: E87820-18, expiration date June 30, 2008. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87820 **EPA Lab Code: ME00009** **(207) 873-7711**

E87820
Northeast Laboratory Services, Inc.
227 China Road
Winslow, ME 04901

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
tert-Butylbenzene	EPA 8260	Volatile Organics	NELAP	7/1/2003
tert-Butylbenzene	NEL SOP 3.3.32, Rev. C/GC-MS	Volatile Organics	NELAP	12/15/2006
Tetrachloroethylene (Perchloroethylene)	EPA 624	Volatile Organics	NELAP	4/24/2002
Tetrachloroethylene (Perchloroethylene)	EPA 8260	Volatile Organics	NELAP	7/28/2004
Tetrahydrofuran (THF)	NE SOP 3.3.32 Rev. A	Volatile Organics	NELAP	7/26/2002
Tetrahydrofuran (THF)	NEL SOP 3.3.13, Rev. I/GC-MS	Volatile Organics	NELAP	12/15/2006
Tetrahydrofuran (THF)	NEL SOP 3.3.32, Rev. C/GC-MS	Volatile Organics	NELAP	12/15/2006
Thallium	EPA 200.7	Metals	NELAP	4/24/2002
Thallium	EPA 6010	Metals	NELAP	7/1/2003
Thallium	EPA 7841	Metals	NELAP	12/15/2006
Titanium	EPA 200.7	Metals	NELAP	7/28/2004
Titanium	EPA 6010	Metals	NELAP	5/23/2006
Toluene	EPA 624	Volatile Organics	NELAP	4/24/2002
Toluene	EPA 8260	Volatile Organics	NELAP	7/28/2004
Total coliforms	SM 9221 B	Microbiology	NELAP	4/26/2006
Total coliforms	SM 9222 B	Microbiology	NELAP	4/26/2006
Total cyanide	EPA 335.2	General Chemistry	NELAP	4/24/2002
Total cyanide	EPA 9010/9014	General Chemistry	NELAP	7/28/2004
Total nitrate-nitrite	EPA 300.0	General Chemistry	NELAP	4/24/2002
Total nitrate-nitrite	EPA 353.2	General Chemistry	NELAP	4/24/2002
Total nitrate-nitrite	EPA 9056	General Chemistry	NELAP	7/1/2003
Total organic carbon	EPA 415.1	General Chemistry	NELAP	4/24/2002
Total organic carbon	EPA 9060	General Chemistry	NELAP	7/1/2003
Total organic carbon	SM 5310C	General Chemistry	NELAP	7/28/2004
Total organic halides (TOX)	EPA 9020	General Chemistry	NELAP	7/1/2003
Total Petroleum Hydrocarbons (TPH)	FL-PRO	Extractable Organics	NELAP	7/28/2004
Total phenolics	EPA 420.1	General Chemistry	NELAP	12/15/2006
Total phenolics	EPA 9065	General Chemistry	NELAP	12/15/2006
Total residual chlorine	SM 4500-Cl D	General Chemistry	NELAP	4/24/2002
Total, fixed, and volatile residue	SM 2540 G	General Chemistry	NELAP	4/24/2002
Toxaphene (Chlorinated camphene)	EPA 608	Pesticides-Herbicides-PCB's	NELAP	4/24/2002
Toxaphene (Chlorinated camphene)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/1/2003
trans-1,2-Dichloroethylene	EPA 624	Volatile Organics	NELAP	4/24/2002
trans-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	7/1/2003
trans-1,3-Dichloropropylene	EPA 624	Volatile Organics	NELAP	4/24/2002

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Attachment to Certificate #: E87820-18, expiration date June 30, 2008. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87820 **EPA Lab Code: ME00009** **(207) 873-7711**

E87820
Northeast Laboratory Services, Inc.
227 China Road
Winslow, ME 04901

Matrix: Non-Potable Water

Analyte	Method/Tech	Category	Certification Type	Effective Date
trans-1,3-Dichloropropylene	EPA 8260	Volatile Organics	NELAP	7/1/2003
trans-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	7/1/2003
Trichloroethene (Trichloroethylene)	EPA 624	Volatile Organics	NELAP	4/24/2002
Trichloroethene (Trichloroethylene)	EPA 8260	Volatile Organics	NELAP	7/28/2004
Trichlorofluoromethane	EPA 624	Volatile Organics	NELAP	4/24/2002
Trichlorofluoromethane	EPA 8260	Volatile Organics	NELAP	7/1/2003
Turbidity	EPA 180.1	General Chemistry	NELAP	4/24/2002
Vanadium	EPA 200.7	Metals	NELAP	4/24/2002
Vanadium	EPA 6010	Metals	NELAP	7/1/2003
Vinyl acetate	EPA 8260	Volatile Organics	NELAP	7/1/2003
Vinyl chloride	EPA 624	Volatile Organics	NELAP	4/24/2002
Vinyl chloride	EPA 8260	Volatile Organics	NELAP	7/1/2003
Xylene (total)	EPA 624	Volatile Organics	NELAP	4/24/2002
Xylene (total)	EPA 8260	Volatile Organics	NELAP	7/28/2004
Zinc	EPA 200.7	Metals	NELAP	4/24/2002
Zinc	EPA 6010	Metals	NELAP	4/24/2002

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Laboratory Scope of Accreditation

Attachment to Certificate #: E87820-18, expiration date June 30, 2008. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87820 **EPA Lab Code: ME00009** **(207) 873-7711**

E87820
Northeast Laboratory Services, Inc.
227 China Road
Winslow, ME 04901

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
1,1,1,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	7/28/2004
1,1,1-Trichloroethane	EPA 8260	Volatile Organics	NELAP	7/28/2004
1,1,2,2-Tetrachloroethane	EPA 8260	Volatile Organics	NELAP	7/28/2004
1,1,2-Trichloroethane	EPA 8260	Volatile Organics	NELAP	4/24/2002
1,1-Dichloroethane	EPA 8260	Volatile Organics	NELAP	7/28/2004
1,1-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	4/24/2002
1,1-Dichloropropene	EPA 8260	Volatile Organics	NELAP	4/24/2002
1,2,3-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	4/24/2002
1,2,3-Trichloropropane	EPA 8260	Volatile Organics	NELAP	4/24/2002
1,2,4-Trichlorobenzene	EPA 8260	Volatile Organics	NELAP	4/24/2002
1,2,4-Trichlorobenzene	EPA 8270	Extractable Organics	NELAP	4/24/2002
1,2,4-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	4/24/2002
1,2-Dibromo-3-chloropropane (DBCP)	EPA 8260	Volatile Organics	NELAP	4/24/2002
1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA 8260	Volatile Organics	NELAP	4/24/2002
1,2-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/28/2004
1,2-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/28/2004
1,2-Dichloroethane	EPA 8260	Volatile Organics	NELAP	7/28/2004
1,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	4/24/2002
1,2-Diphenylhydrazine	EPA 8270	Extractable Organics	NELAP	4/24/2002
1,3,5-Trimethylbenzene	EPA 8260	Volatile Organics	NELAP	4/24/2002
1,3-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/28/2004
1,3-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/28/2004
1,3-Dichloropropane	EPA 8260	Volatile Organics	NELAP	4/24/2002
1,4-Dichlorobenzene	EPA 8260	Volatile Organics	NELAP	7/28/2004
1,4-Dichlorobenzene	EPA 8270	Extractable Organics	NELAP	7/28/2004
1-Chlorobutane	EPA 8260	Volatile Organics	NELAP	4/24/2002
2,2-Dichloropropane	EPA 8260	Volatile Organics	NELAP	4/24/2002
2,4,5-T	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	4/24/2002
2,4,5-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	4/24/2002
2,4,6-Trichlorophenol	EPA 8270	Extractable Organics	NELAP	4/24/2002
2,4-D	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	4/24/2002
2,4-DB	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	4/24/2002
2,4-Dichlorophenol	EPA 8270	Extractable Organics	NELAP	4/24/2002
2,4-Dimethylphenol	EPA 8270	Extractable Organics	NELAP	4/24/2002
2,4-Dinitrophenol	EPA 8270	Extractable Organics	NELAP	4/24/2002
2,4-Dinitrotoluene (2,4-DNT)	EPA 8270	Extractable Organics	NELAP	7/5/2002

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State Laboratory ID: E87820 **EPA Lab Code: ME00009** **(207) 873-7711**

E87820
Northeast Laboratory Services, Inc.
227 China Road
Winslow, ME 04901

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Aluminum	EPA 6010	Metals	NELAP	4/24/2002
Ammonia as N	EPA 350.2	General Chemistry	NELAP	12/15/2006
Aniline	EPA 8270	Extractable Organics	NELAP	4/24/2002
Anthracene	EPA 8270	Extractable Organics	NELAP	4/24/2002
Antimony	EPA 6010	Metals	NELAP	4/24/2002
Antimony	EPA 7041	Metals	NELAP	7/28/2004
Aroclor-1016 (PCB-1016)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	4/24/2002
Aroclor-1221 (PCB-1221)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	4/24/2002
Aroclor-1232 (PCB-1232)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	4/24/2002
Aroclor-1242 (PCB-1242)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	4/24/2002
Aroclor-1248 (PCB-1248)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	4/24/2002
Aroclor-1254 (PCB-1254)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	7/5/2002
Aroclor-1260 (PCB-1260)	EPA 8082	Pesticides-Herbicides-PCB's	NELAP	4/24/2002
Arsenic	EPA 6010	Metals	NELAP	4/24/2002
Arsenic	EPA 7060	Metals	NELAP	7/28/2004
Barium	EPA 6010	Metals	NELAP	4/24/2002
Benzene	EPA 8260	Volatile Organics	NELAP	7/28/2004
Benzidine	EPA 8270	Extractable Organics	NELAP	4/24/2002
Benzo(a)anthracene	EPA 8270	Extractable Organics	NELAP	4/24/2002
Benzo(a)pyrene	EPA 8270	Extractable Organics	NELAP	4/24/2002
Benzo(b)fluoranthene	EPA 8270	Extractable Organics	NELAP	7/5/2002
Benzo(g,h,i)perylene	EPA 8270	Extractable Organics	NELAP	4/24/2002
Benzo(k)fluoranthene	EPA 8270	Extractable Organics	NELAP	7/5/2002
Benzoic acid	EPA 8270	Extractable Organics	NELAP	4/24/2002
Benzyl alcohol	EPA 8270	Extractable Organics	NELAP	4/24/2002
Beryllium	EPA 6010	Metals	NELAP	4/24/2002
beta-BHC (beta-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/24/2002
bis(2-Chloroethoxy)methane	EPA 8270	Extractable Organics	NELAP	4/24/2002
bis(2-Chloroethyl) ether	EPA 8270	Extractable Organics	NELAP	4/24/2002
bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))	EPA 8270	Extractable Organics	NELAP	4/24/2002
bis(2-Ethylhexyl) phthalate (DEHP)	EPA 8270	Extractable Organics	NELAP	4/24/2002
Bromobenzene	EPA 8260	Volatile Organics	NELAP	4/24/2002
Bromochloromethane	EPA 8260	Volatile Organics	NELAP	4/24/2002
Bromodichloromethane	EPA 8260	Volatile Organics	NELAP	7/28/2004
Bromoform	EPA 8260	Volatile Organics	NELAP	7/28/2004
Butyl benzyl phthalate	EPA 8270	Extractable Organics	NELAP	4/24/2002

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State Laboratory ID: E87820 **EPA Lab Code: ME00009** **(207) 873-7711**

E87820
Northeast Laboratory Services, Inc.
227 China Road
Winslow, ME 04901

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
2,6-Dinitrotoluene (2,6-DNT)	EPA 8270	Extractable Organics	NELAP	4/24/2002
2-Butanone (Methyl ethyl ketone, MEK)	EPA 8260	Volatile Organics	NELAP	4/24/2002
2-Chloroethyl vinyl ether	EPA 8260	Volatile Organics	NELAP	4/24/2002
2-Chloronaphthalene	EPA 8270	Extractable Organics	NELAP	4/24/2002
2-Chlorophenol	EPA 8270	Extractable Organics	NELAP	4/24/2002
2-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	4/24/2002
2-Hexanone	EPA 8260	Volatile Organics	NELAP	4/24/2002
2-Methyl-4,6-dinitrophenol	EPA 8270	Extractable Organics	NELAP	4/24/2002
2-Methylnaphthalene	EPA 8270	Extractable Organics	NELAP	4/24/2002
2-Methylphenol (o-Cresol)	EPA 8270	Extractable Organics	NELAP	4/24/2002
2-Nitroaniline	EPA 8270	Extractable Organics	NELAP	4/24/2002
2-Nitrophenol	EPA 8270	Extractable Organics	NELAP	7/5/2002
2-Nitropropane	EPA 8260	Volatile Organics	NELAP	4/24/2002
3,3'-Dichlorobenzidine	EPA 8270	Extractable Organics	NELAP	4/24/2002
3/4-Methylphenols (m/p-Cresols)	EPA 8270	Extractable Organics	NELAP	12/15/2006
3-Nitroaniline	EPA 8270	Extractable Organics	NELAP	4/24/2002
4,4'-DDD	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/24/2002
4,4'-DDE	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/24/2002
4,4'-DDT	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	12/29/2004
4-Bromophenyl phenyl ether	EPA 8270	Extractable Organics	NELAP	4/24/2002
4-Chloro-3-methylphenol	EPA 8270	Extractable Organics	NELAP	4/24/2002
4-Chloroaniline	EPA 8270	Extractable Organics	NELAP	4/24/2002
4-Chlorophenyl phenylether	EPA 8270	Extractable Organics	NELAP	4/24/2002
4-Chlorotoluene	EPA 8260	Volatile Organics	NELAP	4/24/2002
4-Methyl-2-pentanone (MIBK)	EPA 8260	Volatile Organics	NELAP	7/28/2004
4-Nitroaniline	EPA 8270	Extractable Organics	NELAP	4/24/2002
4-Nitrophenol	EPA 8270	Extractable Organics	NELAP	7/5/2002
Acenaphthene	EPA 8270	Extractable Organics	NELAP	7/5/2002
Acenaphthylene	EPA 8270	Extractable Organics	NELAP	4/24/2002
Acetone	EPA 8260	Volatile Organics	NELAP	4/24/2002
Acrolein (Propenal)	EPA 8260	Volatile Organics	NELAP	4/24/2002
Acrylonitrile	EPA 8260	Volatile Organics	NELAP	4/24/2002
Aldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/24/2002
Allyl chloride (3-Chloropropene)	EPA 8260	Volatile Organics	NELAP	4/24/2002
alpha-BHC (alpha-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/24/2002
alpha-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/24/2002

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State Laboratory ID: E87820 **EPA Lab Code: ME00009** **(207) 873-7711**

E87820
Northeast Laboratory Services, Inc.
227 China Road
Winslow, ME 04901

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Cadmium	EPA 6010	Metals	NELAP	4/24/2002
Calcium	EPA 6010	Metals	NELAP	4/24/2002
Carbazole	EPA 8270	Extractable Organics	NELAP	4/24/2002
Carbon disulfide	EPA 8260	Volatile Organics	NELAP	4/24/2002
Carbon tetrachloride	EPA 8260	Volatile Organics	NELAP	7/28/2004
Chlordane (tech.)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/5/2002
Chloride	EPA 9056	General Chemistry	NELAP	4/24/2002
Chlorobenzene	EPA 8260	Volatile Organics	NELAP	7/28/2004
Chloroethane	EPA 8260	Volatile Organics	NELAP	7/28/2004
Chloroform	EPA 8260	Volatile Organics	NELAP	7/28/2004
Chromium	EPA 6010	Metals	NELAP	4/24/2002
Chromium VI	EPA 7196	General Chemistry	NELAP	7/28/2004
Chrysene	EPA 8270	Extractable Organics	NELAP	4/24/2002
cis-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	4/24/2002
cis-1,3-Dichloropropene	EPA 8260	Volatile Organics	NELAP	4/24/2002
Cobalt	EPA 6010	Metals	NELAP	4/24/2002
Copper	EPA 6010	Metals	NELAP	4/24/2002
delta-BHC	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/24/2002
Dibenz(a,h) anthracene	EPA 8270	Extractable Organics	NELAP	4/24/2002
Dibenzofuran	EPA 8270	Extractable Organics	NELAP	4/24/2002
Dibromochloromethane	EPA 8260	Volatile Organics	NELAP	7/28/2004
Dibromomethane	EPA 8260	Volatile Organics	NELAP	4/24/2002
Dichlorodifluoromethane	EPA 8260	Volatile Organics	NELAP	4/24/2002
Dichloroprop (Dichlorprop)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	4/24/2002
Dieldrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/24/2002
Diesel range organics (DRO)	CT-ETPH	Extractable Organics	NELAP	7/28/2004
Diesel range organics (DRO)	EPA 8015	Extractable Organics	NELAP	7/28/2004
Diesel range organics (DRO)	MEDEP 4.1.25	Extractable Organics	NELAP	4/24/2002
Diethyl ether	EPA 8260	Volatile Organics	NELAP	4/24/2002
Diethyl phthalate	EPA 8270	Extractable Organics	NELAP	4/24/2002
Di-isopropylether (DIPE)	NEL SOP 3.3.13, Rev. I/GC-MS	Volatile Organics	NELAP	12/15/2006
Dimethyl phthalate	EPA 8270	Extractable Organics	NELAP	4/24/2002
Di-n-butyl phthalate	EPA 8270	Extractable Organics	NELAP	4/24/2002
Di-n-octyl phthalate	EPA 8270	Extractable Organics	NELAP	4/24/2002
Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	4/24/2002
Endosulfan I	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/24/2002

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State Laboratory ID: E87820 **EPA Lab Code: ME00009** **(207) 873-7711**

E87820
Northeast Laboratory Services, Inc.
227 China Road
Winslow, ME 04901

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Endosulfan II	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	12/29/2004
Endosulfan sulfate	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/5/2002
Endrin	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/24/2002
Endrin aldehyde	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/5/2002
Endrin ketone	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/24/2002
Ethyl methacrylate	EPA 8260	Volatile Organics	NELAP	4/24/2002
Ethylbenzene	EPA 8260	Volatile Organics	NELAP	7/28/2004
Ethyl-t-butylether (ETBE)	NEL SOP 3.3.13, Rev. I/GC-MS	Volatile Organics	NELAP	12/15/2006
Fecal coliforms	SM 9221 E	Microbiology	NELAP	12/15/2006
Fluoranthene	EPA 8270	Extractable Organics	NELAP	4/24/2002
Fluorene	EPA 8270	Extractable Organics	NELAP	4/24/2002
gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	12/29/2004
gamma-Chlordane	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/24/2002
Gasoline range organics (GRO)	EPA 8015	Extractable Organics	NELAP	7/28/2004
Gasoline range organics (GRO)	MEDEP 4.2.17	Extractable Organics	NELAP	4/24/2002
Heptachlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/24/2002
Heptachlor epoxide	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/24/2002
Hexachlorobenzene	EPA 8270	Extractable Organics,Pesticides-Herbicides-PCB's	NELAP	4/24/2002
Hexachlorobutadiene	EPA 8260	Volatile Organics	NELAP	4/24/2002
Hexachlorobutadiene	EPA 8270	Extractable Organics	NELAP	4/24/2002
Hexachlorocyclopentadiene	EPA 8270	Extractable Organics,Pesticides-Herbicides-PCB's	NELAP	4/24/2002
Hexachloroethane	EPA 8260	Volatile Organics	NELAP	4/24/2002
Hexachloroethane	EPA 8270	Extractable Organics	NELAP	4/24/2002
Ignitability	EPA 1010	General Chemistry	NELAP	4/24/2002
Indeno(1,2,3-cd)pyrene	EPA 8270	Extractable Organics	NELAP	4/24/2002
Iodomethane (Methyl iodide)	EPA 8260	Volatile Organics	NELAP	4/24/2002
Iron	EPA 6010	Metals	NELAP	4/24/2002
Isophorone	EPA 8270	Extractable Organics	NELAP	4/24/2002
Isopropylbenzene	EPA 8260	Volatile Organics	NELAP	4/24/2002
Kjeldahl nitrogen - total	EPA 351.3	General Chemistry	NELAP	12/15/2006
Lead	EPA 6010	Metals	NELAP	4/24/2002
Lead	EPA 7421	Metals	NELAP	7/28/2004
Lithium	EPA 6010	Metals	NELAP	4/24/2002

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E87820
Northeast Laboratory Services, Inc.
227 China Road
Winslow, ME 04901

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
m/p-Xylenes	EPA 8260	Volatile Organics	NELAP	12/15/2006
Magnesium	EPA 6010	Metals	NELAP	4/24/2002
Manganese	EPA 6010	Metals	NELAP	4/24/2002
Mercury	EPA 7471	Metals	NELAP	4/24/2002
Methacrylonitrile	EPA 8260	Volatile Organics	NELAP	4/24/2002
Methoxychlor	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	4/24/2002
Methyl bromide (Bromomethane)	EPA 8260	Volatile Organics	NELAP	4/24/2002
Methyl chloride (Chloromethane)	EPA 8260	Volatile Organics	NELAP	4/24/2002
Methyl methacrylate	EPA 8260	Volatile Organics	NELAP	4/24/2002
Methyl tert-butyl ether (MTBE)	EPA 8260	Volatile Organics	NELAP	4/24/2002
Methylene chloride	EPA 8260	Volatile Organics	NELAP	7/28/2004
Molybdenum	EPA 6010	Metals	NELAP	4/24/2002
Naphthalene	EPA 8260	Volatile Organics	NELAP	4/24/2002
Naphthalene	EPA 8270	Extractable Organics	NELAP	4/24/2002
n-Butylbenzene	EPA 8260	Volatile Organics	NELAP	4/24/2002
Nickel	EPA 6010	Metals	NELAP	4/24/2002
Nitrate	EPA 9056	General Chemistry	NELAP	4/24/2002
Nitrite	EPA 9056	General Chemistry	NELAP	4/24/2002
Nitrobenzene	EPA 8260	Volatile Organics	NELAP	7/28/2004
Nitrobenzene	EPA 8270	Extractable Organics	NELAP	4/24/2002
n-Nitrosodimethylamine	EPA 8270	Extractable Organics	NELAP	4/24/2002
n-Nitrosodi-n-propylamine	EPA 8270	Extractable Organics	NELAP	4/24/2002
n-Nitrosodiphenylamine	EPA 8270	Extractable Organics	NELAP	4/24/2002
n-Propylbenzene	EPA 8260	Volatile Organics	NELAP	4/24/2002
Organic nitrogen	TKN minus AMMONIA	General Chemistry	NELAP	12/15/2006
o-Xylene	EPA 8260	Volatile Organics	NELAP	12/15/2006
Pentachlorobenzene	EPA 8270	Extractable Organics	NELAP	7/28/2004
Pentachloroethane	EPA 8260	Volatile Organics	NELAP	4/24/2002
Pentachlorophenol	EPA 8270	Extractable Organics	NELAP	7/5/2002
pH	EPA 9040	General Chemistry	NELAP	6/24/2002
pH	EPA 9045	General Chemistry	NELAP	4/24/2002
Phenanthrene	EPA 8270	Extractable Organics	NELAP	4/24/2002
Phenol	EPA 8270	Extractable Organics	NELAP	4/24/2002
Phosphorus, total	EPA 6010	Metals	NELAP	7/28/2004
p-Isopropyltoluene	EPA 8260	Volatile Organics	NELAP	4/24/2002
Potassium	EPA 6010	Metals	NELAP	4/24/2002

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State Laboratory ID: E87820 **EPA Lab Code: ME00009** **(207) 873-7711**

E87820
Northeast Laboratory Services, Inc.
227 China Road
Winslow, ME 04901

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Propionitrile (Ethyl cyanide)	EPA 8260	Volatile Organics	NELAP	4/24/2002
Pyrene	EPA 8270	Extractable Organics	NELAP	7/5/2002
Pyridine	EPA 8270	Extractable Organics	NELAP	4/24/2002
Residue-total	SM 2540 G	General Chemistry	NELAP	12/15/2006
Salmonella	SM 9260 D	Microbiology	NELAP	12/15/2006
sec-Butylbenzene	EPA 8260	Volatile Organics	NELAP	4/24/2002
Selenium	EPA 6010	Metals	NELAP	4/24/2002
Selenium	EPA 7740	Metals	NELAP	7/28/2004
Silica as SiO2	EPA 6010	Metals	NELAP	7/28/2004
Silver	EPA 6010	Metals	NELAP	7/28/2004
Silvex (2,4,5-TP)	EPA 8151	Pesticides-Herbicides-PCB's	NELAP	4/24/2002
Strontium	EPA 6010	Metals	NELAP	4/24/2002
Styrene	EPA 8260	Volatile Organics	NELAP	4/24/2002
Sulfate	EPA 9056	General Chemistry	NELAP	4/24/2002
T-amylmethylether (TAME)	NEL SOP 3.3.13, Rev. I/GC-MS	Volatile Organics	NELAP	12/15/2006
tert-Butyl alcohol	EPA 8260	Volatile Organics	NELAP	7/28/2004
tert-Butylbenzene	EPA 8260	Volatile Organics	NELAP	4/24/2002
Tetrachloroethylene (Perchloroethylene)	EPA 8260	Volatile Organics	NELAP	7/28/2004
Tetrahydrofuran (THF)	NEL SOP 3.3.13, Rev. I/GC-MS	Volatile Organics	NELAP	12/15/2006
Thallium	EPA 6010	Metals	NELAP	4/24/2002
Thallium	EPA 7841	Metals	NELAP	7/28/2004
Titanium	EPA 6010	Metals	NELAP	6/24/2002
Toluene	EPA 8260	Volatile Organics	NELAP	7/28/2004
Total cyanide	EPA 9010/9014	General Chemistry	NELAP	4/24/2002
Total Petroleum Hydrocarbons (TPH)	FL-PRO	Extractable Organics	NELAP	7/28/2004
Toxaphene (Chlorinated camphene)	EPA 8081	Pesticides-Herbicides-PCB's	NELAP	7/5/2002
Toxicity Characteristic Leaching Procedure	EPA 1311	General Chemistry	NELAP	4/24/2002
trans-1,2-Dichloroethylene	EPA 8260	Volatile Organics	NELAP	4/24/2002
trans-1,3-Dichloropropylene	EPA 8260	Volatile Organics	NELAP	4/24/2002
trans-1,4-Dichloro-2-butene	EPA 8260	Volatile Organics	NELAP	4/24/2002
Trichloroethene (Trichloroethylene)	EPA 8260	Volatile Organics	NELAP	7/28/2004
Trichlorofluoromethane	EPA 8260	Volatile Organics	NELAP	4/24/2002
Vanadium	EPA 6010	Metals	NELAP	4/24/2002
Vinyl acetate	EPA 8260	Volatile Organics	NELAP	4/24/2002
Vinyl chloride	EPA 8260	Volatile Organics	NELAP	4/24/2002

Clients and Customers are urged to verify the laboratory's current certification status with the Environmental Laboratory Certification Program. **Issue Date: 7/1/2007** **Expiration Date: 6/30/2008**

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Attachment to Certificate #: E87820-18, expiration date June 30, 2008. This listing of accredited analytes should be used only when associated with a valid certificate.

State Laboratory ID: E87820 **EPA Lab Code: ME00009** **(207) 873-7711**

E87820
Northeast Laboratory Services, Inc.
227 China Road
Winslow, ME 04901

Matrix: Solid and Chemical Materials

Analyte	Method/Tech	Category	Certification Type	Effective Date
Xylene (total)	EPA 8260	Volatile Organics	NELAP	7/28/2004
Zinc	EPA 6010	Metals	NELAP	4/24/2002

CHAPTER 7
ANALYSIS OF QUALITY CONTROL SAMPLES

7.1.0 Introduction

This LQAP discusses samples that are routinely analyzed with the normal laboratory samples to demonstrate that the laboratory is operating within data quality objective requirements for accuracy and precision. Quality control samples are of a known content and concentration, with the exception of field blanks and microbiology samples, so that accuracy and precision can be determined.

7.2.0 Types of Quality Control Samples

7.2.1 Trip Blank and Field Blank Samples

Trip blanks are reagent water-filled sample containers that are prepared at the lab and shipped with the sample containers to monitor any contamination that may be picked up during sample shipment or storage. Field blanks are reagent water samples that are prepared at the sampling site to monitor contamination from sampling equipment, and/or site specific contamination. Field blanks with regard to microbial analyses of air samples, may include specimens of blank sampling media from the same source lot as was used for collecting the field samples. The field blank shall be assigned a unique identifier as with all samples and results and will be reported to the client as part of their data package. Test reports will contain notes as to whether blanks were analyzed and that if they were analyzed they were treated like a normal sample. NEL does not correct for blank contamination unless it is specifically written in the method to do so.

7.2.2 Reagent Blank Analyses

A reagent blank is a volume of analyte free, distilled laboratory water for water samples, or a purified solid matrix for soil/sediment samples carried through the entire analytical procedure. The volume or weight of the blank must be approximately equal to the sample volume or sample weight processed. A reagent blank verifies that method interference caused by contaminants in solvents, reagents, glassware, and other sample processing hardware are known and minimized. Optimally, a reagent blank should contain analyte concentrations no greater than the method detection limit (MDL) for the parameter. For Navy samples, if the method blank contamination exceeds one-half the reporting limit, the laboratory shall evaluate whether reprocessing of the samples is necessary based on the above criteria. The concentration of common laboratory contaminants shall not exceed the reporting limit. Any sample associated with a blank that fails these criteria checks shall be reprocessed in a subsequent preparation batch except when the sample analyses resulted in a non-detect. If no sample volume remains for reprocessing, the results shall be reported with appropriate data qualifying codes that are required for each analytical batch or at a minimum of 5%, whichever is the more frequent. Results of reagent blank analyses should be maintained with the corresponding analytical data in the project file and may be used to flag data on the final report.

7.2.3 Duplicate Sample Analysis

Duplicate analyses are performed to evaluate the precision of an analysis. Results of the duplicate analyses are used to determine the relative percent difference between replicate

samples. For chemical analyses, a duplicate analysis will be performed whenever a batch of samples is analyzed, or at a minimum of 5%, whichever is the more frequent. Frequency shall be determined by the methodology. Duplicate analysis results should be summarized on the data form. For microbiological analyses, duplicates will be run on 5% of suspected positive samples.

7.2.4 Check Standard Analyses - Chemical Analyses Only

Standards and calibration curves are subject to change and can vary from one day to the next. Either a calibration curve or a passing continuing calibration standard will be run with each batch of samples.

7.2.5 Surrogate Standard Analyses - Chemical Analyses Only

Surrogate standard determinations should be performed on all appropriate samples and blanks for organics. All samples and blanks are fortified with surrogate spiking compounds before purging or extraction to monitor preparation and analysis of the samples. Recoveries should meet acceptance criteria as defined by the SOP or cited method. Surrogate failure on the first extraction will result in re-extraction of the sample. Second failure will be flagged as matrix interference. Surrogate standard data should be summarized on the surrogate form.

7.2.6 Matrix Spike Analyses - Chemical Analyses Only

To evaluate the effect of the sample matrix on the accuracy of the performance, a separate aliquot of sample should be spiked with the analyte of interest and analyzed with the sample. A matrix spike shall be run a minimum of one per batch. The percent recovery for each respective compound will then be calculated. If the percent recovery falls outside established quality control standards the QC check sample data or laboratory control sample data should be evaluated. Matrix Spike (MS) results should be summarized on the quality control data summary sheets. Matrix Spike Duplicates (MSD) should be run in lieu of 7.2.3 Duplicate Analysis when there is sufficient sample to do so. If matrix spike analysis is not possible because insufficient sample volume is received, the laboratory will perform laboratory control sample (LCS) laboratory control sample duplicate (LCSD) analyses (see Section 7.2.10).

7.2.7 QC Check Sample

A QC check sample is a laboratory fortified blank of either an aqueous or solid matrix, which is carried through the entire analytical sequence with the sample batch. This verifies the analyst(s) performance of the method. These results are to be evaluated against the method criteria.

7.2.8 External Quality Control Samples

7.2.8.1 NELAC certification analytes only:
The Laboratory shall analyze all pertinent proficiency tests samples (WP, WS and soil type). Proficiency test samples are prepared at concentrations unknown to the laboratory and are purchased through a NIST accredited NVLAP provider. In order to maintain drinking water certification (WS) as well as to demonstrate proficiency in wastewater analyses (WP), along with obtaining/maintaining NELAP accreditation for drinking water, waste water, groundwater and soil analyses, results shall be submitted to the NVLAP provider and are reported to the primary laboratory certifying/accrediting agency as well as to any other states specified by the laboratory. Performance studies are performed at least two times/year/matrix.

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7.2.8.2 A2LA Certified Analytes Only:

The laboratory shall participate in external proficiency testing schemes that are suitable for the materials examined by the laboratory. Consideration shall be given to those external schemes that are based on and/or accredited to ISO Guide 43-1. If an external scheme is not available for the material or test method, the laboratory must develop a suitable in-house scheme to demonstrate competency in the conduct of the method and ability to obtain acceptable results. Proficiency testing shall be done on a laboratory basis following the normal working practices in the laboratory. Participation is required at least twice per year for as many analytes and methods as possible, at a frequency sufficient to ensure that all testing sub-disciplines listed on a laboratories scope of accreditation is covered over a four year period.

7.2.8.2.1 Proficiency testing results that are graded as "Unsatisfactory" will be reported to A2LA upon receipt and another PT round will be ordered for the unsatisfactory analyte. Results that are within the acceptable reporting range, but cannot be judged accurately because the result was above or below the limits of our method will be marked as "Needs Review". Results in a dataset that are within the acceptable range, but the dataset has been judged as inadequate to provide an adequate comparison for the result (ie: there were fewer than 10 results, or there is too much variation in the results obtained in the dataset) are marked as "Not Determined". For PT results graded "Needs Review" and "Not Determined" on the result evaluation, a CAR will be initiated and the laboratory will compare the reported result to the consensus result to evaluate the labs performance. Grades of "Needs Review" and "Not Determined" are not required to be submitted to A2LA.

7.2.8.3 Proficiency samples are treated in the same manner as client samples. Discussion of results with other laboratories, or with the proficiency sample provider prior to official results becoming available, is strictly prohibited.

7.2.8.4 The lab will not send out any proficiency sample to another lab for any analysis for which it seeks accreditation, or is accredited. It will also not receive any proficiency or a portion of a proficiency sample from another lab for any analysis it is accredited for.

7.2.9 Laboratory Control Sample (LCS)/Blank Spike - Chemical Analyses Only

An LCS will be analyzed with each batch of samples. A batch will consist of no more than 20 samples. The LCS is defined as a blank spiked with a known concentration of target analyte(s). A LCSD may be performed in place of an MSD if sufficient sample is unavailable to perform an MSD. Each analytical method that supports the use of a LCS should have calculated control limits for recovery. Vendor derived control limits are used where applicable.

7.2.10 Negative Controls - Microbiological Analysis Only
Negative control samples demonstrate that the sample equipment and growth media have not become contaminated through handling, preparation or environmental exposure. Negative controls include sterility checks of media and blank samples such as filtration blanks, bottle and buffer blanks. All blanks and inoculated controls specified by the test method shall be prepared and analyzed at the frequency stated in the method.

A minimum of one inoculated control shall be prepared and analyzed unless the same equipment set is used to prepare multiple samples. In such cases, the analyst shall prepare

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at least one beginning and one ending control, with additional controls inserted after every ten (10) samples.

Negative culture controls are used to verify that selective properties of the culture media have not deteriorated during storage. These controls utilize either direct inoculation of the culture medium with a known negative culture or processing of a control sample with a defined amount of a known negative culture. A negative culture is a bacterial culture that either should not grow under the conditions of the test or does not produce a positive reaction on the culture media. A negative culture control is run with each batch of test samples. For analyses that are performed infrequently, a negative control will be run on a monthly basis or per lot of test medium whichever is more frequent. Any growth in a negative control must be investigated and the assay invalidated if the growth is deemed to interfere with the assay or if the growth is identified as the pathogen of concern.

7.2.11 Positive Controls - Microbiological Analyses Only

Positive control samples are used to demonstrate that the medium and culture conditions can support the growth of the target organism and the specified or expected reactions are produced. The positive control provides visual reference for comparison in biochemical reactions and also validates the media and incubators used.

A positive control sample is defined as a direct inoculation of the test medium with a known culture of a target organism or the processing of a control sample containing a defined amount of a known target organism. Such target organism is defined in the method SOP and must demonstrate adequate growth and desired/expected reactions on the test medium under the conditions of the test.

A positive control sample is run with each batch of test samples. For analyses that are performed infrequently, a positive control will be run on a monthly basis or per lot of test medium, whichever is more frequent. The failure of a positive control culture to grow through all phases of the assay constitutes an invalid test. Invalid tests must be reset (if time allows) or re-sampled.

7.2.13 For microbiology analyses target-organism to reference samples are formulated and analyzed in the laboratory on a quarterly basis. The reference samples are prepared from an 18-24 hour culture and will have a defined organism concentration, based on a standard plate count (HPC) of the formulation.

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CHAPTER 8

DATA VERIFICATION & REVIEW

8.1.0 Introduction

Data verification is a series of steps taken within the laboratory so that reported results correctly reflect the analyses performed. There are two basic activities:

- The processing of quality control sample results to demonstrate that analyses are in control with respect to accuracy, precision and completeness.
- Data validation to demonstrate that numerical computation of data is correct and that it is correctly reported.

This section discusses how data verification is performed.

Data review is a procedure to insure that results of field and quality control samples are correctly verified and reported. Review may occur as the data is being processed by the analyst or being validated by the Laboratory Manager, but will occur as required by contract/customer when reports of data have been prepared for transmittal outside the laboratory.

8.2.0 Processing of Quality Control Data

This section describes the analytical treatment of the data resulting from the quality control samples discussed in LQAP Chapter 7. This treatment of data may vary according to methodology.

8.2.1 Specific Routine Procedures Used to Assess Data Precision and Accuracy

8.2.1.1 A reagent blank, method blank or negative control is prepared and analyzed with each set of samples.

8.2.1.2 For chemistry analyses, trip/field blanks are analyzed to determine possible sample contamination during collection and shipment to the laboratory.

8.2.1.3 For instrument-based analyses, a calibration curve or a passing continuing calibration standard is run with each analytical sequence. The frequency is method dependent.

8.2.1.4 For chemical analyses, one sample is to be analyzed in duplicate for each matrix and each batch of samples, but no less than one for every twenty samples. For microbiological analyses, duplicates are run as defined in Section 7.2.3. Duplicates should be within method criteria.

8.2.1.5 For chemistry analyses, one sample in every batch of samples or for each matrix, but no less than one in every twenty samples, is to be spiked at an appropriate level. Recoveries shall meet method criteria. A matrix spike duplicate (MSD) should be used in lieu of a sample duplicate if most target analytes are anticipated to be ND or if sufficient sample is available to perform an MSD analysis. RPD should meet method criteria. If not, the QC check sample will be evaluated.

8.2.1.6 For chemical analyses, a blind replicate sample or a blind Standard Reference Material sample purchased from an NIST approved provider, **unknown** to the Analyst, is introduced at least semi-annually. Blind replicates or Standard Reference Materials, are routinely used for the analysis of metals, water quality parameters (non-microbiological), and organics analyses.

8.2.17 For microbiological analyses, a positive control is run with each batch of samples.

8.2.2 When the analyses of a sample set are completed, the results will be reviewed and evaluated to assess the validity of the data set. Review is based on the following criteria:

8.2.2.1 Reagent Blank and Negative Control Evaluation - The reagent and/or method blank results are evaluated for high readings characteristic of background contamination. If high blank values are observed, laboratory glassware and reagents should be checked for contamination. A high background is defined as a background value sufficient to result in a difference in the sample value, if not corrected, greater than or equal to the smallest significant digit known to be true. For Navy samples, if the method blank contamination exceeds one-half the reporting limit, the laboratory shall evaluate whether reprocessing of the samples is necessary based on the above criteria. The concentration of common laboratory contaminants shall not exceed the reporting limit. Any sample associated with a blank that fails these criteria checks shall be reprocessed in a subsequent preparation batch, except when the analysis resulted in a non-detect. If no sample volume remains for reprocessing, the results shall be reported with appropriate data qualifying codes. A reagent blank should contain no greater than one-half the reporting limit for any parameter. A reagent blank should contain no greater than five times (5X) the parameter detection limit for most parameters. At higher levels, common lab contaminates such as methylene chloride, phthalates, acetone, will be documented and given to the Total Quality Manager or designee for a decision as to the applicability of the data for its' intended use. Notations will be made on final reports. For microbiological samples, negative controls should show either no growth or the lack of positive reactions, depending on the type of control sample. Control samples that do not comply and the associated test samples will be evaluated by the Microbiology Manager or designee and by the QA Manager to determine the validity of the test data. If necessary all test results will be invalidated. If test results are reported, notations will be made on the final report.

8.2.2.2 Field Blank Evaluation (see 7.2.1) - Field blank results are evaluated for high readings similar to the reagent and/or method blanks described above. If high field blank readings are encountered, the procedure for sample collection, shipment, and laboratory analysis should be reviewed. If both the reagent and/or method blanks and the field blanks exhibit significant background contamination, the source of contamination is probably within the laboratory. In the case of VOA's, ambient air in the laboratory and reagents should be checked as possible sources of contamination. High field blank readings for other parameters may be due to contaminated sample bottles, cross-contamination of samples and sampling equipment.

8.2.2.3 Calibration Standard Evaluation- The daily calibration curve is evaluated to determine linearity through its full range, and that sample values are within

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the range defined by the low and high standards. If the curve is not linear, sample values must be corrected for non-linearity by deriving sample concentrations from a graph or by using an appropriate algorithm to fit a nonlinear curve to the standards. If a continuing calibration standard is used to validate the curve for the day, it must meet method criteria.

If using linear regression, the curve's correlation coefficient must be at least 0.995 or higher unless specifically addressed otherwise in the method. The number of calibration points is defined by the method. Additional points may be used but never fewer. If the analytical method does not specify a criteria for the second source or CCV standard, 15% of the true value must be used unless historical data supports a larger window.

8.2.2.4 Replicate Sample Evaluation (chemical analyses only) - Duplicate sample analysis for the sample set is used to determine the precision of the analytical method for the sample matrix. The duplicate results are used to calculate the precision as defined by the relative percent difference (RPD). The precision value, RPD, should be calculated for the parameter determined. If the precision value exceeds the warning limit for the given parameter, the Laboratory Manager is notified in writing. If the precision value exceeds the control limit, the sample set must be reanalyzed for the parameter in question. Attainable precision limits will be specified by the Laboratory Manager and updated periodically following review of data.

8.2.2.5 Matrix Spike Evaluation (chemical analyses only) - The observed recovery of the spike versus the theoretical spike recovery is used to calculate accuracy as defined by the percent recovery. The accuracy value and the percent recovery may be plotted on a control chart for the parameter determined. If the accuracy value exceeds the warning limit for the given parameter, the Laboratory Manager is notified in writing. A discrepancy report must be filled out and source of the problem must be identified. If the accuracy value exceeds the control limit, QC check sample (or LCS) will be evaluated.

8.2.2.6 Blind Replicate Evaluation (chemical analyses only) - The blind replicate analysis is evaluated in the same manner as described above for the duplicate sample analysis and is treated as a duplicate result for purposes of evaluating the precision of the analytical method. This evaluation is performed independently by the Laboratory Manager.

8.2.2.7 Reference Standard Evaluation (chemical analyses only) - Standard Reference Materials analyses are compared with true values and acceptable ranges. Values outside the acceptable ranges require corrective action to determine the source of error and provide corrective action. All sample analyses should be halted pending this evaluation. Following correction of the problem, the Standard Reference Material should be reanalyzed and meet acceptance criteria. Documentation of the problem, its' solution, samples impacted, and reanalysis of the batch will be given to the Total Quality Manager.

8.2.2.8 Standard Reference Material Evaluation (chemical analyses only) - The results of standard reference material (SRM) analysis are compared with the true values and the percent recovery of the material is calculated. If correction is required, the SRM should be reanalyzed to demonstrate that the corrective action has been successful. Many times these check standards are used as the Blind Replicate Samples due to the fact that they are introduced "blindly" to the Analysts.

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8.2.2.9 Surrogate Standard Evaluation (chemical analyses only) - The results of surrogate standard determination are compared with the true values spiked into the sample matrix prior to extraction and analysis and the percent recoveries of the surrogate standards are determined. Unless laboratory statistical limits have been determined the percent recoveries recommended as attainable by the USEPA for volatile and semi-volatile compounds are followed. When surrogate recoveries fail criteria a corrective action form/discrepancy report will be completed requiring that the sample be re-extracted and analyzed again. Samples failing the second time will be footnoted as having possible matrix interference on the final report. Should analyte concentration force the dilution of the sample greater than 10 fold the surrogate value will not be reported.

8.2.2.10 Laboratory Control Sample (LCS)/Blank Spike Evaluation (chemical analyses only) – LCS limits are normally calculated taking the recovery results from at least 30 LCS samples, calculating the standard deviation and mean of the recoveries, and then adding and subtracting three standard deviations from the mean. These limits are then compared to USEPA limits, if they are in the methods, and the more stringent of the two are used as the laboratory criteria. The LCS limits determine if the lab analysis is functioning properly. If the LCS fails, the analysis is stopped and checked for errors. Data will not be reported or if it is reported, it will be noted that the LCS does not pass acceptable criteria.

8.2.2.10a For Air Force Center for Environmental Excellence (AFCEE) samples, NEL will track all analytes spiked into the LCS using control charts. NEL will update the charts annually and review them quarterly for possible trends that may compromise data quality. Control charts will be maintained for both solid and liquid matrices. The control limits established by NEL will be equal to or within the AFCEE Quality Assurance Action Prevention Plan (QAPP) limits whenever such limits exist. The following conditions, at a minimum, will be considered out-of-control events for the control charts and data will not be reported:

- Any one point outside control limits.
- Any three consecutive points outside warning limits.
- Any six consecutive points such that each point is larger (or smaller) than its immediate predecessor.
- Any eight consecutive points on the same side of the centerline.

Reanalysis of all affected samples is necessary once the system is back in control except when an analyte in the LCS exceeds the upper control limit and the analyte is not detected in the associated samples. Out-of-control events detected in the control charts will be documented in a corrective action/discrepancy report.

8.2.2.11 Reporting Limit – The Laboratory's reporting limit shall be within the calibration range, at or above the lower quantitation limit. It is laboratory policy to establish the reporting level at the lowest initial calibration concentration, unless the annual MDL study does not justify this. The reporting level must be at least three times the statistically derived MDL. If the MDL is above this 3 x factor, either a new MDL study will be initiated or

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the reporting level will be adjusted higher. Also, please refer to SOP 3.7.13, SOP for MDL Determination.

Dilution is the rule at NEL but due to method specific requirements for metals, turbidity and anions, the following schedule for Linear Dynamic Range (LDR) performance is followed:

ICP:	Semi-annually
Furnace:	Semi-annually
IC:	Quarterly
TRAACS:	Quarterly
Turbidity:	Quarterly

LDRS are logged into Labworks at the required frequency by the Total Quality Manager and all data is stored in the Total Quality Manager's office for a period of two years and then is boxed up and stored in the attic of NEL for the remaining eight years.

8.2.2.12 Positive Control Evaluation (microbiological analyses only) - Positive controls should exhibit growth of the target organism and any positive/expected reactions on the test media. Positive control samples and their associated test samples that do not comply will be evaluated and handled as described for negative control samples in Section 8.2.2.1.

8.2.3 Statistical Evaluation of Quality Control Data

As part of the analytical quality control program, NEL will determine precision and accuracy for each parameter analyzed. Initially, when these data are compiled, the evaluation is applied over a broad concentration range. As more data are accumulated, precision and accuracy determinations will be updated and criteria developed to define precision and accuracy over all specific concentration ranges.

8.2.3.1 Evaluation of Analytical Precision

General Considerations - To determine the precision of the method and/or laboratory Analyst, a routine program of replicate analyses is performed. The results of the replicate analyses are used to calculate the relative percent difference (RPD), which is the governing quality control parameter for precision. When the RPD is obtained for at least ten replicate pairs, the average RPD and the standard deviation are calculated.

8.2.3.2 Evaluation of Analytical Accuracy

When a program for evaluation of analytical accuracy is established, the evaluation is applied over the entire range of spiking concentrations. To determine the accuracy of an analytical method and/or the Laboratory Analyst, a periodic program of sample spiking is conducted. The results of sample spiking are used to calculate the quality control parameter for accuracy evaluation or the Percent Recovery (%R).

8.3.0 Data Validation

Data validation begins with the processing of data. Data processing can be performed by the analyst who obtained the data or by peer review. Validation continues with checking that the data processing has been done correctly. This step is performed by the Laboratory Manager or can be performed as peer review by an independent analyst. All those who validate or review data processing shall indicate this by initials and date on the documents validated or reviewed.

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8.3.1 In general, data will be processed by an Analyst in one of the following ways:

- Manual computation of results directly on the data sheet or on calculation pages attached to the data sheets
- Input of raw data for computer processing
- Direct acquisition and processing of raw data by a computer

8.3.2 If data are manually processed by an Analyst, all steps in the computation shall be provided, including equations used and the source of input parameters such as response factors, dilution factors and calibration constants. The analyst shall initial and date in ink each page of calculations.

8.3.3 For data that are input by an analyst and processed using a computer, a copy of the input shall be kept and uniquely identified with the project number and other information as needed. The samples analyzed shall be evident and the input signed and dated by the analyst. All computer program calculations shall be validated manually, prior to use and then at least annually. This shall be documented and on file with the Total Quality Manager.

8.3.4 If data are directly acquired from instrumentation and processed, the analyst shall verify that the following are correct: project and sample numbers, calibration constants and response factors, that the linearity of the curve meets method criteria, output parameters such as units, and numerical values used for detection limits (if a value is reported as less than). The analyst shall sign and date in ink the resulting output.

8.4.0 Review of Data Reports

8.4.1 Review of data reports is required to verify that information reported by the Laboratory corresponds with processed analytical results. Preparation of the data reports and the Data Package Summary Sheet for Level IV packages, (attached) is the responsibility of the analyst. Review of a data package by the Laboratory Manager or designee is done prior to transmission of the report from the laboratory. Those who prepare and review data package reports shall indicate such by a signature or initials and date in ink on the report.

8.4.2 If required, EPA completed sample data file packages (CSF) will include a cover sheet detailing each document to be included. As these are completed and checked the cover sheet will be so marked.

8.4.3 Review of EPA CSF will include all steps outlined above and assembly and cross checking of:

* Sample tags, custody records, laboratory bench sheets, other sample tracking records, any chromatographic charts, and strip charts.

* Any computer print outs, raw data summaries, any correspondence, and any other written documents.

8.4.4 Original CSF paper work will be furnished as required by the contract. Compilation of this package will be the responsibility of the Laboratory Manager, or designee.

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DATA PACKAGE: SUMMARY SHEET

QC Data Package Number:_____

Analytical Method in Package	Check	Analyst	
Percent Solids Data			
Lowest Point Performance Standard			
Multi Point Curves			
Or Daily Calibration			
Continuing Calibration Curve Check(s)			
Method Blank: Quantitation Report			
Laboratory Reagent Blank Quantitation Report			
Raw Data on Blanks			
Surrogate Recovery Summary Form			
Internal Standard Area Summary			
Spike/Duplicate Recovery Data and Summary			
External QC or Standard Reference Material			
QC Sample(s) or LFB(s)			
Sample(s): Quantitation Report			
Raw Data			
Runlog			
Extraction or Prep Log			
Surrogate Solution ID		Date Made	
Spike Solution ID		Date Made	
Cal Curve Stock Solution ID		Date Made	

Data Validation	
Date:	
Validator:	
Acceptable?	

Case Narrative/Comments/Problems

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CHAPTER 9
DATA REPORTS

9.1.0 The **format and content of a data report** is dependent upon project needs, such as: whether or not explanatory text is required, client or contract requirements, and government agency reporting formats. However, the following minimum requirements are necessary:

9.1.1 The final data presentation shall be checked in accordance with data verification requirements of LQAP 8 and approved by the Laboratory Manager in the form of a certificate of analysis.

9.1.2 Quality Control Data are presented in a tabular format and corresponding samples identified whenever possible.

9.1.3 Data presentation for standard QC level 1 reports includes but is not limited to:

- The title "Certificate of Analysis"
- The name and address of the laboratory
- A unique sample identification number used by NEL and repeated on each page;
- The name and address of the client;
- Chemical parameters analyzed, reported values, and units of measurement and methods used;
- Sample identification/description provided to the laboratory by the client;
- Reporting limit of the analytical procedure ;
- Footnotes referenced to specific data if required to explain reported values;
- Date and time of analysis and analyst's initials;
- Report date;
- Sample collection date and time;
- Sample receipt date and time;
- Sampling plan and procedures, if relevant;
- Name, function and signature of persons authorizing the report;
- Where relevant, a statement that the results relate only to the items tested;
- Surrogate recoveries (organics only)
- Chain of custody form
- Page numbers and total number of pages;
- Customer Feedback cards

Where necessary for the interpretation of test results, especially with regard to environmental microbiology results, the following shall be included:

- Environmental conditions
- Opinions and Interpretations
- Additional information as required by the client
- Location of sampling
- Standard or other specification for the sampling method or procedure and deviations, additions to or exceptions from the specification concerned

Example reports can be found attached to this chapter as well as Northeast Laboratory's Quality Control Reporting Levels.

Initiated By: Total Quality Manager

Approved By: Pamela Doughty
Total Quality Manager

Approved By: A Galary
Chemistry Lab Manager

Approved By: Brian M Woodruff
Microbiology Manager

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9.1.4 **Only the Laboratory Manager or designee should transmit data from the laboratory. Due to the confidentiality of our data, it will only be transmitted to the person responsible for the payment of the analytical services or their written designee. Verbal results will not be given unless absolute certainty of the caller's identity is known. Clients should be made aware that verbal results are considered only preliminary results.**

The Microbiology Laboratory has established maximum contamination levels for Total Coliforms and *E. coli* in potable samples. When the maximum contamination levels are exceeded, the positive results are reported as "unsatisfactory" and the client is contacted by mail, phone, fax or e-mail. Retesting is scheduled for a later date.

Positive results for the analysis of fecal coliforms in non-potable waters are reported and clients are contacted by mail, phone, fax or e-mail as to sample conditions. Resampling is scheduled for a later date.

9.1.5 As necessary, the laboratory will provide additional information. This information should include the following:

- Documentation of any sample damage while in transit to the laboratory. This is to include damaged shipping containers and/or damage to the container in which the samples were shipped.
- Documenting that incorrect sample containers were used for the tests requested, and/or that incorrect volumes were provided by the client.
- Verification of Chain of Custody documentation and any other paperwork which may be pertinent to keeping the integrity of the sample intact.
- If necessary, discussion, with the client, of any apparent changes in the original data quality objectives, which were discussed before the onset of the project.
- If necessary, discussion, with the client, of any analytical difficulties while performing the analysis requested. Notations shall appear on the final report and if applicable in the case narrative, if problems were encountered and the lab was unable to complete the analysis as promised. The following codes will be used for chemical analyses only:

DATA QUALIFIERS

1A. B **(Inorganics)** The reported value was obtained from a reading that was less than the reporting limit but greater than or equal to the instrument detection limit (IDL).

1B. B **(Organics)** The compound is found in the associated method blank as well as in the sample.

2. U The analyte was analyzed for but not detected above the reported sample detection limit (essentially the analyte was not detected).

3A. E **(Inorganics)** The reported value is estimated because of the presence of interference.

3B. E **(Organics)** Compound exceeds the upper level of the calibration range and needed to be diluted.

4. M Duplicate injection precision was not met.

5A. N **(Inorganics)** Spiked sample recovery not within control limits.

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5B. N **(Organics)** The analysis indicates the presence of an analyte for which there is enough evidence to make a "tentative identification". (Mass spec analysis only)

6. S The reported value was determined by the method of standard additions (MSA).

7. * Duplicate analysis not within control limits.

8. + Correlation coefficient for the MSA is less than 0.995.

Entering "S", or "+" is mutually exclusive. No combination of these qualifiers can appear in the same field.

9. J **(Organics)** The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample (the result is an estimate below the detection limit)

10. P Used with pesticide/aroclor identification in which there is a 25% or more difference between the original analysis and the confirmation run. The higher value should be reported and flagged. (**Organics only**)

11. C Pesticide result was confirmed by GC/MS (**Organics only**)

12. D Represents a sample result that had to be diluted.

13. NJ The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration (**Organics only**).

14. R Sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified. A narrative explanation is provided.

15. UJ The material was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise.

9.1.6 If a problem arises after the report has been issued to the client and the client requests a re-analysis or an addition to the original report, an amended report will be issued. All of the client and sample information contained on the original report will also be present in the amendment. All corrections or additions to a test report shall be documented on a customer complaint form and/or discrepancy report. A comment will be made on the amended report stating that it is an amended report. The original laboratory ID number will be referenced in the amended report comment. If equipment in the analytical laboratory is found to be unfit for use and/or out of calibration, it shall not be used and shall immediately be withdrawn from service. If a piece of equipment is found to be unfit for use, the laboratory shall, if necessary, examine previous test results and notify affected clients if deemed necessary. An amended report will be issued if warranted.

9.2.0 **Completed worksheets** will be entered into the LIMS system by the analysts. All raw data and processed data from the laboratory is submitted in the form of raw data worksheets to the Laboratory and/or QA Manager or designee for review and validation in the LIMS system. These worksheets will be initialed and dated in ink by the analyst performing the analysis prior to submittal. Only the QA, and/or Laboratory Manager will validate results in the LIMS system. When the QA or Laboratory Manager or designee has validated all of the analyses for a sample in the LIMS system, the preliminary report will be generated, along with a preliminary invoice. The Administrative Staff will match the preliminary invoice with the preliminary report and submit them all to the Laboratory Manager or designee for final review. Upon completion of the review, the Manager or designee will return the reports and invoices to the staff for any corrections that are needed and for release of those reports. These reports are stamped and dated by the computer and matched up with the final invoice prior to sending them out to the client. This task may be accomplished by either of the administrative staff. All raw data will be kept in storage a minimum of five years or more if required by law. For all State of Massachusetts raw data and reports must be kept for a minimum of 10 years. Computer stored data is archived to disk or tape storage and

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kept as above. Any changes to final reports or to the raw data must be initialed and dated, with the full knowledge of the QA Manager. If final reports are changed after submittal to the client, a customer complaint form is initiated and if necessary, an amended report will be issued with an amended comment on it.

9.3.0 **Analysis of all samples** will require the following:

9.3.1 Log book entries:

* To be signed and dated
* To be entered in a chronological manner
* To be entered onto sequentially numbered pages

9.3.2 Instrument run logs will be maintained to ensure the reconstruction of the run sequence of individual instruments.

9.3.3 Completed sample data file package (CSF) must include a list of all enclosures, when, where, and to whom it was sent, and the method of shipment.

9.3.4 A transmitted letter must be sent to Viar & Company and NEIC/CEAT stating that the CSF was submitted.

9.4.0 In the event that a PUBLIC DRINKING WATER SUPPLY SAMPLE exceeds the allowable concentration the operator of the system and the appropriate regulatory agency will be notified within the time limit set (24 hours) by their regulatory authority.

9.5.0 Analytical records shall be kept for a period of five (5) years. For the state of Massachusetts, all records pertaining to samples received from Massachusetts will be kept for a minimum of 10 years. The following data records are maintained by NEL:

* Final reports
* Analytical standards books
* Analytical run logs
* Raw data produced by instruments
* All controlled documents

9.6.0 If the laboratory goes out of business, the laboratory will contact the client regarding the disposition of data (i.e. either return to the client or disposal).

9.7.0 Any changes in laboratory ownership, location, management, quality control personnel or any other change that significantly affects the laboratory's capability, scope of accreditation or ability to meet accreditation requirements, shall be reported in writing to all the appropriate accrediting authorities within twenty (20) business days of the change.

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Northeast Laboratory SERVICES
P.O. Box 796
Waterville, Maine 04903-0796

227 China Road
Winslow, Maine 04901

ANALYSIS REPORT

Attention: AILLI
NEL CHEMISTRY
NEL
DO NOT MAIL ME 04901

Lab ID Number: AJ08210
Project Number: WS 132
P.O. Number: AJ08208-2
Date Collected: 07/11/2007
DateReceived: 07/11/2007
Date Reported: 08/07/2007

Sample Matrix: D-WATER
Sample Description: Inorganics
Sample Type: Grab

Parameter	Result	Qualifier	Unit	Detection Limit	Method	Preparation Date/Time
Alkalinity, Titrimetric	42		mg/L	1	EPA 310.1	07/18/2007 10:1
Chloride	58		mg/L	2	EPA 325.2	07/24/2007 22:4
Specific Conductance @ 25 degrees C	540		umhos/cm	1	EPA 120.1	07/23/2007 14:5
Fluoride	1.6		mg/L	0.4	EPA 300.0	07/24/2007 22:4
Potassium Total	38		mg/L	2	EPA 200.7	08/06/2007 15:4
** A 1:2 dilution was performed in order to bring the concentration of potassium into the calibration range. Reporting limit has been increased to factor.						
Nitrate Nitrite Total	7.8		mg/L	0.05	EPA 353.2	07/26/2007 12:5
** A 1:3 dilution was performed in order to bring the concentration of NO2-NO3 into the calibration range. The reporting limit has been adjusted						
Nitrate-N	1.8		mg/L	1.5	EPA 353.2	07/26/2007 12:5
** A 1:3 dilution was performed in order to bring the concentration of Nitrate into the calibration range. The reporting limit has been adjusted ac						
Sulfate	86		mg/L	1.0	EPA 300.0	07/24/2007 22:4
Total Dissolved Solids	330		mg/L	2	SM 2540 C	07/12/2007 14:1
** Reporting limits are adjusted for sample volume.						

Comments:

Please see the specific analysis comment(s) above.

Results are reported on a wet weight basis.

This report shall not be reproduced, except in full, without written permission from Northeast Laborato

Results meet the requirements of the NELAC standards unless otherwise noted above.

Reviewed By: James F. Galasyn **Review Date:**
James F. Galasyn, Ph.D., Chemistry Lab Manager

Analytical results and reports are generated by NEL at the request of and for the exclusive use of the person or entity (cl
Results, reports or copies of same will not be released by NEL to any third party without the prior express written consen
report. This report applies only to those samples taken at the time, place and location referenced by the client. This re
implied warranty or guarantee as to the sampling methodology used by the individual performing the sampling. The clie
use and interpretation of these results and NEL makes no express or implied warranties as to such use or interpretation.
does not make a determination as to the environmental soundness, safety or health of a property from only the samples
analysis. Unless otherwise specified by the Client, NEL reserves the right to dispose of all samples after the testing of s
completed or after a thirty-day period, whichever period is greater. NEL liability extends only to the cost of the testing.

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Data Included	Level 1		Level 2		Level 3		Level 4	
	Inorganic	Organic	Inorganic	Organic	Inorganic	Organic	Inorganic	Organic
Cover Letter	X	X	X	X				
Report of Analysis	X	X	X	X	X	X	X	X
Chain of Custody Form	X	X	X	X	X	X	X	X
Sample Receipt Condition Report	X	X	X	X	X	X	X	X
Method Blank Results			X	X	X	X	X	X
Surrogate Summary				X		X		X
Surrogate Charts				X		X		X
Lab Control Sample Results (LCS)			X	X	X	X	X	X
Sample Duplicate Results(if performed)			X	X	X	X	X	X
MS/MSD Results (if performed)			X	X	X	X	X	X
Chromatograms			NA		NA		NA	X
Formal Project Narrative					X	X	X	X
Sample Run Log					X	X	X	X
Tentatively Identified Compounds (TICs)		MS-upon request		MS-upon request		MS-upon request		MS-upon request
Tune Summary						MS only		MS only
Initial Calibration Summary					X	X	X	X
Continuing Calibration Summary					X	X	X	X
Instrument Blank Results					X			
Internal Standard Area and RT Summary						MS only		MS only
Pesticide Breakdown Summary						Pest only		Pest only
Analytical Sequence					X	X	X	X
Pesticide Florisil Cartridge Check						Pest only(if appl)		Pest only (if appl)
Pesticide GPC Calibration Summary						Pest only(if appl)		Pest only (if appl)
Pesticide Ident. of Analyte Summary						Pest only(if appl)		Pest only (if appl)
CRDL Standard for AA and ICP					X		X	
ICP Interference Check Sample					X		X	
Standard Addition Results					X		X	
Post-digestion Spike Results					X		X	
ICP Serial Dilutions					X		X	
IDL Studies					if requested	if requested	if requested	if requested
MDL Studies					if requested	if requested	if requested	if requested
ICP Interelement Correction Factors					X		X	
Raw Data							X	X
Sample Preparation Logs					X		X	X
Internal Chain of Custody Forms							X	X

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CHAPER 10

CORRECTIVE/PREVENTIVE ACTION PROCEDURES

10.1.0 Introduction

This standard Laboratory Quality Assurance Procedure (LQAP) provides information on corrective/preventive action procedures performed by Northeast Laboratory.

10.2.0 Procedure

10.2.1 Corrective and preventive actions are assigned, performed and documented as described in NEL SOP# 3.1.4 "Process Improvement Goals (PIG) Program" and through the use of discrepancy reports and customer complaint forms attached to this LQAP chapter. The QA Manager will keep a log of each discrepancy customer complaint and PIG for tracking within the discrepancy database created by the QA Manager.

10.2.2 Any corrective or preventive action resulting in changes to documented procedures shall be implemented and recorded according to SOP 3.1.4 "Process Improvement Goals Program".

10.2.3 Any corrective or preventative action taken to eliminate the causes of actual or potential non-conformities shall be to the degree appropriate for the magnitude of the problem and the risk encountered.

10.3.0 Control of Nonconforming Testing

10.3.1 NEL handles nonconforming work via NEL SOP 3.1.4, which addresses complaints and corrective actions. Nonconforming work would result in an internal complaint and subsequent investigation and corrective action.

(a) It is the responsibility of all personnel to report nonconforming work to the area supervisor, laboratory director or quality manager.

(b) The supervisory staff assesses the significance or the nonconforming work.

(c) Corrective action plans are implemented.

(d) If a report was issued the client is notified and if necessary, an amended report is issued.

(e) Work is resumed with the supervisory staff and quality manager's authorization.

10.4.0 Corrective Action

10.4.1. The procedures for corrective action shall include:

10.4.1.1 The effective handling and documentation of customer complaints and reports of non-conformities (customer complaint form attached).

10.4.1.2 Process and Quality System Regulation, and recording the results of the investigation.

10.4.1.3 Determination of the corrective action needed to eliminate the root cause of non-conformities.

10.4.1.4 Application of controls to ensure that corrective action is taken and that it is effective.

Initiated By: Total Quality Manager

Approved By: Pamela Doughty
Total Quality Manager

Approved By: A Haley
Chemistry Lab Manager

Approved By: Brian Mordock
Microbiology Manager

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10.4.2 NEL shall establish and maintain a documented feedback system to provide early warning of quality problems and for input in the corrective action system.

10.4.3 After a report has been released to the client, any failure of that report to meet the client's data quality objectives shall be investigated. A written record of the investigation, including conclusions and follow up, shall be made (see customer complaint form).

10.4.4 NEL shall notify the appropriate regulatory authority of any data related incidents or proficiency failure that may affect NEL's accreditation status.

10.4.5 NEL shall establish, document and maintain procedures for contacting clients regarding any quality issues that may affect their data.

10.4.6 All feedback information such as reported customer complaints and reanalysis shall be documented, investigated, interpreted, collated and communicated in accordance with defined procedures by a designated person. If any complaint is not followed by corrective action, the reason shall be recorded

10.4.7 Trend analysis shall be performed on customer complaints, if the number of complaints is large enough for meaningful application of statistical rules.

10.4.8 NEL shall maintain records of all complaint investigations.

10.5.0 Implementation Summary

Early warning of quality problems is provided by Discrepancy Reports (DR) (see attached form). Whenever there is any quality problem within the lab which may affect, in any way, the quality of the data NEL generates, a DR is generated to document the problem and appropriate corrective action.

10.5.1 Data, which fails Quality Control criteria, is investigated but never reported until reanalysis can occur that may confirm or deny matrix or otherwise identify the cause of the quality control failure.

10.5.2 The following procedure is to be applied when unacceptable results are obtained in the analysis of quality control samples. The steps are to be done in sequence. If at any step the problem is corrected, the sequence can be terminated.

10.5.1.1 The analyst shall inform the Laboratory Manager/or Quality Manager that the above situation has occurred.

10.5.1.2 Raw data, equations and calculations used are to be validated and verified as correct or corrected as necessary.

10.5.1.3 Calibration of the instrument is to be tested with check standards. In all cases, if the instrument calibration does not meet criteria the following actions shall be taken: For single analyte analyses, the operation is stopped. The procedure is checked for errors, including instrument problems and standard solution problems. Routine maintenance and remaking standards may be required. Data obtained during an out of calibration time may not be reported without an indication of the exact problem and whether the data may be biased high or low. This should only occur when all other attempts (i.e. instrument maintenance, subcontracting) have failed. All efforts must be made not to report data obtained during calibration problems. Reporting data out of calibration without noting the exact problems is considered fraud. For multi analyses, all efforts must be made to meet calibration criteria for all analyses. If multiple attempts at calibration have failed with less than 10% of the analytes being less than 10% out of criteria, the data may be flagged as being biased high or low. A corrective action must be initiated to determine the problem and prevent reoccurrence. Reporting data out of calibration without noting the exact problems is considered fraud.

10.5.1.4 Concentration of the check standards will be verified. If any question as to their accuracy (age of standard, etc.) exists, a fresh one will be prepared and used.

10.5.1.5 Quality control samples are to be reanalyzed. All samples run in the preparation and/or analytical batch with and out-of-control QC sample(s) will be reanalyzed.

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10.5.1.6 The instrument is to be shut down for complete calibration, cleaning, tuning, aligning, etc. as recommended by the manufacturer.

10.5.1.7 The client/customer is to be notified of the problem and given an estimate of time for the delay.

10.5.1.8 Documentation of the problem and its resolution will be supplied to the QA Manager in the form of a Discrepancy Report.

10.5.1.9 The system in question will have a new calibration curve and its verification performed prior to being placed back in service.

10.5.1.10 Service related customer contact comments are processed, investigated, recorded and where appropriate, fed into the corrective action system by the individual receiving the complaint or comment. Each customer complaint will be detailed on the attached customer complaint form.

10.6.0 Preventative Action

NEL uses different systems to generate and analyze quality data for preventive actions. Being a small company, NEL employees and managers interact constantly within and between departments. Furthermore, upper Management is intimately involved with issues as they happen; often time information is conferred as it happens. This process allows for rapid evaluation of issues and quick response with preventative actions.

10.6.1 The procedures for preventative action shall include:

10.6.1.1 The use of appropriate sources of information such as processes and work operations which affect data quality, audit results, quality records, proficiency reports and customer complaints to detect, analyze and eliminate potential causes of non-conformities.

10.6.1.2 Determination of the steps needed to deal with any problem requiring preventive action.

10.6.1.3 Initiation of preventive action and application of controls to ensure that it is effective.

10.6.1.4 Ensuring that relevant information on actions taken is submitted for management review.

10.6.2 Implementation Summary

10.6.2.1 NEL's formal preventive action system is outlined here with additional process improvement, if necessary, outlined in NEL SOP #3.1.4 - Process Improvement Goal (PIG) Program. The procedures described in the corrective action section above also apply to preventive actions.

10.6.2.2 The effectiveness of corrective and preventive actions implementation is tracked through the Discrepancy/Process Improvement Goal Form (PIG) NEL SOP #3.1.4 and the discrepancy database.

10.6.2.3 At the close of each internal or external Quality Audit, DRs are assigned to address all remaining Quality System problems, if any, uncovered during the audit. The purpose of such DRs is to assure that procedure and/or system changes are made to preclude recurrence of these or similar problems. A follow up audit assesses the effectiveness of actions recorded on the DR form.

10.6.2.4 Customer Complaints and Discrepancy Reports are evaluated quarterly by QA. Trending is used only if volume is high enough to apply statistical methods yielding meaningful results. Preventive action will be included if indicated.

10.6.2.5 The status of the Quality System is reviewed annually by Management; among the data reviewed by Management are summaries of PIGs/Discrepancy Reports, quality audit results, proficiency results, and customer complaints.

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Dr #_____
Date:_____

DISCREPANCY REPORT

A)

Person who discovered the problem			Possible ramifications if not discovered:
Sample #:	Client:	Lab Area:	

B) Describe Discrepancy:

Problem Type:

	Equipment		Insufficient Sample		QC Failure
	Process Change Effectivity		Lab Accident		Proficiency Failure
	Log-in		Labeling Error		Other (describe below)
Initiator:				Date:	

C) Recommended Disposition:

	Accept as is/No Process Change Required (provide justification below)		Reanalyze Sample
	Accept as is/Process Change Required (P.C.#:)		Reissue Report
	Discrepancy Corrected or Void		
Comments:			
Initiator/Supervisor:		Date:	

D) Authorization:

Department	Signature	Date
QA Manager		
Laboratory Manager		
Technical Manager		
Supervisor		
Other Appropriate Party		
Comments:		

E) Final Disposition:

F) Corrective/Preventive Action:

Performed by:		Date:	
Verified by:		Date:	

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RETURN/CREDIT AUTHORIZATION

Authorization No.:	Recorded by:	Date:

CUSTOMER INFORMATION		REPORT INFORMATION	
Account Name:			
Account #:		Sample #:	
Contact Name:		Report Date:	
Phone #:		Invoice Date:	

ADDITIONAL INFORMATION (Required for Credits Only)			
Original Invoice/Sales Order #		Price Charged:	
Credit Amount:		Freight Charged:	

REASON FOR RETURN/CREDIT				
Department				Reason for Return/Credit
	Microbiology Lab		Log-In	
	Chemistry Lab		Miscellaneous	

FINAL DISPOSITION (Check all that apply)					
	Credit		Reissue Invoice		Reissue Report

FOLLOW-UP/CORRECTIVE ACTION

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CUSTOMER COMPLAINT FORM

Date:		By:		Type:		Other
						Laboratory Use Only

Laboratory Number:		Customer:	
Date Received:		Customer #:	
Date Reported:		Address:	
Department Affected:		Inquiry Made by:	
P.O. #		Phone #:	

Customer Report:

Technical Service:
Summary Response & Recommended Action (if any):

Disposition:					
Issue Credit:		Yes		No	Credit Memo #:	
Laboratory #					Amount:	
Credit Issued:	Date:				By:	
Reanalyze Sample:		Yes		No	Parameter(s) Reanalyzed:	
New Lab # for Reanalysis:					By:	
Reissue Report:		Yes		No	Report Reissue Date:	
Reporting/Accounting Notified to Issue Credit:						

RETURN FORM TO QA UPON COMPLETION OF DISPOSITION

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CUSTOMER COMPLAINT FORM
(pg. 2)

QA FOLLOW-UP TO CUSTOMER COMPLAINT

Client Name:		Sample No.(s)	

COMPLAINT:

Contamination		Other:	
Performance			

RETEST PROCEDURE & RESULTS:

Similar Complaints for this Dept?	Yes		No		How Many?	
Date Investigation Completed:				By:		
Date Technical Service Review:				By:		
Conclusions:						

Date:		Laboratory Manager:	

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CHAPER 11

PERFORMANCE AND SYSTEM AUDITS

11.1.0 Definitions

Performance audits are independent checks used to evaluate the quality of data produced by the analysis system. These audits are performed by the analyst independent of and in addition to, normal quality control checks. A system audit is an on-site inspection and review of the quality assurance system. Whereas performance audits are a quantitative appraisal, system audits are for the most part qualitative appraisals.

The Total Quality Manager shall administer the audit system to ensure that data of known and defensible quality are produced. Performance audits shall be conducted periodically; however, certain contracts/project plans may involve more specific checks on the analytical systems. Performance audits will be performed using EPA references or check samples for wastewater and drinking water standards, groundwater and/or solid matrices.

11.2.0 Performance Audits

11.2.1 Performance Audits require testing measurement systems with samples of known composition to evaluate precision and accuracy.

11.2.2 Standard Reference Material samples are blindly submitted to the analysts, for analysis. These SRM samples are used as a quality control device in the performance audits.

11.2.3 Results of the analyses of Performance Audits shall be evaluated and deficiencies shall be identified. Corrective action forms shall be completed where applicable.

11.3.0 System Audits

11.3.1 Internal System Audits are qualitative evaluations of all components of the laboratory quality control measurement systems. They determine if the measurement systems are being used correctly and will be done on an annual basis. Selected projects may require a different format for analytical measurement systems of interest. If the results of internal system audits indicate a problem with data that has already been reported to a client, the client will be notified in writing. Systems audits will include a review of the following:

- Standard Operating Procedures (S.O.P's)
- Instrument Calibrations
- Maintenance Logs
- Standards and Reagents Logs
- Compliance to Current Methods and knowledge to those methods
- Tracebility
- Good Laboratory Practice (GLP)
- Safety
- Waste Disposal

11.3.2 Audits will be conducted by trained technical personnel with sufficient authority, access to work areas and freedom to observe all activities and report findings to QA and other laboratory management.

11.3.3 All internal systems audits will be recorded on audit checklists and stored with specific method SOP's. Any corrective actions are entered and monitored through the discrepancy database.

Initiated By: Total Quality Manager

Approved By: Total Quality Manager

Approved By: Chemistry Lab Manager

Approved By: Microbiology Manager

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11.3.4 External system audits include proficiency testing for Water Supply (WS), Water Pollution (WP), Soil and MAPEP studies required by NELAC and the State of Maine. As a participant in the State Certification Program and NELAC, it is required that NEL receive these external audits. NEL follows the NELAC requirement to participate in two PT Studies each year for

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CHAPTER 12
INSTRUMENT AND EQUIPMENT MAINTENANCE

12.1.0 Up to date instructions in the use and maintenance of equipment, including any relevant manuals shall be readily available for use by the appropriate laboratory personnel

12.2.0 Analytical measurement instruments and equipment are subject to routine and non-routine maintenance. Routine/Preventative Maintenance is usually performed by the analyst/operator.

12.3.0 Non-routine maintenance actions can be performed by the analyst/operator, Laboratory Manager, or instrument manufacturer's service technicians. Non-routine maintenance actions are taken when instruments are "out of control"/inoperable.

12.4.0 All maintenance is to be performed as recommended by the manufacturer's instruction manuals.

12.5.0 All equipment in the analytical division will be clearly marked of their status, either in service or out of service, and will also be documented in the labs equipment maintenance log.

INORGANIC SECTION

Year	Type of Equipment	Manufacturer	Model	Analyses
1988	HGA-Graphite Furnace	Perkin Elmer	5100PC	Drinking Water,G.W., SW & Solid Metals
1988	w/Autosampler	Perkin Elmer	AS-60 (Not in service)	
1995	Optima 3000XL I.C.P.	Perkin-Elmer	3000XL	Metals
1995	w/Autosampler	Perkin-Elmer	AS-91	
1995	Automated-Mercury System	Perkin-Elmer	Fims 100	Mercury
1995	w/Autosampler	Perkin-Elmer	AS-90	
1996	Optima 3000DV I.C.P.	Perkin-Elmer	3000DV	Metals
1996	w/Autosampler	Perkin-Elmer	AS-90	
1996	Digestion Microwave	CEM	MDS 2100 (Not in service)	
2003	Furnace	Perkin-Elmer	Simma 6000	Drinking Water,G.W., SW & Solid Metals
2005	Digi Prep Digestion System	SCP Science	Verscon 5	Metals Digestion

PHYSICAL CHEMISTRY SECTION

Year	Type of Equipment	Manufacturer	Model	Analyses
1985	Turbidimeter	Hach	2100 A	Turbidity
1985	w/Spectrophotometer*	Milton Roy	Spec 21D	
1985	w/Spectrophotometer*	Bausch and Lomb	Spec 21	
1986	Auto Analyzer	Technicon	TRAACS	NO2, NO3, Chloride, Ammonia
1986	w/Data Station	IBM PC	AT	
1986	w/Software	Bran & Luebbe	Ver 3.03	
1986	Carbon Analyzer	Dohrmann	DC80	TOC/TIC
1986	w/TOC Autosampler	Dohrmann	ASM 1	
1988	Liquid Scintillation Analyzer	Packard	Tricarb 4640	NO2, NO3, Chloride, Ammonia
1993	Auto Analyzer	Technicon	TRAACS	
1993	w/Data Station	Duracom PC	486/33	
1993	w/Software	Bran & Luebbe	Ver 3.03	
2000	Ion Chromatograph	Dionex	600DX	Anions
1998	Auto Burette, sample changer with 6.3 software and titration manager	Radiometer/Copenhager	TIM 90W TIM 900 ABV 900 SAC 80	Alkalinity, pH

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Total Quality Manager

Approved By: K Halas
Chemistry Lab Manager

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Microbiology Manager

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2007	Ion Chromatograph	Lachat		
2007	Carbon Analyzer	Lachat		TOC

ORGANIC SECTION

Year	Type of Equipment	Manufacturer	Model	Analyses
1986	GC #1 Capillary/Packed Gas Chromatograph	Perkin Elmer	Sigma2000	GRO
1986	W/FID/ECD/NPD			
1986	W/Data Station	Epson PC	Equity3	
1992	W/Software	Perkin Elmer	TurboChrom 4.0	
1995	Archon	Varian	5100	
1988	GC #2 Capillary/Packed Gas Chromatograph	Perkin Elmer	8500	Not in use
1986	W/Hall & PID in series			
1986	W/Autosampler: Head space	Perkin Elmer	HS-101	Not in use
1986	W/Autosampler: Purge & Trap	Tekmar	3000	GRO
1986		Tekmar	LCS2016	
1986	W/Data Station	Epson PC	Equity3	
1992	W/Software	Perkin Elmer	TurboChrom 4.0	
1988	GC #3 Packed Gas Chromatograph	Perkin Elmer	8500	Not in use
1986	W/ECD/FID			
1986	W/Data Station	Duracom PC	486/33	
1992	W/Software	Perkin Elmer	TurboChrom 4.0	
1989	GC #5 Capillary/Packed Gas Chromatograph	Hewlett Packard	5890-II	Chlorinated Organics
1989	W/ECDx2			
1989	W/Autosampler	Hewlett Packard	7673	
1989	W/Data Station	Duracom PC	486/33	
1992	W/Software	Perkin Elmer	TurboChrom 4.0	
1990	Cap./Packed GC/Mass Spectrometer	Hewlett Packard	5890II/5970B	Volatile Organics
1990	GC/MS #1 w/Autosampler: Semivolatiles	Hewlett Packard	3396A	
1990	w/Autosampler: Purge & Trap	Tekmar	LSC2000	
1990	Autosampler	Archon	5100	
1997	w/Data Station	Hewlett Packard	HP Chemstation	
1997	W/Software & NBS 49K Library	Hewlett Packard	Pentium 133	
1991	GC #6 Capillary Gas Chromatograph	Perkin Elmer	Auto/system	DRO
1991	w/FID			
1991	w/Autosampler	Perkin Elmer		
1991	w/Data Station	Epson-PC	Equity3	
1991	w/Software	Perkin Elmer	OMEGA 4	
1989	GC/MS	Hewlett Packard	5890/3396A	Semi-volatile Organics
	w/Autosampler	Hewlett Packard	7673A	
1998	GC #7 Gas Chromatograph w/FID/ECD w/autosampler 7673A	Hewlett Packard	HP 5890 Series II Plus	DRO, PCBs, Pesticides, Herbicides
2001	Total Organic Halogen Measurement System	Mitsubishi Kasei	TOX-10E	TOX, AOX
2001	Gas Chromatograph w/FID/ECD, Autosampler 7673A	Hewlett Packard	HP5890	GRO
2001	Purge & Trap	Tekmar	3000	GRO
2001	Autosampler	Tekmar	ALS2016	GRO
1994	GC/MS with Autosampler	Finnigan Varian	MAT MS 2	Volatile Organics (convertible to Semi-volatile organics)

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1986	Purge & Trap	Tekmar	2000	VOA
1986	Autosampler	Tekmar	ALS2016	VOA

MICROBIOLOGY SECTION

EQUIPMENT	NEL#	DESCRIPTION/MODEL #	SERIAL #	ID	IN SERVICE
BALANCES		Ohaus Scout Balance SC6010	BJ216064	B-1	Thrown Out
		Ohaus Scout Balance SC6010	BJ221916	B-2	Out Of Order
	5291	Metlet BasBal BB300	J41185	B-3	In Service
	6004	Sartorius 2842	2701097	B-4	Out of Service
		Ohaus Scout Pro SP401	7122310294	B-5	In Service
		Sartorius A2005	37030211	B-7	In Service
INCUBATORS	5029	Precision 30M (35oC +/- 0.5o)	9405-012	I-1	In Service
	5013	Fisher Isotemp 255D (55oC +/- 0.5o)	2326	I-2	In Service
	5014	VWR 1530 (30oC+/- 1.0o)	1530587	I-3	In Service
	5119	Precision Thelco 4 (28oC +/- 1.0o)	20-Z-5	I-4	In Service
	6036	Tenney THjr (30oC +/- 0.5o)	9878-520	I-5	In Service
	5006	Fisher 146 Low Temp Inc 61AR (25o)	82487129	I-6	Out of Service
	5028	Fisher Econotemp 55D (40oC +/- 1.0o)	163	I-7	In Service
	5007	Fisher 307 Low Temp Inc FU199LRW3 (4o +/- 2.0o)	WB10507333	I-8	In Service
	6192	Thermo Electron Corp 3502	102821	I-9	In Service
THERMOMETERS		Fisher Total 14-983-10B	N/A	T-1	Out of Service
		Ertco Exact Temp I-310-01(25-45oC)	2082	T-2	In Service
		VWR Total 61054-503 (20-50oC)	91-2128	T-3	In Service
		Ertco F-010-1	4219	T-4	In Service
		Ertco Refrigerator Thermometer R-020	12977	T-5	In Service
		Ertco Oven Thermometer OV-070	12936	T-6	In Service
		Ertco I-030	10327	T-7	In Service
		Ertco I-030	2731	T-8	In Service
		Ertco Refrigerator Thermometer R-020	13127	T-9	In Service
		Ertco I-030	8874	T-10	In Service
		Ertco Refrigerator Thermometer R-020	10804	T-11	In Service
		Ertco F-010-1	6535	T-12	Broken
		Ertco Refrigerator Thermometer R-020	13181	T-13	In Service
		Ertco F-010-1	7507	T-14	In Service
		Ertco Refrigerator Thermometer R-020	13119	T-15	In Service
		Ertco I-030	3020	T-16	In Service
		Ertco	6588	T-17	In Service
		Ertco Refrigerator Thermometer R-020	13045	T-18	In Service
		Fisher 15-171-2	7095	T-19	In Service
		Eversafe 76mm N16B	16120	T-20	In Service
		ASTM 64C	2791	T-21	Out of Service
		Eversafe 76mm N16B	15890	T-22	In Service
		Ertco Refrigerator Thermometer R-020	13208	T-23	In Service
		VWR Frio-Temp61225-716	T41663	T-24	In Service
		GCA/PS (20-100oC)	314950	T-25	In Service
		Fisher Brand Refrigerator Thermometer	15-035	T-27	In Service
		Fisher Brand Refrigerator Thermometer	20598	T-28	In Service
		Fisher Brand Refrigerator Thermometer	22499	T-29	In Service

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		Barnstead International	13994	T-31	In Service
		Ertco Incubator Thermometer	13075	T-32	In Service

EQUIPMENT	NEL#	DESCRIPTION/MODEL #	SERIAL #	ID	IN SERVICE
REFRIGERATORS	5015	Panasonic Combo NR1625W	870412142	R-1	In Service
		Frigidaire 125A		R-2	Out of Service
	5008	Unknown	107032-1	R-3	In Service
	5318	Marvel 61ARS	22692049	R-4	In Service
	5319	U Line 65R	65-66890	R-5	In Service
	5317	U Line 75R	75R101426	R-6	Out of Service
		U Line Combo 75	75C113281	R-7	In Service
	5117	Glenco Guardian/ ALA-74TE	BB-278055	R-8	In Service
	5115	Indesit	88100617	R-9	In Service
FREEZERS	5015	Panasonic NR1625W	870412142	F-1	In Service
		Frigidaire 125A	10AL9559	F-2	Out of Service
	5008	Unknown	107032-1	F-3	In Service
WATER BATHS	5016	Fisher Versabath	66850	W-1	In Service
	6106	GCA/Precision elev.col.inc.bath	10AE9-110	W-2	In Service
		Fisher 10L	9A2284	W-3	In Service
		LabLine Equatherm 299728	0523-0207	W-4	Out of Service
		Precision 186 66634-27	27AX-8	W-5	Out of Service
PIPETTES		Wheaton 50-200ul	08041134	P-1	In Service
		Fisherbrand Finnipipette 50-300ul	F37987	P-2	In Service
		Helena Labs Quickpipette 5-50ul		P-3	Out of Service
		Oxford Sampler Micropipetter 50ul		P-4	Out of Service
		Oxford Sampler P-7000 25ul		P-5	In Service
		Eppendorf 500ul-2500ul		P-6	Out of Service
		Excalibur by Ulster 1-5ul		P-7	Out of Service
		Excalibur by Ulster 5-250ul		P-8	Out of Service
		Eppendorf 10-100ul	4710 50514P	P-9	Out of Service
		MLA 1-5ml		P-10	In Service
		Autoclavable Oxford Benchmate 10-100ml		P-11	Out of Service
		Eppendorf 100-1000		P12	In Service
		Socorex 20-200 ul Pipette, Single Channel	15112702	P-13	In Service
AUTO PIPETTES		Drummond Pipet-Aid XP	70645	AP-1	In Service
		Drummond Pipet-Aid XP	70845	AP-2	In Service
		Drummond Pipet-Aid	52057	AP-3	In Service
		Drummond Pipet-Aid XP	66129	AP-4	Out of Service
		Drummond Pipet-Aid XP	87091	AP-5	In Service
AUTOCLAVES		Peltor & Crane Validator Plus 10		A-1	In Service
		Market Forge Sterilmatic		A-2	In Service
MICROSCOPES	6041	Fisher Scientific Stereomaster SPT-IH	LR33310	M-1	In Service
		Nikon Eclipse E600	762713	M-2	In Service
	6116	Nikon Labophot-2	436136	M-3	In Service
		Olympus CH	688314	M-4	In Service

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	5010	Nikon TMS	212301	M-5	In Service
		Leitz Laborlux S	512859/105352	M-6	In Service

EQUIPMENT	NEL#	DESCRIPTION/MODEL #	SERIAL #	ID	IN SERVICE
HOT PLATES		Fisher Stirring Hot Plate	70903341	SH-1	Out of Service
	5019	Fisher Thermamix Stirring Hot Plate	106	SH-2	In Service
		Fisher Sci Thermix Stirrer 120S	51202561	SH-3	Out of Service
		Fisher 14-5050-21	173104	SH-4	Out of Service
COLONY COUNTERS		Darkfield Quebec 3525		CO-1	In Service
	6040	Spectroline Fluorescence Cabinet CM-10		CO-2	In Service
	6039	Spectroline 365nm UV Lamp EA-160		CO-3	In Service
MF EQUIPMENT		Gelman Sciences 300 ml Filtration Cups	4242	N/A	In Service
		UV Sterilization Box		N/A	In Service
		Millipore Microbiological Dishes	PD10047S5	N/A	In Service
		Millipore Sterile 47mm 0.47micron filter	EZHAWG474	N/A	In Service
INNOCULATION		Kendall Presterilized Bioloop 510Z010STR		N/A	In Service
OVENS	5017	Boekel Industries 132000	04097-249	O-1	Out of Service
	5035	Boekel Industries 131500	02083-17	O-2	Out of Service
CENTRIFUGES	5042	IEC Centra 4B	23730111	C-1	In Service
HOODS	5011	NuAire, Inc. Cabinet NU-425-400	12691RW	H-1	In Service
MEASURING		Fisher 1mLx0.1mL disposable glass pipet	13-678-25A	N/A	In Service
		Fisher 10mLx0.1mL disposable glass pipet	13-678-25E	N/A	In Service
		Falcon 10mLx0.1mL disposable plastic pipet	357504	N/A	In Service
		Fisher 1mLx0.1mL disposable plastic pipet	3031003G	N/A	In Service
		Fisher 10mLx0.1mL disposable plastic pipet	1367812E	N/A	In Service
CULTURE DISHES		Prepared MI agar	NEL 36	N/A	In Service
		Prepared m-endo agar	NEL10xP6020-PS	N/A	In Service
		Prepared m-tec agar	NEL P7300	N/A	In Service
		Prepared R2A agar	NEL10xP6020-PS	N/A	In Service
		Prepared MEA agar	NEL 8	N/A	In Service
RINSE WATER		Phosphate Buffered Water	NEL 28	N/A	In Service
RINSE WATER		Phosphate Buffered Water	NEL 37	N/A	In Service
pH METER		Fisher XL15	XL94000297	PH-1	In Service

MAINTENANCE SCHEDULE:

Appropriate checks shall be made on-site to confirm the calibration status and fitness of use of equipment before testing or calibration begins; especially on equipment whose performance is sensitive to movement.

Balances: Balances are to be calibrated each day prior to use, and serviced annually by a suitable outside contractor.

Regular cleanings are to performed as needed.

Incubators: Incubators are to be cleaned as needed.

Thermometers: Thermometers are to be calibrated annually by a suitable outside contractor, or in-house following developed, method and performed by qualified personnel equipped with adequate resources.

Refrigerators: Refrigerators are to be cleaned as necessary.

Freezers: Freezers are to be defrosted and cleaned as necessary.

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Water baths: Water baths are to be drained and cleaned as necessary.
Autoclaves: Autoclaves are to validated annually and cleaned as necessary. The timers shall be checked quarterly.
Microscopes: Microscopes are to be wiped clean daily or more frequently if necessary, and calibrated annually by a suitable outside contractor.
Colony Counters: Colony Counters are to be wiped clean daily and bulb changed as necessary.
Membrane Filtration Equipment: MF equipment shall be sterilized as appropriate. The UV bulb shall be replaced as necessary
Ovens: Ovens are to be cleaned as necessary.
Centrifuges: Centrifuges are to be cleaned as necessary.
Hoods: Hoods are to be checked for performance each day of use, calibrated annually and cleaned as necessary.
Flow shall be monitored.
Pipettes: Shall be calibrated annually by a suitable outside contractor.
PBW NEL 37 : Shall be checked quarterly using the Students T Test evaluation against prior lots.
Automatic dispensers shall be calibrated quarterly.
Disposable pipettes: Shall be verified for performance for each new lot received in the laboratory.
Timers: Timers are to be checked quarterly against a NIST traceable reference.

* Cleaned and calibrated yearly by QC Services Inc.

12.6.0 Documentation Requirements

All maintenance actions shall be documented in the instrument maintenance notebooks located near instruments. It is the responsibility of the analyst/instrument operator performing the maintenance or supervising the repairs by service technicians to complete maintenance documentation. A maintenance schedule may be found in Section 18 of each NEL SOP involving instrumentation.

Maintenance notebooks are specific to each measurement system. The maintenance notebooks shall contain the following information (please see NEL SOP #3.7.10 for further information) :

* The name of the instrument
* Manufacturer's name, model and serial number
* Location of the instrument
* Date and condition of the instrument when it was put into service
* Type of repair/preventative maintenance
* Person performing the repair/maintenance
* Date of repair/maintenance
* Action narrative as specified below

If the instrument cannot be repaired, it must be labeled "Out of Service" until it is replaced or repaired.

Regardless of whether the maintenance action is routine or non-routine the action shall be documented in a chronological order in the appropriate maintenance notebooks.

The action narrative will specifically include:

A) Non-Routine Maintenance: Date, signature, narrative of symptom that caused instrument "out of control" or inoperable condition. Or narrative of the corrective action taken that specifically references the proof of correctly operating equipment. A list of parts needed in repair and a copy of the field service technician's reports/work order if applicable.

B) Routine Maintenance: Date, signature, and narrative of maintenance action.

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CHAPTER 13

GOOD LABORATORY PRACTICES

13.1.0 Introduction

Intrinsic to the production of quality analytical data is the quality of laboratory services available to the analyst. Without adequate quality control being exercised with regard to facilities, services, laboratory environment, instrumentation and laboratory supplies, an analyst cannot be expected to provide reliable analytical data. To that end, NEL has strict guidelines for the purchase of supplies and services. In the Analytical Division, the purchasing of services and supplies was developed to ensure that vendors are selected that continually adhere to our high quality of standards. The SOP detailing this procedure is NEL SOP # 4.1.4. In the Chemistry Department, specific guidelines can be found in each method SOP. Recognizing the necessity of maintaining control over general laboratory operation, the following good laboratory practices shall be performed. Quality checks for sample bottles and equipment in SOP 3.7.1.

13.2.0 Preparation/Storage/Documentation of Standards

13.2.1 Standards will be prepared and stored according to specific methodology.

13.2.2 Preparation of stock standards that are NIST traceable will be recorded in the appropriate standard logbook with the following information:

- date prepared, and analyst
- analyte source, lot number, solvent and lot number
- final concentration
- expiration date

13.2.3 The identification of each standard prepared should be unique. Documentation should be sufficient so that a standard used in an analysis can be traced back to the original stock standard.

13.2.4 The original standard or reagent container must exhibit an expiration date.

13.3.0 Microbiological Reference Cultures

In order to demonstrate traceability and selectivity, Northeast Laboratory shall use reference cultures of microorganisms obtained from a recognized national collection or close traceability to such a collection.

13.3.1 Reference cultures may be subcultured once to provide reference stocks and shall be preserved by a technique which maintains the desired characteristics of the strains, such as liquid nitrogen storage or other accepted deep-freeze methods.

13.3.2 Reference stocks shall be used to prepare working stocks for routine use.

13.3.3 Working stocks shall not be subcultured sequentially more than five (5) times, except when:

a) It is required by standard test methods

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Total Quality Manager

Approved By: [illegible]
Chemistry Lab Manager

Approved By: [illegible]
Microbiology Manager

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b) The laboratory can provide documented evidence demonstrating that there has been no loss of validity, no changes in biochemical activity and/or morphology.

13.3.4 Working stocks shall not be used to prepare reference stocks.

13.4.0 Laboratory Supplies

13.4.1 Glassware

a. Glassware used in general laboratory operations must be of a high quality borosilicate glass, e.g., "Pyrex" or "Kimax." Class "A" volumetric glassware will be used when required by methodology.

b. Clean glassware in hot water with a suitable detergent, rinse in hot water to remove detergent residue, and will be rinsed in laboratory pure water. Glassware used in special analyses, e.g., metals and organics may require more scrupulous cleaning, e.g. acid and/or solvent rinsing.

c. Perform the Inhibitory Residue Test on initial use of a washing compound and whenever a different formulation of washing compound is used in order to insure that microbiological glassware is free of toxic residue.

d. Add Bromthymol Blue Test for detection for residual alkaline or acidic residue (see SOP 3.7.1 Glassware Cleaning).

13.4.2 Chemicals, Reagents, Solvents, Standards, Gases, and Media

a. The quality of chemicals, reagents, solvents, standards gases and media used in the laboratory is determined by the sensitivity and specificity of the analytical technique being used. Reagents of lesser purity than specified by a method will not be used.

b. Reagents and media (excluding high-demand items) should be purchased in small quantities to minimize extended shelf storage.

c. All reagents, solvents, standards, and media are dated upon receipt and when opened or prepared. All are discarded when outdated, or when evidence of discoloration or deterioration is detected. All media, solutions and reagents shall be prepared, used and stored according to a documented procedure following the manufacturer's instructions on the test method.

d. Reagents and culture media may be prepared in the laboratory from the specified raw ingredients, from commercial dehydrated powders or may be purchased ready to use.

e. Reagents, commercial dehydrated powders and media shall be used within the shelf life of the product and be documented according to NELAP 5.10.5, or according to appropriate actions amending or superceding this section. The laboratory will retain all manufacturer-supplied "quality specifications" or "quality control" statements, which may contain such information as product shelf-life, storage conditions, assay results and acceptability criteria, and other such information validating the quality of the material.

f. All microbiological media shall be checked to ensure that they support the growth of specific (target) microbial cultures. In addition, selective media shall be checked to

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ensure they suppress the growth of non-target organisms. Media purchased as pre-prepared shall be checked monthly where the use of a single lot exceeds one month in time. When possible, a quantitative or semi-quantitative onoculum will be used in preference to the more common stock method, in order to ensure that low levels of organisms are used to verify adequate growth on the media.

13.4.3 Laboratory Pure Water

a. The laboratory pure water system consists of a reverse osmosis and deionization supply followed by a carbon module, two ion exchange modules and an organic module. The system is also equipped with a direct reading resistivity meter.

b. System modules are changed as recommended by the manufacturer or as indicated by water quality. Modules are dated and initialed when changed.

c. For microbiological analyses, the water used to make media and dilution water shall be free of biochemical and inhibitory substances. This shall be verified by an accepted method at least on a yearly basis.

d. The quality of the water shall be monitored for attributes such as pH, conductivity, chlorine residue, metals content and heterotrophic bacteria at specified frequencies and evaluated according to documented and accepted standards. Please refer to *Standard Methods for the Examination of Water and Wastewater* 20th Edition, see below.

TABLE 9020:II. QUALITY OF REAGENT WATER USED IN MICROBIOLOGY TESTING

Test	Monitoring Frequency	Maximum Acceptable Limit
Chemical tests:		
Conductivity	Continuously or with each use	>0.5 megohms resistance or <2 μmhos/cm at 25°C
pH	With each use	5.5–7.5
Total organic carbon	Monthly	<1.0 mg/L
Heavy metals, single (Cd, Cr, Cu, Ni, Pb, and Zn)	Annually*	<0.05 mg/L
Heavy metals, total	Annually*	<0.10 mg/L
Ammonia/organic nitrogen	Monthly	<0.10 mg/L
Total chlorine residual	Monthly or with each use	<0.01 mg/L
Bacteriological tests:		
Heterotrophic plate count (See Section 9215)	Monthly	< 1000 CFU/mL
Use test (see 4e)	Quarterly and for a new source	Student's $t \leq 2.78$

* Or more frequently if there is a problem.

13.4.4 Purchasing of Service and Supplies.

a. The laboratory will ensure that purchased supplies/reagents or consumables that affect the quality of tests are not used until they have been inspected or otherwise verified or complying with standard specifications or requirements defined in the methods concerned. Supplier certifications may be accepted after reliability of the suppliers has been established.

b. Please see SOP 4.1.4 for the complete procedure.

13.5.0 Laboratory Apparatus and Instruments

13.5.1 Incubators and Waterbaths

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a. If an automatic temperature recorder is not used, place a calibrated thermometer with a bulb immersed in liquid on a central shelf and record the temperature twice daily at least 4 hours apart, when the incubator or water bath is in use.

b. Drain and clean waterbath as required and refill with laboratory pure water.

13.5.2 Refrigerators and Freezers

a. Check and record temperature once daily at least 4 hours apart in the microbiology department and daily in the chemistry department. If the temperature is outside the specified range corrective action shall be taken. All corrective action reports will be kept in the Quality Assurance Manager's office. During use, the temperature charts will be stored in the appropriate section. All completed charts will be stored in the QA Manager's Office. The large walk-in cooler temperature charts will also be stored in the QA Manager's Office.

b. Clean periodically and discard outdated materials.

c. Do not store food for consumption in any laboratory refrigerator or freezer.

d. Please refer to NEL SOP #3.7.2 for further information.

13.5.3 Balances

a. Check with Class-S weights each day balance is used and record in the QC log. QA Manager or designee should be immediately notified of balances that do not meet specifications. A discrepancy report must be filled out and the problem investigated and resolved.

b. Clean and level balances as required.

c. Maintain annual maintenance services contract with QC Services or a similar qualified firm.

d. Please refer to NEL SOP #3.7.2 for further information.

13.5.4 pH Meters

a. Date all pH buffer solutions when opened.

b. Standardize meter with pH buffers before each use.

13.5.5 Thermometers

Check all laboratory thermometers annually with a reference National Bureau of Standards thermometer or one which meets the requirements of NIST Monograph 150. Mark any necessary corrections on each thermometer and record in the QC logbook.

13.5.6 Fumehood

a. Fumehood laminar flow shall be monitored each day the fume hood is used using a velometer and recorded.

b. Please refer to NEL SOP #3.7.2 for further information.

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13.5.7

13.6.0 Laboratory Waste Disposal Practices

13.6.1 Chemical waste can only be disposed of after it has been determined either hazardous or non-hazardous. Analysts should under no circumstance dispose of waste that may be hazardous. To determine whether or not a waste is hazardous it should be brought to the Hazardous Waste Manager.

13.6.2 Disposal of all laboratory chemical wastes must be done in a manner that maximizes personnel and environmental protection and minimizes the risk of adverse effects. All disposal practices must comply with appropriate safety, health, and environmental regulations. Every effort should be made to minimize the amount of chemical wastes requiring disposal.

13.6.3 Sterilize all contaminated microbiological glassware, material, and supplies before washing or disposal, or dispose of in an approved biohazard bag. The Maine DEP will dispose of waste for a fee.

13.6.4 Chemical wastes may be disposed of in the following ways: disposal through sanitary sewer system, and disposal at regulated treatment and disposal facility. Only the Hazardous Waste Manager should dispose of waste. The method of disposal is dependent upon:

- Nature of the waste, hazard potential of the waste
- Applicability of waste under the Resource Conservation and Recovery Act (RCRA) regulations.

13.6.5 The following discussion describes the analyst's role in the disposal of hazardous waste:

13.6.5.1 Wastes that are known to be hazardous should be properly labeled with the hazardous constituents and the date. The container should then be placed in the appropriate hazardous waste shed. The three sheds are designated as follows:

a. The shed on the left is for all flammable waste.

b. The shed on the right is for all radioactive waste. Only the Radiation Safety Officer should enter this shed.

c. The shed in the middle is for corrosive and all other types of hazardous waste.

13.6.5.2 Unknown wastes should be brought to the hazardous waste manager for designation.

13.6.5.3 Satellite accumulation areas can be found at most workstations. Analysts should place appropriate waste in such containers. When the container is filled it should be properly labeled with the date and hazardous constituents and placed in the appropriate waste shed.

13.6.6 A contract waste transporter will periodically pick up waste. All waste leaving this facility will be properly manifested and complete manifest records will be maintained on all waste disposed of in this manner. Waste will be transported only by a permitted transporter and disposed of at a permitted waste disposal facility.

13.7.0 Environmental Monitoring of Microbiology Areas

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Air settling plates will be evaluated weekly to monitor the environment throughout the micro lab. The acceptable limit is 15 CFU per 15 minute TSA with 5% Sheep's Blood plate. Air monitoring plates will be monitored on a weekly basis using an Anderson Air sampler and R2A with Cycloheximide and MEA with Streptomycin. The acceptable limit is less than or equal to 200 bacteria/m^3 or fungi/m^3. If the limit is exceeded, a discrepancy report will be initiated, the colonies identified to the genus level, and the area immediately cleaned with 70% isopropyl alcohol.

13.8.0 Cleanliness of Microbiology Areas

Walls, floors, ceilings and work surfaces shall be non-absorbent and easy to clean and disinfect. At the end of the day all work surfaces will be disinfected using 70% Isopropyl Alcohol. Surfaces will be disinfected throughout the day as needed.

13.9.0 Separation of Microbiological Areas

There shall be effective separation between neighboring areas in which there are incompatible testing being performed. Measures shall be taken to prevent cross-contamination (see 13.7.0 and 13.8.0).

13.10.0 In microbiological areas, the humidity levels will be monitored once every day.

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CHAPTER 14

DOCUMENTATION PROCEDURES

14.1.0 Introduction

This standard Laboratory Quality Assurance Program Procedure (LQAP) provides guidelines on documentation procedures to be used at the Laboratory.

14.2.0 Responsibility

The Laboratory Manager or appropriate analysts are responsible for training the staff in proper documentation procedures.

14.3.0 Corrective Action

Problems in documentation shall be referred to the Laboratory Manager and/or the QA Manager, who shall take the appropriate corrective action.

14.4.0 Documentation Procedures

The laboratory manager shall communicate project documentation requirements that exceed current laboratory policies to the project staff. All Laboratory staff who enter or review entries in the notebook shall print and sign their names and write their initials on the official signature record maintained by the QA Officer. Thereafter, initials may be used in place of signatures. A current list of signatures is attached to LQAP Chapter 2.

14.4.1 Laboratory Notebooks/Forms

14.4.1.1 Record all entries legibly in permanent, preferably black ink.

14.4.1.2 Enter information directly into the notebook or appropriate form as analysis is performed.

14.4.1.3 The Section Supervisor or designee shall frequently review entries and initial and date the bottom of each page of raw data.

14.4.1.4 Document all deviations from standard operating procedures (SOP's) and include a reason for such deviation.

14.4.1.5 Corrections/Additions: Draw a single line through the incorrect entry so that the original entry remains legible. Add the correct entry then explain, initial and date the correction. New information may be added to the original page if it is initialed and dated. Erasures/white-outs shall not be permitted. Reason for line out will be noted and circled using the numerical designation defined below:

1) Clerical Error
2) Data Error
3) Entry Location Error
4) Other (Describe)

Appropriate number will be entered and circled. Number 4 will require a brief description.

14.4.2 Equipment Printouts

14.4.2.1 Record legibly, in permanent ink, the following information on each printout or instrument run log:

* Sample Lab identification number
* Case identification (Project Code) if applicable

Initiated By: Total Quality Manager

Approved By: Pamela Daughtry
Total Quality Manager

Approved By: A Halaby
Chemistry Lab Manager

Approved By: Beth Yazdosh
Microbiology Manager

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* Date work was performed
* Other pertinent information (analyst, sample #, instrument params, etc.)

14.4.2.2 Sign or initial and date each printout or instrument run log.

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CHAPTER 15

TRAINING PROCEDURES

15.1.0 Introduction

It is the intent of this Laboratory Quality Assurance Procedure (LQAP) to illustrate how the Laboratory trains and keeps track of the training of new employees.

15.2.0 Guidance for Training

It is necessary that all new employees view a safety film provided by the laboratory manager or his/her designee. The new employees should be given a full tour of the facility so that they become aware of not only the hazards which could be incurred within their own section, but also those of surrounding sections and where to go for help.

15.3.0 Responsibility for Training

New employees shall not be allowed to operate independently without the supervision of their trainer until that trainer is satisfied with that employee's performance. Chemistry employees must successfully complete and document an initial demonstration of capabilities (IDC) before they are authorized to perform any analysis unassisted (see below). To ensure that the entire preparation, extraction, and analysis process is completed by a group of capable individuals, NEL ensures that each analyst, whether it be prep or analytical analysts, demonstrate capability in his/her area of responsibility in the sequence of analytical events. Each analyst is listed on the cover sheet and their responsibility is defined through the use of extraction, digestion and or analytical raw data sheets. The cover sheets not only reference the person (s) involved in the analytical procedure, but also reference which SOP the procedure is directed by and the correct revision letter for that SOP so that traceability is complete. For microbiology methodology training, refer to Chapter 2 of this document. Upon completion of the IDC and within the trainee's 90-day probationary period, the trainer will submit the trainee's record of accomplishment to the Laboratory Manager who will make the final decision as to that employee's ability to operate independently. If approved, the employee will be allowed to perform tasks without the assistance of their trainer and their training record will be put into their permanent training file. Upon completion of the annual MDL study, the MDL's will be included in the training records as continuing demonstration of proficiency (CDP). Thereafter, annual MDL's will be considered a continuing demonstration of proficiency and will also be kept in the employees training records. Method detection limits are calculated on an annual basis unless specified otherwise by a particular project. MDL's are calculated by the method defined by 40CFR Part 136, Appendix B. For reporting purposes, NEL uses its PQL or Practical Quantitation Limit, which is usually 2-5 times higher than the MDL.

The progress of training can be summarized as follows:

a. The trainee reads the SOP and the EPA or other official method
b. The trainee assists the trainer in the analysis
c. The trainer assists the trainee in the analysis
d. The trainee successfully completes an IDC

15.4.0 Initial Demonstration of Capabilities (IDC) - (chemical analyses only)

15.4.1 The IDC must be performed for every analysis/method the analyst hopes to perform. The IDC is usually performed after the analyst has assisted the trainer and the trainer feels that the trainee is

competent. To successfully complete the IDC the new employee must prepare and/or analyze four replicates of an outside/secondary quality control standard at a concentration approximately 10 times the Method Detection Limit (MDL). Using the four results, calculate the average percent recovery and the standard deviation (s) of the population sample (n-1). Compare the average percent recovery and the standard deviation to the corresponding acceptance criteria in the test method. If the average mean recovery and the standard deviation meet the acceptance criteria listed in the method for all parameters, the new employee can move onto analyzing samples. If they are outside of the acceptance criteria for any parameter, the test must be repeated. If the repeated test also fails, this indicates a problem with the system. The source of the problem must be identified and corrected before the test is repeated. There is a chart at the end of this LQAP where the trainer may keep track of his/her trainee's results for each IDC.

15.4.2 For those analytes that cannot be spiked, i.e. EPA Method 1311., the analyst will undergo a NELAC audit for that test. The NELAC Chemistry/Micro Checklist will be used. The analyst must demonstrate proficiency by successfully completing the audit. The analyst must receive a greater than 90% adherence to the method rating in order to be considered trained. Corrective actions will be initiated where necessary and resolved prior to signing off on the training form attached. All training materials will be signed by the appropriate individuals and stored in the analyst's training file.

15.4.3 For analytes that require a calculation (ie. hardness and organic nitrogen) the IDCs for the analytes which can be spiked (ie. calcium, magnesium, total kjeldahl nitrogen and ammonia nitrogen) will be used to verify that the analyst has the ability to use the correct calculation to obtain the correct result. Using the four calculated results, the average percent recovery and standard deviation(s) will be calculated. The average percent recovery and standard deviation will be compared to the acceptance criteria for the spiked analytes. If the average mean recovery and the standard deviation meet the acceptance criteria of the spiked analyte, the analyst will be considered trained. If they are outside the acceptance criteria, the test must be repeated.

15.5.0 Training in Specific Sections

Due to the diversity of tests being run within the different sections of the laboratory, it will be the responsibility of the QA Manager, the section leader or their designee to determine which check samples will be used for training in their sections. Upon completion of the check samples by the trainee, the trainer will review the results and pass them onto the QA Manager and to the Chemistry/Microbiology Laboratory Manager for final approval.

15.6.0 Final Approval

Within the trainee's 90-day probationary period the Laboratory Manager will review the results and will determine if the trainee is able to operate independently or not. A certification statement shall be signed by the Laboratory Manager and the QA Manager to complete each IDC. A copy of the certification statement will be retained in the employee's training records. The training record will then become a permanent piece of the employee's training file.

15.6.1 Continuing Demonstration of Capability

Analysts will be required to maintain their proficiency in a particular test by participating in external proficiencies evaluations or blind internal known at least once per year. If multiple analysts are trained, each analyst will need to participate in a yearly internal or external audit. Analysts will also be required to demonstrate continued proficiency through the performance of annual MDL studies. All continuing demonstration of capability data will be attached to the signed certification statement attached to this LQAP Chapter

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Trainee:_____

Trainer:_____

Analytical Procedure:_____

Method Number:_____

New Employee Training Chart
Initial Demonstration of Capabilities

Date Analyzed	Rep.#	Measured Value Please circle conc. (ug/L, mg/L) or (ug/Kg, mg/Kg)	True Value Please circle conc. (ug/L, mg/L) or (ug/Kg, mg/Kg)	% Recovery	(s) Std. Dev. (n-1)
	1				
	2				
	3				
	4				
	Mean		N/A		N/A

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Trainee:_____

Trainer:_____

Analytical Procedure:_____

Method Number:_____

Results of Quality Control Samples

QC Samples Analyzed	Date Analyzed	Measured Value Please circle conc. (ug/L, mg/L) or (ug/Kg, mg/Kg)	True/Sample Value Please circle conc. (ug/L, mg/L) or (ug/Kg, mg/Kg)	% Recovery
a. method reagent blank			N/A	N/A
b. blank spike				
c. sample duplicate				
d. QC checks: _____ _____	_____ _____	_____ _____	_____ _____	_____ _____
e. matrix spike				
f. matrix spike duplicate				

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Trainee:_____

Trainer:_____

Analytical Procedure:_____

Method Number:_____

Results of NELAC Audit for Non-Spiked Approach of Demonstration of Capability

Types of Audit Questions	Date of Audit	Demonstration of the Method	Method Documentation	% Recovery
Preparation Steps				
Analysis Steps				
Log Book Entry				
Good Lab Practices				
Quality Control				
Quality Assurance				

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Taken from:

NELAC, Quality Systems, Chapter 5, June 5, 2003

Initial Demonstration of Capability

Certification Statement

Date:
Laboratory Name: Northeast Laboratory Services, Inc.
Laboratory Address: P.O. Box 788 Waterville ME 04903-0788/227 China Rd., Winslow, Me 04901
Analyst(s) Name(s):

Prep Name(s):

Matrix: Aqueous
(examples: laboratory pure water, soil, air, solid, biological tissue)

Method number, SOP# and Analyte or Class of Analytes or Measured Parameters
(examples: barium by 200.7, trace metals by 6010, benzene by 8021, etc.)

We, the undersigned, CERTIFY that:

1. The analyst(s) identified above, using the cited test method, which is in use at this facility for the analyses of samples under the National Environmental Laboratory Accreditation Program, have met the Continuing Demonstration of Capability.

2. The test method was performed by the analyst(s) identified on this certification.

3. A copy of the test method and the laboratory-specific SOPs are available for all personnel on-site.

4. The data associated with the Continuing Demonstration of Capability are true, accurate, complete, and self-explanatory (1).

5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and that the associated information is well organized and available for review by authorized inspectors.

James F. Galasyn, Chemistry Laboratory Manager	_____	_____
Technical Director's Name and Title	Signature	Date
Pamela Doughty	_____	_____
Quality Assurance Officer's Name	Signature	Date

This certification form must be completed each time a demonstration of capability study is completed.

(1) True: Consistent with supporting data

Accurate: Based on good laboratory practices consistent with sound specific principles/practices.

Complete: Includes the results of all supporting performance testing.

Self-Explanatory: Data properly labeled and stored so that the results are clear and require no additional explanation.

Taken from:

UNCONTROLLED

NELAC, Quality Systems, Chapter 5, June 5, 2003

Continuing Demonstration of Capability

Certification Statement

Date:
Laboratory Name: Northeast Laboratory Services, Inc.
Laboratory Address: P.O. Box 788 Waterville ME 04903-0788/227 China Rd., Winslow, Me 04901
Analyst(s) Name(s):

Prep Name(s):

Matrix: Aqueous
(examples: laboratory pure water, soil, air, solid, biological tissue)

Method number, SOP# and Analyte or Class of Analytes or Measured Parameters
(examples: barium by 200.7, trace metals by 6010, benzene by 8021, etc.)

We, the undersigned, CERTIFY that:

6. The analyst(s) identified above, using the cited test method, which is in use at this facility for the analyses of samples under the National Environmental Laboratory Accreditation Program, have met the Continuing Demonstration of Capability.

7. The test method was performed by the analyst(s) identified on this certification.

8. A copy of the test method and the laboratory-specific SOPs are available for all personnel on-site.

9. The data associated with the Continuing Demonstration of Capability are true, accurate, complete, and self-explanatory (1).

10. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and that the associated information is well organized and available for review by authorized inspectors.

James F. Galasyn, Chemistry Laboratory Manager	_____	_____
Technical Director's Name and Title	Signature	Date
Pamela Doughty	_____	_____
Quality Assurance Officer's Name	Signature	Date

This certification form must be completed each time a demonstration of capability study is completed.

(2) True: Consistent with supporting data
Accurate: Based on good laboratory practices consistent with sound specific principles/practices.
Complete: Includes the results of all supporting performance testing.
Self-Explanatory: Data properly labeled and stored so that the results are clear and require no additional explanation.

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CHAPTER 16

Quantitation Limit, Curve Fitting, and SURROGATE REPORTING PROCEDURES

16.1.0 Introduction

It is the intent of this Laboratory Quality Assurance Procedure (LQAP) to illustrate how the Laboratory reports data on samples that require dilution to be accurately quantitated.

16.2.0 Guidance for Surrogate Recovery

Surrogates will be reported as "Diluted out of Range" for any dilution greater than 10 (ten) fold. This policy is based upon the inaccuracy of the quantitation at greater dilutions.

16.3.0 Reporting of Non-Detects on Diluted Samples
The Detection Limit or Practical Quantitation Limit of an analyte will be multiplied by the dilution factor necessary to achieve accurate quantitation on that sample.

16.4.0 Policy for Calibration Curves

To ensure the accuracy of analytical data multipoint curves shall be used. The number of calibration levels is specified in the analytical methods. Acceptable determination of linearity are typically RF's (response factors) of ± 20%, a linear regression or quadratic fit with a coefficient of 0.995 (inorganics) and 0.990 (organics). Instrument sensitivity will also be monitored. Second order curves require a minimum of 6 standards, excluding the zero point. Refer to US EPA, methods, other referenced methods and SOPs for appropriate guidance. The QA Manager or the Lab Manager must approve any exceptions to these criteria.

In instances where analytical time to establish the curve is prohibitive (e.g. GC/MS) the initial curve shall be verified by analyzing a standard from a second source at the midpoint of the curve. Should this midpoint curve check fail routine trouble shooting of the analytical system will be conducted. If this identifies a problem the standard will be re-analyzed. If trouble shooting does not identify a problem or re-analysis of the standard fails the second time, a new standard will be prepared. If this standard also fails further trouble shooting and/or a new initial calibration will be prepared. At a minimum, the initial calibration will be prepared annually when the MDL's are performed and following major maintenance. When deemed feasible, a standard will be analyzed at the reporting level and will meet the accuracy criteria of the method proof to reporting data from this sequence. Any deviations from this policy will be approved in writing by the Environmental Laboratory Manager and filed with the QC Batch Data.

Initiated By: Total Quality Manager

Approved By: Pamela Dougherty
Total Quality Manager

Approved By: A Galas
Chemistry Lab Manager

Approved By: B Yardrich
Microbiology Manager

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DOCUMENT CONTROL PROCEDURES

17.1.0 Introduction
The Laboratory has certain documents that contain information that employees must be familiar with, keep confidential, and may require periodic updating.

17.2.0 Controlled Documents will be numbered and stamped with the appropriate confidentiality stamp prior to distribution to the employees. The Laboratory QA Manager will establish a file to track these documents.

17.3.0 Controlled Documents will be issued with a memo requiring the signature of the employee to whom the Document is issued. This memo will detail that the employee has read and understood the Document and is aware of proper handling procedures for the Document.

17.4.0 All revisions of the Document will be issued with an interoffice memo (IM). The employee will return both documents after signing that they have read and understand the updated controlled document. The IM will contain the revision number in the case of the LAQP and the revision letter in the case of Standard Operating Procedures (SOPs). The QA Manager will file a copy of the signed interoffice memo and Table of Contents with the applicable documents highlighted, in each employee's training file as indication that they have read and understand the SOP and/or LQAP. The most current revision of any controlled documents will be the only ones available to laboratory personnel. The QA Manager will be responsible for filing old revisions, replacing old with new documents in all master books, and keeping the analytical SOP table of contents current so that there is no confusion as to which revision of any controlled document is the current one. Please see NEL SOP # 3.1.2 Procedure for Updating Controlled Documents for further information.

17.5.0 All controlled documents to include SOPs and the LQAP will be reviewed at least annually under the direction of QA. The most current controlled document will contain the most recent date and revision number or letter and will agree with the controlled copy of the chemistry SOP table of contents. The analyst or manager that is updating the SOP will ensure that they are comparing the SOP to the most recent revision of the method using either 40CFR or the FDA website. Any documents that need to be removed from master books for any reason will be done under the direction of QA and will be reflected as "removed" on the Table of Contents in the revision code column and the date of removal.

17.6.0 Upon termination of employment the employee will return all Controlled Documents to the Quality Assurance Manager and/or Laboratory Manager.

17.7.0 All external documents received at Northeast Laboratory Services will be considered controlled by the manufacturer or accreditor. QA will remove all external documents as they are replaced or reissued by the accreditor/manufacturer and will be stamped "obsolete" before being filed by QA.

17.8.0 NEL maintains a secure facility at all times. During normal business hours, all doors are secure from the outside except the front door. The sample log-in staff monitors the front door and sample receipt is managed through a receiving window. The sample receiving area is not in the laboratory and non-NEL personnel are required to sign in and wear a badge while being escorted by an NEL staff member.

17.9.0 Data confidentiality is maintained through employee training. Clients are only permitted to receive or view data that pertains to their samples only. Requests for data from other clients may be taken verbally only if the person requesting the data is known to the laboratory and permission has been given to the laboratory from the client who owns that data. All correspondence from NEL, i.e. email and fax, will contain the following confidentiality notice, "The information contained in this email and any attachments may be privileged, confidential, and/or proprietary and is intended solely for the use of the person(s) to whom it is addressed. If you are not the intended recipient, any review, retransmission, dissemination or any other use of the information contained in this email and any attachments is strictly prohibited. If you have received this communication in error, please notify the sender immediately by replying to this email and then delete this material from any system that it may be on. Viewing the attached report requires

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Adobe Acrobat Reader Version 4.0 or better. You can download the newest version free from http://www.adobe.com/products/acrobat/readstep2.html."

17.10.0 LIMS, instrument and processing software is required to be certified by the laboratory QA Manager in conjunction with the Laboratory Manager. Instrument software must be approved software by the vendor or other approved party as acceptable software for the applications used by NEL. All data related software requires the user to log-on to a PC with a specified password not known to any unauthorized personnel to ensure security of the software. PC's are maintained, including backup, by either automated processes or manual tape backup. The tapes are documented and stored in the server room.

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CHAPTER 18

QA REPORTS TO MANAGEMENT

18.1.0 There will be reports to management by the Analytical Division QA Manager department annually. The reports should include but are not limited to the following areas: Key QA Activities, Data Quality Assessments, Performance Evaluation Assessments, Corrective Actions and Status, External Audit Assessments, and Internal Audit Assessments as well as client feedback and proposed new directions for the laboratory. Quarterly, the QA Manager will provide reports to laboratory management regarding QA matters. These reports will include information on internal audits, proficiencies, performance over time and discrepancies initiated and closed during the previous quarter.

18.1.1 Key QA Activities will stress measures that are being taken to improve data quality; describe significant quality problems observed; report any changes in certification/accreditation status; describe involvement in resolution of quality issues with clients or agencies; report any QA organizational changes; and provide notice of the distribution of revised documents controlled by the QA Manager.

18.1.2 Data Quality Assessments includes the assessment of QC Data gathered over the period, the frequency of analyses repeated due to unacceptable QC performance, and the reason for the unacceptable performance.

18.1.3 Performance Evaluation Assessments summarizes the laboratory's performance on performance evaluation studies in which the laboratory participated, such as WS and WP studies. This is to include the results of any corrective actions required due to unacceptable performance test results.

18.1.4 Corrective/Preventive Actions and Status summarizes the major categories/reasons in which formal corrective actions were initiated and the status of the corrective actions.

18.1.5 External Audit Assessments summarizes the findings from audits or assessments of the Chemistry section conducted by an outside party. This includes the reason for the outside audit, any major deficiencies identified by the auditors and the corrective actions that have been or will be implemented to correct the deficiencies.

18.1.5 Internal Audit Assessments summarizes the results of any system or performance audits initiated by the QA/QC department. It includes the reason the audit was conducted, the results of the audit, and any corrective actions that were required due to deficiencies or unacceptable results found during the audits.

18.2.0 The report must specify the frequency schedule for the QA reports, the report recipient, and the report preparer.

18.3.0 The report to management should be followed up with a written response for management to the QA Manager. A Management Review Board Meeting will be held following review of the QA Report. Please refer to NEL SOP #3.1.6 for further information.

Initiated By: Total Quality Manager

Rev. 20.0 01/2008

Approved By: Pamela Dougherty
Total Quality Manager

Approved By: A Halasz
Chemistry Lab Manager

Approved By: D M Friedrich
Microbiology Manager

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Chapter 19
Glossary and Acronyms

CAR	Corrective Action Report
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CLP	Contract Laboratory Program. The program set up by the USEPA to regulate superfund (CERCLA) activities including sampling, analysis, and data validation.
DQO	Data Quality Objectives
GLP	Good Laboratory Practice
IDL	Instrument detection limit. The ability of an instrument to differentiate a signal generated from the analyte from the background noise.
LCS	Laboratory Control Sample. A blank matrix spiked with a known concentration of analyte used to determine system control. In some instances the laboratory may analyze a laboratory control sample duplicate (LCSD). The LCS may be purchased from and outside source.
LIMS	Laboratory Information Management System
MDL	Method Detection Limit. A calculated limit determined by analyzing at least 7 replicate samples of the same concentration (2-5 times the expected MDL). The standard deviation is determined and multiplied by the student T value for the number of samples analyzed. This method originates from CFR 40 Part 136 Appendix B.
MS	Matrix Spike Sample. A sample matrix spiked with a known concentration of analyte used to determine possible matrix interferences. In some instances the laboratory will analyze a matrix spike duplicated (MSD).
MD	Matrix duplicate. A sample duplicate.
NPDES	National Pollutant Discharge Elimination System
PQL	Practical Quantitation Limit. Usually a factor of approximately 3-5 times the laboratory MDL to better simulate real sample matrix since the MDL is determined in a deionized water matrix.
PARCC	Precision, Accuracy, Representativeness, Comparability and Completeness. Part of DQOs.
QA/QC	Quality Assurance/Quality Control
QAPP	Quality Assurance Project Plan
RCRA	Resource Conservation and Recovery Act
SOP	Standard Operating Procedure
TSCA	Toxic Substances and Control Act
USEPA	United States Environmental Protection Agency

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