N60478.AR.000885
NWS EARLE
5090.3a

Sampling and Analysis Plan

for

Site 23 Groundwater Sampling

Naval Weapons Station Earle
Colts Neck, New Jersey

Naval Facilities Engineering Command
Mid-Atlantic

Contract Number N62470-08-D-1001
Contract Task Order WE15

July 2010

TETRA TECH

SAP Worksheet No. 1 -- Title and Approval Page
(UFP-QAPP Manual Section 2.1)

FINAL

SAMPLING AND ANALYSIS PLAN
(Field Sampling Plan and Quality Assurance Project Plan)
July 2010

Site 23 Groundwater Sampling
NWS Earle
Colts Neck, New Jersey

Prepared for:
Naval Facilities Engineering Command Mid-Atlantic

Prepared by:
Tetra Tech NUS, Inc.
234 Mall Boulevard, Suite 260
King of Prussia, PA 19406
610-491-9688

Prepared under:
N62470-08-D-1001
CTO WE15

Review Signatures:

Kelly J. Cooper for 7/19/10
Tom Johnston, Ph.D. /CLEAN QAM/Date
Tetra Tech

Mary M. Mang 7/19/10
Mary M. Mang/Project Manager/Date
Tetra Tech

Approval Signatures:

Roberto Pagtalunan 7/26/10
Roberto Pagtalunan/RPM/Date
NAVFAC BRAC PMO, NE

'No Comment' Letter to Follow
Jessica Mollin/RPM/Date
EPA Region 2

'No Comment' Letter to Follow
Erica Bergman /PM/Date
NJDEP

EXECUTIVE SUMMARY

Tetra Tech NUS, Inc. (Tetra Tech) has prepared this Sampling and Analysis Plan (SAP) for Groundwater Sampling for Site 23 at Naval Weapons Station (NWS) Earle, Colts Neck, New Jersey (**Figure 10-2**). The contaminants of concern (COCs) associated with Site 23 groundwater are metals, principally arsenic, beryllium, cadmium, chromium, and iron.

Site 23, the Paint Disposal Area, was used from the early 1970s until approximately 1993 for the disposal of paint wastes generated from repainting and stenciling large ordnance. The site consists of approximately 200 square feet of ground surface west of the northwest corner of the former Building D-5. Paint waste disposal to the ground surface reportedly occurred in the past.

This SAP contains the technical scope of work and associated sampling plan to perform groundwater level measurements and groundwater sampling at Site 23. The purpose of the work is to reevaluate the presence of metals contamination in site groundwater that was reported in the 1995 Remedial Investigation. The RI found metals in groundwater at levels above regulatory guidelines including aluminum, arsenic, beryllium, cadmium, chromium, iron, manganese, lead, and thallium. However, the lowest sampling endpoint turbidity value of 457 NTU, indicates that the unfiltered groundwater sample results may not be representative of metals concentrations in the formation. No organic compounds were found at concentrations above regulatory guidelines in groundwater.

A Record of Decision (ROD) for Site 23 was signed in 1999 based on the results of the RI. The selected remedy addressed soil contamination at Site 23 by instituting land use controls (LUCs) and five-year reviews. The 2008 Five Year Review for Site 23 and a review of the RI groundwater analytical data, upon which the ROD was based, revealed high turbidity levels in several of the samples, which call into question the validity of the reported metals concentrations. Representative groundwater data (i.e., with low turbidity) are needed to accurately assess the levels of TAL Metals concentrations in site groundwater and compare them to current NJDEP GWQS criteria.

SAP Worksheets

SAP Worksheet No. 1 -- Title and Approval Page 1
SAP Worksheet No. 2 -- SAP Identifying Information 6
SAP Worksheet No. 3 -- Distribution List 9
SAP Worksheet No. 4 -- Project Personnel Sign-Off Sheet 10
SAP Worksheet No. 5 -- Project Organizational Chart 11
SAP Worksheet No. 6 -- Communication Pathways 12
SAP Worksheet No. 7 -- Personnel Responsibilities and Qualifications Table 13
SAP Worksheet No. 8 -- Special Personnel Training Requirements Table 19
SAP Worksheet No. 9 -- Project Scoping Session Participants Sheet 20
SAP Worksheet No. 10 -- Problem Definition 22
SAP Worksheet No. 11 -- Project Quality Objectives/Systematic Planning Process Statements 26
SAP Worksheet No. 12 -- Measurement Performance Criteria Table 28
SAP Worksheet No. 13 -- Secondary Data Criteria and Limitations Table 29
SAP Worksheet No. 14 -- Summary of Project Tasks 30
SAP Worksheet No. 15 -- Reference Limits and Evaluation Table 36
SAP Worksheet No. 16 -- Project Schedule / Timeline Table 37
SAP Worksheet No. 17 -- Sampling Design and Rationale 38
SAP Worksheet No. 18 -- Sampling Locations and Methods/SOP Requirements Table 39
SAP Worksheet No. 19 -- Analytical SOP Requirements Table 40
SAP Worksheet No. 20 -- Field Quality Control Sample Summary Table 41
SAP Worksheet No. 21 -- Project Sampling SOP References Table 42
SAP Worksheet No. 22 -- Field Equipment Calibration, Maintenance, Testing, and Inspection Table 43
SAP Worksheet No. 23 -- Analytical SOP References Table 44
SAP Worksheet No. 24 -- Analytical Instrument Calibration Table 45
SAP Worksheet No. 25 -- Analytical Instrument & Equipment Maintenance, Testing, & Inspection Table 47
SAP Worksheet No. 26 -- Sample Handling System 48
SAP Worksheet No. 27 -- Sample Custody Requirements Table 49
SAP Worksheet No. 28 -- Laboratory QC Samples Table 51
SAP Worksheet No. 29 -- Project Documents and Records Table 54
SAP Worksheet No. 30 -- Analytical Services Table 55
SAP Worksheet No. 31 -- Planned Project Assessments Table 56
SAP Worksheet No. 32 -- Assessment Findings and Corrective Action Responses 57
SAP Worksheet No. 33 -- QA Management Reports Table 58
SAP Worksheet No. 34 -- Verification (Step I) Process Table 59
SAP Worksheet No. 35 -- Validation (Steps IIa and IIb) Process Table 60
SAP Worksheet No. 36 -- Analytical Validation (Steps IIa and IIb) Summary Table 63
SAP Worksheet No. 37 -- Usability Assessment 64

REFERENCES

APPENDICES

Appendix A - Tetra Tech Standard Operating Procedures
Appendix B - Laboratory Standard Operating Procedures
Appendix C - Figures

Acronyms

°C	Degrees Celsius
%D	Percent Difference
%R	Percent Recovery
ACT-POC	Activity Point of Contact
ARAR	Applicable or Relevant and Appropriate Requirements
bgs	Below Ground Surface
CCB	Continuing Calibration Blank
CCV	Continuing Calibration Verification
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act of 1980
CFR	Code of Federal Regulations
CLEAN	Comprehensive Long-term Environmental Action Navy
CLP	Contract Laboratory Program
COC	Contaminant of Concern
CTO	Contract Task Order
CVAA	Cold Vapor Atomic Absorption
DO	Dissolved Oxygen
DoD ELAP	Department of Defense Environmental Laboratory Accreditation Program
DoD QSM	Department of Defense Quality Systems Manual
DQI	Data Quality Indicator
DQO	Data Quality Objective
EPA	Environmental Protection Agency
FOL	Field Operations Leader
FSP	Field Sampling Plan
ft/day	Feet per Day
FTMR	Field Task Modification Request
GIS	Geographical Information System
GWQS	Groundwater Quality Standard
HASP	Health and Safety Plan
HAZWOPER	Hazardous Waste Operations and Emergency Response
HDPE	High Density Polyethylene
HNO_3	Nitric Acid
HSM	Health and Safety Manager
IAS	Initial Assessment Study
ICB	Initial Calibration Blank
ICP	Inductively Couple Plasma
ICP-AES	Inductively Couple Plasma-Atomic Emission Spectroscopy
ICP-MS	Inductively Couple Plasma-Mass Spectroscopy
ICS	Interference Check Sample
ICV	Initial Calibration Verification
IDL	Instrument Detection Limit
IDQTF	Interagency Data Quality Task Force
IDW	Investigation-Derived Waste
IS	Internal Standard
LCS	Laboratory Control Sample
LUC	Land Use Control
MCL	Maximum Contaminant Level
MDL	Method Detection Limit
mL	Milliliter
MPC	Measurement Performance Criteria
MS	Matrix Spike
MSA	Method of Standard Addition
MSD	Matrix Spike Duplicate
MSL	Mean Sea Level
mV	Millivolt

Acronyms
(Continued)

N.J.A.C	New Jersey Administrative Code
NA	Not Applicable
NAVFAC	Naval Facilities Engineering Command
NELAP	National Environmental Laboratory Accreditation Program
NJDEP	New Jersey Department of Environmental Protection
NR	Nor Recorded
NTU	Nephelometric Turbidity Unit
NWS	Naval Weapons Station
ORP	Oxidation Reduction Potential
OSHA	Occupational Safety and Health Administration
OU	Operating Unit
PAH	Polynuclear Aromatic Hydrocarbons
PDF	Portable Document Format
PM	Project Manager
PQOs	Project Quality Objectives
PSL	Project Screening Level
QA	Quality Assurance
QAM	Quality Assurance Manager
QAO	Quality Assurance Officer
QAPP	Quality Assurance Project Plan
QC	Quality Control
QL	Quantitation Limit
RI	Remedial Investigation
ROD	Record of Decision
RPD	Relative Percent Difference
RPM	Remedial Project Manager
RSD	Relative Standard Deviation
SAP	Sampling and Analysis Plan
SDG	Sample Delivery Group
SI	Site Investigation
SMCL	Secondary Maximum Contaminant Level
SOP	Standard Operating Procedure
SQL	Structured Query Language
SSO	Site Safety Officer
TAL	Target Analyte List
TBC	To Be Considered
TOM	Task Order Manager
Tetra Tech	Tetra Tech NUS, Inc.
UFP-QAPP	Uniform Federal Policy for Quality Assurance Project Plans
µg/L	Micrograms per Liter
UTL	Upper Tolerance Limit

SAP Worksheet No. 2 -- SAP Identifying Information
(UFP-QAPP Manual Section 2.2.4)

Site Name/Number: Site 23 – Paint Disposal Area
Operable Unit: OU 4
Contractor Name: Tetra Tech NUS, Inc.
Contract Number: N62470-08-D-1001
Contract Title: Comprehensive Long-Term Environmental Action Navy (CLEAN)
Work Assignment Number: Contract Task Order (CTO) WE15

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

2. Identify regulatory program: Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA)

3. This Sampling and Analysis Plan (SAP) is a project-specific SAP.

4. List dates of scoping sessions that were held:

Data Quality Objectives (DQOs) Scoping Session	Date
Project Scoping Meeting – Tetra Tech only	3/19/09
Project Scoping Meeting - Tetra Tech and Navy	5/29/09

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

Title	Date
Remedial Investigation (RI) Report for (NWS) Earle	July 1996
Removal Actions at Sites 22, 23 and 27 NWS Earle	February 1997
Record of Decision (ROD) Operable Unit 4 (OU-4) Sites 14, 20, 22, 23, 24, 25, 27, and 29 - NWS Earle	August 1999

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

The United States Environmental Protection Agency, Region 2 (EPA) (regulatory oversight), NJDEP (regulatory oversight), U.S. Navy (property owner), Naval Facilities Engineering Command (NAVFAC) (Environmental Restoration Program supervision), Tetra Tech (Navy contractor)

7. Lead organization

U.S. Navy (property owner), NAVFAC (Environmental Restoration Program supervision)

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

Not Applicable– all required SAP elements are addressed in this document.

UFP-QAPP Worksheet No.	Required Information	Crosswalk to Related Information
A. Project Management		
Documentation		
1	Title and Approval Page	Not Applicable
2	Table of Contents SAP Identifying Information	Not Applicable
3	Distribution List	Not Applicable
4	Project Personnel Sign-Off Sheet	Not Applicable
Project Organization		
5	Project Organizational Chart	Not Applicable
6	Communication Pathways	Not Applicable
7	Personnel Responsibilities and Qualifications Table	Not Applicable
8	Special Personnel Training Requirements Table	Not Applicable
Project Planning/ Problem Definition		
9	Project Planning Session Documentation (including Data Needs tables) Project Scoping Session Participants Sheet	Not Applicable
10	Problem Definition, Site History, and Background. Site Maps (historical and present)	Not Applicable
11	Site-Specific Project Quality Objectives	Not Applicable
12	Measurement Performance Criteria Table	Not Applicable
13	Sources of Secondary Data and Information Secondary Data Criteria and Limitations Table	Not Applicable
14	Summary of Project Tasks	Not Applicable
15	Reference Limits and Evaluation Table	Not Applicable
16	Project Schedule/Timeline Table	Not Applicable
B. Measurement Data Acquisition		
Sampling Tasks		
17	Sampling Design and Rationale	Not Applicable
18	Sampling Locations and Methods/ SOP Requirements Table/ Sample Location Map(s)	Not Applicable
19	Analytical Methods/SOP Requirements Table	Not Applicable
20	Field Quality Control Sample Summary Table	Not Applicable
21	Project Sampling SOP References Table Sampling SOPs	Not Applicable
22	Field Equipment Calibration, Maintenance, Testing, and Inspection Table	Not Applicable
Analytical Tasks		
23	Analytical SOPs Analytical SOP References Table	Not Applicable
24	Analytical Instrument Calibration Table	Not Applicable
25	Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table	Not Applicable
Sample Collection		
26	Sample Handling System, Documentation Collection, Tracking, Archiving and Disposal Sample Handling Flow Diagram	Not Applicable
27	Sample Custody Requirements, Procedures/SOPs Sample Container Identification Example Chain-of-Custody Form and Seal	Not Applicable

UFP-QAPP Worksheet No.	Required Information	Crosswalk to Related Information
Quality Control Samples		
28	QC Samples Table Screening/Confirmatory Analysis Decision Tree	Not Applicable
Data Management Tasks		
29	Project Documents and Records Table	Not Applicable
30	Analytical Services Table Analytical and Data Management SOPs	Not Applicable
C. Assessment Oversight		
31	Planned Project Assessments Table Audit Checklists	Not Applicable
32	Assessment Findings and Corrective Action Responses Table	Not Applicable
33	QA Management Reports Table	Not Applicable
D. Data Review		
34	Verification (Step I) Process Table	Not Applicable
35	Validation (Steps IIa and IIb) Process Table	Not Applicable
36	Validation (Steps IIa and IIb) Summary Table	Not Applicable
37	Usability Assessment	Not Applicable

SAP Worksheet No. 3 -- Distribution List
(UFP-QAPP Manual Section 2.3.1)

Name of SAP Recipients	Title/Role	Organization	Telephone Number	E-mail Address or Mailing Address	Document Control Number
Roberto Pagtalunan	Remedial Project Manager (RPM)	NAVFAC Mid-Atlantic	757-444-0808	roberto.pagtalunan@navy.mil	Not Applicable (NA)
Eric Helms	Environmental Director Activity Point of Contact (ACT-POC)	NWS Earle	732-866-2540	eric.helms@navy.mil	NA
Bonnie Capito	Administrative Record/Librarian	NAVFAC Atlantic	(757) 322-4785	bonnie.capito@navy.mil	NA
Jessica Mollin	EPA RPM	EPA Region 2	212-637-3921	Mollin.Jessica@epamail.epa.gov	NA
Erica Bergman	NJDEP Project Manager	NJDEP	609-292-7406	erica.bergman@dep.state.nj.us	NA
Mary Mang	Project Manager (PM)	Tetra Tech	610-491-9688	mary.mang@tetratech.com	NA
Joseph Tomalavage	Field Operations Leader (FOL)/Site Safety Officer (SSO)	Tetra Tech	610-491-9688	joe.tomalavage@tetratech.com	NA
Tom Johnston	Quality Assurance Manager (QAM)	Tetra Tech	412-921-8615	tom.johnston@tetratech.com	NA
Megan Ritchie	Project Chemist	Tetra Tech	610-382-1527	megan.ritchie@tetratech.com	NA
Kate Zaleski	Laboratory PM	Katahdin Analytical Services (Katahdin)	207-874-2400	acolby@katahdinlab.com	NA

SAP Worksheet No. 4 -- Project Personnel Sign-Off Sheet

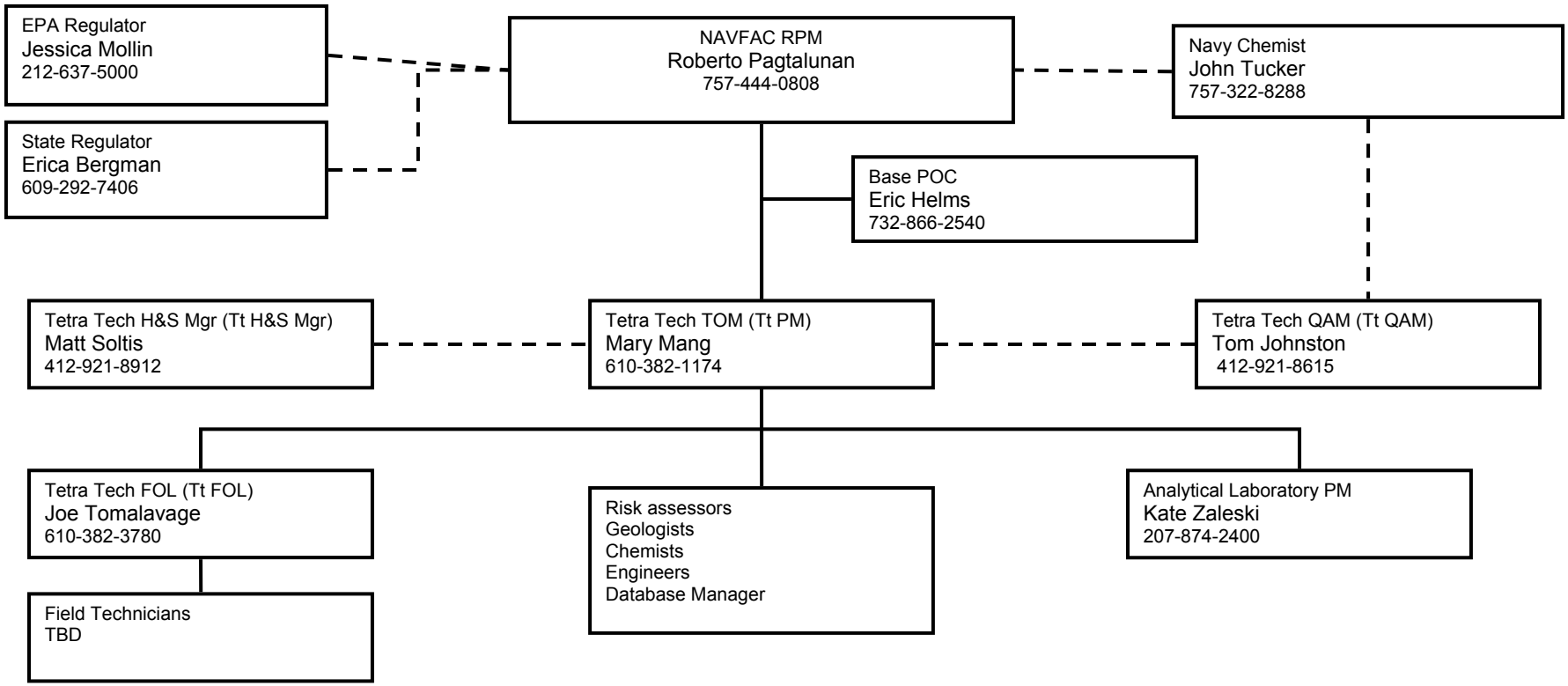
(UFP-QAPP Manual Section 2.3.2)

Key personnel will be instructed to read the SAP prior to attending an internal site-specific kick-off meeting for field activities. The Tetra Tech PM will track when the reviews have been completed, obtain signatures, and insure that the completed sign-off sheet is included in the central project file.

Name	Organization/Title/Role	Telephone Number	Signature/email receipt	SAP Section Reviewed	Date SAP Read
Jessica Mollin	EPA RPM	212-637-3921	See **Worksheet No. 1** for signature	All	
Erica Bergman	NJDEP PM	609-292-7406	See **Worksheet No. 1** for signature	All	
Roberto Pagtalunan	NAVFAC RPM	757-444-0808	See **Worksheet No. 1** for signature	All	
Mary Mang	Tetra Tech PM	610-491-9688	See **Worksheet No. 1** for signature	All	
Tom Johnston	Tetra Tech QAM	412-921-8615	See **Worksheet No. 1** for signature	All	
Joseph Tomalavage	Tetra Tech FOL/SSO	610-491-9688		All	
Megan Ritchie	Tetra Tech Project Chemist	610-491-1527		All	
Lee Leck	Database Manager	412-921-8856		Worksheet Nos. 12, 14, 15, 19, 20, 23-28, 30, 34-37	
Kate Zaleski	Laboratory PM	207-874-2400		Worksheet Nos. 12, 14, 15, 19, 20, 23-28, 30, 34-37	

SAP Worksheet No. 5 -- Project Organizational Chart
(UFP-QAPP Manual Section 2.4.1)

Lines of Authority ————————
Lines of Communication ·····



SAP Worksheet No. 6 -- Communication Pathways
(UFP-QAPP Manual Section 2.4.2)

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or e-mail	Procedure (timing, pathway to & from, etc.)
Field Task Modification Request (FTMR)	Tetra Tech FOL/SSO	Joseph Tomalavage	610-382-3780	Gets approval from Tetra Tech PM within one working day; Document via FTMR form.
SAP Amendments	Tetra Tech PM Navy RPM	Mary Mang Roberto Pagtalunan	610-382-1174 757-444-0808	After realizing an amendment is needed, PM will send NAVFAC a concurrence letter within 7 days. The NAVFAC RPM will sign the letter within 5 days of receipt.
Changes in Schedule	Tetra Tech PM	Mary Mang	610-382-1174	When impact is realized, send NAVFAC a schedule concurrence letter within 7 days or prior to the first affected deliverable date.
Field issues that require changes in scope or implementation of field work	Tetra Tech FOL/SSO Tetra Tech PM	Joseph Tomalavage Mary Mang	610-382-3780 610-382-1174	FOL informs PM the day the issue is realized. PM informs the NAVFAC RPM of the issue within 1 day of the FOL's notification. PM also sends a concurrence letter to the NAVFAC within 7 days, if project scope is affected. The NAVFAC RPM will sign the letter within 5 days of receipt. Document changes on a FTMR form.
Stop work recommendations, for example, to protect workers from unsafe conditions/situations or to prevent a degradation in quality of work	Tetra Tech PM Tetra Tech FOL/SSO Tetra Tech QAM Tetra Tech Health and Safety Manager (HSM) Navy RPM	Mary Mang Joseph Tomalavage Tom Johnston Matt Soltis Roberto Pagtalunan	610-382-1174 610-382-3780 412-921-8615 412-921-8912 757-444-0808	On the day the problem is identified, FOL, SSO, or QAM informs PM and documents problem on a FTMR. On the same day of the notification from the FOL, PM informs the NAVFAC and Installation RPMs.
Analytical data quality issues	Katahdin PM Tetra Tech Project Chemist	Kate Zaleski Megan Ritchie	207-874-2400 610-382-1527	When issue is related to chemical data, Laboratory PM notifies Project Chemist within 2 days of identification of problem. Project Chemist notifies Tetra Tech PM/ within 1 day.

SAP Worksheet No. 7 -- Personnel Responsibilities and Qualifications Table
(UFP-QAPP Manual Section 2.4.3)

Name	Title/Role	Organizational Affiliation	Responsibilities	Education and/or Experience Qualifications
Mary Mang, CHMM	PM	Tetra Tech	Oversees project, financial, schedule, and technical day-to-day management of the project. • Ensures timely resolution of project-related technical, quality, and safety questions associated with Tetra Tech operations. • Functions as the primary Tetra Tech interface with the Navy RPM, NWS Earle, Tetra Tech field and office personnel, and laboratory points of contact. • Ensures that Tetra Tech health and safety issues related to this project are communicated effectively to all personnel and off-site laboratories. • Monitors and evaluates all Tetra Tech subcontractor performance. • Coordinates and oversees work performed by Tetra Tech field and office technical staff (including data validation, data interpretation, and report preparation). • Coordinates and oversees maintenance of all Tetra Tech project records. • Coordinates and oversees review of Tetra Tech project deliverables. • Prepares and issues final Tetra Tech deliverables to the Navy.	M.S.E. Civil Engineering B.S. Pomology 25 years environmental experience
Joseph Tomalavage	FOL/SSO	Tetra Tech	Supervises, coordinates, and performs field sampling activities. Specific responsibilities include: • Functions as the on-site communications link between field staff members, subcontractors, and Tetra Tech PM.	B.S. Geology, 35 years environmental experience

Name	Title/Role	Organizational Affiliation	Responsibilities	Education and/or Experience Qualifications
			• Oversees the mobilization and demobilization of all field equipment and subcontractors. • Coordinates and manages the field technical staff. • Adheres to the work schedules provided by the Tetra Tech PM. • Ensures the proper maintenance of site logbooks, field logbooks, and field recordkeeping. • Initiates FTMRs (field change orders) when necessary. • Identifies and resolves problems in the field, implements and documents corrective action (CA) procedures, and provides communication between the field team and project management. Alerts off-site analytical laboratories of any special health and safety hazards associated with environmental samples. • Ensures that all health and safety requirements for the investigation are implemented. • Alerts off-site analytical laboratories of any special health and safety hazards associated with environmental samples. • FOL responsibilities include initiating and conducting equipment inventories to ensure equipment is available, purchasing equipment as required, staging equipment for efficient loading and transport from the Tetra Tech office to the site, and, after field activities are completed, demobilizing the equipment. The SSO will be responsible for training and monitoring site conditions. Details of these responsibilities are presented in the HASP and include:	

Name	Title/Role	Organizational Affiliation	Responsibilities	Education and/or Experience Qualifications
			• Controlling specific health and safety-related field operations such as personnel decontamination, monitoring of worker heat or cold stress, and distribution of safety equipment. • Conducting and documenting a daily health and safety briefing each day while on site. • Assuring that field personnel comply with all procedures established in the HASP. • Identifying SSOs in their absence. • Terminating work in an imminent safety hazard, emergency situation, or other potentially dangerous situation is encountered. • Assuring the availability and the condition of health and safety monitoring equipment. • Coordinating with the FOL and Tetra Tech PM to institute and document any necessary HASP modifications. • Ensuring that facility personnel and subcontractors are adequately advised and kept clear of potentially contaminated materials.	

Name	Title/Role	Organizational Affiliation	Responsibilities	Education and/or Experience Qualifications
Tom Johnston, PhD	QAM	Tetra Tech	Reviews SAP, oversees preparation of lab scope, coordinates with lab, and data quality review. Ensures Quality aspects of the CLEAN program. • Develops, maintains, and monitors Quality Assurance and Quality Control (QA/QC) policies and procedures. • Provides training to Tetra Tech staff in QA/QC policies and procedures. • Conducts systems and performance audits to monitor compliance with environmental regulations, contractual requirements, SAP requirements, and corporate policies and procedures. • Audits project records. • Monitors subcontractor quality controls and records. • Assists in the development of corrective action plans and ensuring correction of non-conformances reported in internal or external audits. • Ensures that this SAP meets Tetra Tech, Navy, and NJDEP requirements. • Oversees the responsibilities of the Tetra Tech Project QA/QC Advisor. • Prepares QA reports for management.	PhD Analytical Chemistry, 30 years environmental experience

Name	Title/Role	Organizational Affiliation	Responsibilities	Education and/or Experience Qualifications
Megan Ritchie	Project Chemist	Tetra Tech	Coordinates analyses with laboratory chemists, ensures the scope is followed, that QA has been performed for data packages, and communicates with Tetra Tech staff. • Ensures that the project meets objectives from the standpoint of laboratory performance. • Provides technical advice to the Tetra Tech team on matters of project chemistry. • Monitors and evaluates subcontractor laboratory performance. • Ensures timely resolution of laboratory-related technical, quality, or other issues effecting project goals. • Functions as the primary interface with the subcontracted laboratory and the Tetra Tech PM. • Coordinates and oversees work performed by the subcontracted laboratory. • Oversees the completion of Tetra Tech data validation. • Coordinates and oversees review of laboratory deliverables. • Recommends appropriate laboratory corrective actions.	B.S. Biology and Environmental Studies, 12 years environmental experience.
Matt Soltis	HSM	Tetra Tech	Oversees CLEAN Program Health and Safety Program • Provides technical advice to the Tetra Tech PM on matters of health and safety. • Oversees the development and review of the Health and Safety Plan (HASP). • Conducts health and safety audits. • Prepares health and safety reports for management.	B.S. Industrial Safety Sciences, 24 years environmental experience

Name	Title/Role	Organizational Affiliation	Responsibilities	Education and/or Experience Qualifications
Kate Zaleski	PM	Katahdin	Coordinates analyses with laboratory chemists, ensures the scope is followed, reviews QA data packages, and communicates with Tetra Tech staff.	Can be provided upon request

In some cases, one person may be designated responsibilities for more than one position. For example, the FOL may also be responsible for SSO duties. This action will be performed only as credentials, experience, and availability permits.

SAP Worksheet No. 8 -- Special Personnel Training Requirements Table

(UFP-QAPP Manual Section 2.4.4)

No special training requirements are necessary.

SAP Worksheet No. 9 -- Project Scoping Session Participants Sheet
(UFP-QAPP Manual Section 2.5.1)

Project Name: Site 23 Groundwater Sampling Projected Date(s) of Sampling: 06/15/2009 – 06/17/2009 Project Manager: Mary Mang	Site Name: Site 23 Site Location: Colts Neck, New Jersey

Date of Session: 3/19/2009
Scoping Session Purpose: Scoping Meeting

Name	Title	Affiliation	Phone No.	E-mail Address	Project Role
Mary Mang	PM	Tetra Tech	610-491-9688	mary.mang@tetratech.com	Management
Roberto Pagtalunan	Navy RPM	Navy	757-444-0808	roberto.pagtalunan@navy.mil	Management
Joe Tomalavage	FOL/SSO	Tetra Tech	610-491-9688	joe.tomalavage@tetratech.com	Management
Mark Traxler	Project Chemist	Tetra Tech	610-382-1171	mark.traxler@tetratech.com	Chemist
Erica Bergman	NJDEP PM	NJDEP	609-292-7406	erica.bergman@dep.state.nj.us	Regulatory
Jessica Mollin	EPA RPM	EPA	212-637-3921	Mollin.Jessica@epamail.epa.gov	Regulatory

Comments/Decisions: Defined general sampling approach, number of samples, sample analyses.

Action Items: Start Worksheets and text for SAP.

Consensus Decisions: Background concentrations will be determined by sampling the up gradient well at Site 23 (MW23-02) and two wells designated as background wells for the Kirkwood Formation during the RI: BGGW01 and BGGW02. Samples will be collected for the full list of 23 Target Analyte List (TAL) Metals. Filtered samples will be collected if turbidity less than 10 Nephelometric Turbidity Units (NTUs) is not achieved. Sample results from the site monitoring wells will be compared to background well sample results to determine if the observed metals concentrations are site related.

SAP Worksheet No. 9 con't.

Project Name: Site 23 Groundwater Sampling Projected Date(s) of Sampling: August 2009 Project Manager: Mary Mang	Site Name: Site 23 Site Location: Colts Neck, New Jersey

Date of Session: 5/29/2009
Scoping Session Purpose: Scoping Meeting

Name	Title	Affiliation	Phone No.	E-mail Address	Project Role
Mary Mang	PM	Tetra Tech	610-491-9688	mary.mang@tetratech.com	Management
Roberto Pagtalunan	Navy RPM	Navy	757-444-0808	roberto.pagtalunan@navy.mil	Management
Ken Bowers	Chemist	Navy	757-322-8341	ken.bowers@navy.mil	QA Chemist
Joe Tomalavage	FOL/SSO	Tetra Tech	610-491-9688	joe.tomalavage@tetratech.com	Management
Megan Ritchie	Project Chemist	Tetra Tech	610-382-1527	megan.ritchie@tetratech.com	Chemist

Comments/Decisions: Discussed Project Quality Objectives (PQOs) including analyte list and screening criteria. Discussed study questions and decision statements.

Action Items: Tetra Tech will procure laboratory services and complete SAP worksheets.

Consensus Decisions: Analyte list will be the TAL Metals without the nutrients including calcium, magnesium, potassium, and sodium. Study question is whether there is contamination in Site 23 groundwater. Decision statement is if metals concentrations in Site 23 groundwater exceed screening criteria of NJDEP GWQS or MCL (for barium only), then the project team will discuss an approach for groundwater; if not, then continue to implement land use controls based on the soil remedy).

SAP Worksheet No. 10 -- Problem Definition
(UFP-QAPP Manual Section 2.5.2)

10.1 SITE LOCATION

NWS Earle is located in Monmouth County, New Jersey, approximately 47 miles south of New York City. The station consists of two areas, the 10,248-acre Main Base (Mainside Area), located inland, and the 706-acre Waterfront Area (**Figure 10-1**). The two areas are connected by a Navy-controlled right-of-way. The Mainside area is located approximately 10 miles inland from the Atlantic Ocean in Colts Neck, Howell and Wall Townships, and Tinton Falls Borough. Site 23 is located in the central portion of the Mainside Area (Figure 10-2).

10.2 SITE HISTORY

NWS Earle was commissioned in 1943 and its primary mission is to supply ammunition to the Atlantic fleet. Site 23, the Paint Disposal Area, was used from the early 1970s until approximately 1993 for disposal of paint wastes from the repainting and stenciling of torpedoes, aerial bombs, and other large ordnance. The site consists of approximately 200 square feet of ground surface west of the northwest corner of the former location of Building D-5. Paint waste disposal to the ground surface reportedly occurred in the past. As a result, the Navy conducted a focused remedial action to address several specific areas of soil contamination in December 1996. Approximately 86 tons of contaminated material was excavated and disposed off site. The site is currently cleared of all structures.

10.3 PHYSICAL SETTING

Land Use

The Mainside area consists of a large, undeveloped portion associated with ordnance operations, production, and storage; this portion is encumbered by explosive safety quantity distance arcs. Other land use in the Mainside area consists of residences, offices, workshops, warehouses, recreational space, open space, and undeveloped land. Site 23 is currently unused.

Surface Features

Site 23 is bordered by a drainage ditch on the west, a railroad spur to the northeast, and an 8- to 10-foot-high berm to the west and south (**Figure 10-3**). No drainage swales or streams are located on the site.

Geology of Site 23

Regional geologic mapping indicates that Site 23 is within the outcrop area of the Kirkwood Formation, upper colluvium. The Vincentown formation underlies the Kirkwood. The upper colluvium has a maximum thickness of 10 feet, the Kirkwood Formation ranges between 60 and 100 feet in thickness. Soil borings completed during previous investigations are no more than 27 feet deep. The lithology of the soils encountered in the on-site borings generally agrees with the published description of the upper colluvium, Kirkwood and Vincentown Formations. In general, the borings encountered alternating beds of yellowish-brown, fine- to coarse-grained sand (upper colluvium), olive brown fine to medium-grained sand (Kirkwood), and greenish-gray, glauconitic fine to medium-grained sand (likely representative of the Vincentown). Based on the monitoring wells drilling logs, the wells are screened across the Kirkwood and Vincentown formations.

Hydrogeology

Groundwater in the aquifer beneath Site 23 occurs under unconfined conditions at depths of approximately 2 feet below ground surface (bgs). The direction of shallow groundwater flow in the aquifer is toward the northeast. There does not appear to be a significant seasonal variation in groundwater flow direction. The hydraulic conductivities calculated at monitoring wells MW23-01 and MW23-02 are 7.91 feet per day (ft/day) and 5.78 ft/day, respectively.

10.4 PREVIOUS INVESTIGATIONS

Initial Assessment Study (IAS)

The IAS, completed in 1983, consisted of personnel interviews and a visual inspection of the site. The IAS concluded that a bare area of approximately 200 square feet had been used for paint disposal to surface soil. The site was not recommended for confirmation study because it was believed that the amount of paint dumped was not enough to pose a significant environmental or public health threat.

Site Investigation (SI)

During the 1993 SI, sampling results indicated that elevated levels of metals (mainly chromium and lead) at concentrations sometimes above regulatory guidelines were found in soil and sediments. Elevated levels of metals (mainly arsenic, beryllium, chromium, and lead) were found in the one groundwater sample collected by Hydropunch®.

Phase I Remedial Investigation (RI)

RI activities were conducted at Site 23 in 1995 and consisted of installation of three shallow monitoring wells (MW23-01, MW23-02, and MW23-03); slug testing to determine local aquifer permeability; and measuring of static groundwater levels to determine groundwater flow direction. Sampling and analysis of subsurface soils, groundwater, surface water, and sediments were conducted. The RI human health risk assessment concluded that Site 23 posed an unacceptable risk to human health.

The NJDEP Residential and Non-Residential Soil Direct Contact standards were not exceeded in the results from the subsurface soil samples.

Lead and chromium exceeded the sediment ecological toxicity threshold values at one sediment sampling location. Polycyclic Aromatic Hydrocarbons (PAHs) were also detected above Applicable or Relevant and Appropriate Requirements (ARARs) and To Be Considered criteria (TBC) at one sediment location.

Aluminum, arsenic, beryllium, cadmium, chromium, lead, iron, manganese, and vanadium were detected in one or more of the site monitoring wells in concentrations that exceeded the NJDEP GWQS. The metal concentration exceedances may be due in part to turbidity. When the analytical results for filtered metals are compared to total metals, the only metals that exceed the NJDEP GWQS are aluminum, arsenic, cadmium, iron, and manganese.

One surface water sample exceeded NJDEP Fresh Water Ecological Screening Criteria for barium, lead, and vanadium.

In order to compare site-related metals concentrations found in groundwater during RI sampling to facility-wide naturally occurring (background) groundwater concentrations, it was necessary to choose facility monitoring wells deemed to have been installed in "background" locations upgradient of the RI sites. Wells or well locations, representative of background or non-impacted conditions, were selected for each interpreted aquifer underlying NWS Earle by agreement among the Navy, EPA, and NJDEP. The interpreted aquifer underlying Site 23 is in the Kirkwood/Vincentown Formations. Three wells were selected as background wells for the Kirkwood/Vincentown Formations: monitoring wells MW5-02, MW5-03, and MW19-01. These three wells were combined with background wells screened in the Cohansey Sand and Kirkwood formation (for a total of 11 wells) in order to perform a statistical evaluation of background groundwater metals data for comparison with the analytical results obtained from the sampling activities at Site 23.

These three formations were combined based on similar lithology and stratigraphic proximity. Upper tolerance limits (UTLs) for the 95th percentile were calculated for background concentrations and compared to the individual maximum site related concentrations for corresponding wells grouped in the same interpreted aquifer. The background results for inorganics (metals) are shown in the following table. As with the Site 23 groundwater metals concentrations, turbidity may have affected the reported background metals concentrations. The Site 23 analytical results for filtered metals indicate the only

metals that exceed the NJDEP GWQS are aluminum, arsenic, cadmium, iron, and manganese in well MW23-02.

TABLE 10-1
OCCURRENCE AND DISTRIBUTION OF RI INORGANIC CONCENTRATIONS FOR SITE-RELATED AND BACKGROUND GROUNDWATER FOR THE COHANSEY SAND, KIRKWOOD, AND VINCENTOWN FORMATIONS

Parameter	Aquifer Background Mean (µg/L)	Background 95% UTL (µg/L)	Up Gradient Well (MW23-02) (µg/L)	Site Wells (MW23-01 and MW23-03) (µg/L)	NJDEP GWQS (µg/L)
Aluminum	1,560	13,500	9,500	25,600 - 41,700	200
Arsenic	1.85	3.79	8.4	27.7 - 40.4	3
Beryllium	0.246	1.11	1	3.7 - 6.5	1
Cadmium	0.604	1.73	3	3.4 - 15	4
Chromium	14.7	46.4	486	1,210 - 2,380	70
Iron	2,100	6,670	25,800	60,100 - 108,000	300
Lead	1.03	2.97	19.8	45.3 - 50.1	5
Manganese	17	91.4	59.3	18.6 - 48.3	50
Vanadium	8.24	32.1	211	612 - 1,140	60

µg/L – micrograms per liter

Removal Action

The Navy elected to prepare a work plan and EE/CA for the removal and disposal of contaminated soil at Site 23. A focused removal action was initiated by the Navy in December 1996. Approximately 86 tons of contaminated material was removed and disposed off-site. Clean off-site fill material was subsequently placed in the excavated areas. Confirmation soil sampling results indicated that contaminant levels were generally below regulatory cleanup levels when compared with NJDEP Residential Direct Contact, Non-Residential Direct Contact, and Impact to Groundwater soil cleanup criteria. Thallium was the only contaminant which exceeded any of the soil cleanup criteria (Residential Direct Contact and Non-Residential Direct Contact). None of the confirmation soil samples exceeded the impact to groundwater soil cleanup criteria. No remedial activities were performed for groundwater or sediments.

ROD

A ROD for OU-4, including Site 23, was signed September 1999. Institutional controls (in the form of land use restrictions placed in the NWS Earle Master Plan) were the selected remedy for Site 23.

As outlined in the ROD none of the confirmation soil sample results exceeded the Impact to Groundwater criteria. The selected remedy presented in the 1998 OU-4 ROD addressed soil contamination. Institutional controls, in the form of a notation on the NWS Earle Master Plan for Site 23, were implemented to restrict future residential use of the site. The Navy and the regulators concluded that no further action for Site 23 groundwater, beyond institutional controls was necessary.

Five-Year Reviews

Site 23 was not included in the first Five Year Review required under CERCLA. No activities or additional remedial actions have been performed since 1998 when the Navy prepared an addendum for Site 23 in the NWS Earle Master Plan.

A site inspection and document and analytical data reviews were conducted at Site 23 during the second Five Year Review in 2008. The review resulted in the recommendation to sample the Site 23 monitoring wells and analyze the groundwater samples for TAL metals.

10.5 SITE CONCEPTUAL MODEL

The objective of the removal action was to eliminate soils impacted by former paint disposal operations in order to minimize potential migration of contaminants in soils to surface water via run-off and to groundwater via infiltration. The geologic formation associated with Site 23 is predominantly unconsolidated fine to medium-grained sand, which allows for dispersion and attenuation of suspended and dissolved metals.

Analytical results from the soil confirmation samples collected during the removal action indicated that contaminant levels were generally less than NJDEP soil cleanup criteria. Thallium was the only contaminant which exceeded any of the soil cleanup criteria (Residential Direct Contact and Non-Residential Direct Contact).

Groundwater results from the RI indicated that several unfiltered metals concentrations exceeded the New Jersey GWQS including aluminum, arsenic, beryllium, cadmium, chromium, iron, lead, manganese, and vanadium. Based on the RI risk assessment, the principal chemicals of concern (COCs) for the site groundwater are arsenic, cadmium, chromium, and iron. In addition, several metals exceeded background UTLs (see Table 10-1).

The RI human health risk assessment yielded unacceptable cancer risks (greater than 1E-04) and non-cancer risks (HI > 1) for the future industrial receptor for groundwater ingestion. Unacceptable cancer risks were determined for the future lifetime resident for groundwater ingestion and dermal contact. Unacceptable non-cancer risks were determined for the future child resident for groundwater ingestion and dermal contact. The IEUBK lead model yielded unacceptable risk (greater than 5% above 10 ug/dL) for the future residential child.

Currently there is no activity at, or use of, Site 23. Anticipated exposure scenarios include the future residential and industrial receptor scenarios; however, the Navy has no plans to change the current Site status. Humans engaged in activities associated with future exposure scenarios potentially may come into contact with groundwater through ingestion or dermal adsorption. Currently, there is no human exposure to groundwater at the site because there is no access to groundwater at Site 23 other than the monitoring wells.

Ecological receptor use of the site is minimal. The habitat is limited to a small bermed area surrounded by a chain link fence. No threatened or endangered species are present on or around the site.

The ecological risk assessment, conducted during the RI, determined that potential risks to ecological receptors from contaminants detected in surface water and sediment samples taken as part of the 1995 RI were relatively low. Since potential risks to ecological receptors at Site 23 appear to be low and off-site contaminant migration is minimal, further study or remediation based on ecological risk concerns at the site is unnecessary.

10.6 PROBLEM DEFINITION

The 2008 Five Year Review for NWS Earle included a review of the RI groundwater analytical data (upon which the ROD was based) which indicated high turbidity levels in several of the samples and calling into question the validity of the reported metals concentrations. Several metals concentrations exceeded background UTLs. Representative site-related and background groundwater data (i.e., with low turbidity) are needed to accurately assess the metals concentrations in site groundwater. Based on the Five-Year Review, additional investigation of groundwater at Site 23 is required.

The environmental question being asked:

- Are metals present in Site 23 groundwater at concentrations greater than regional background or NJDEP GWQS?

SAP Worksheet No. 11 -- Project Quality Objectives/Systematic Planning Process Statements
(UFP-QAPP Manual Section 2.6.1)

11.1 STUDY GOALS

Determine whether TAL Metals are present in the site groundwater at concentrations exceeding the regulatory criteria. If so, then the project team (Navy, EPA, and NJDEP) will determine if further investigation or monitoring of groundwater are required. If TAL metals concentrations in groundwater do not exceed regulatory criteria, then no further investigation or monitoring of groundwater is required.

11.2 INPUTS REQUIRED TO MAKE THE DECISION

Data and information that will be required to make this decision include the following:

1. Field Parameters
   Field measurements of pH, specific conductivity, turbidity, dissolved oxygen (DO), temperature, and redox potential will be collected as part of the low-flow purging and sampling procedure to ensure the representativeness of the groundwater that is collected during this sampling event.

2. Chemical Data
   Concentrations of total TAL Metals (and dissolved TAL Metals if turbidity is greater than 10 NTU at any location) in the Site 23 monitoring wells and from facility background wells will be determined. The TAL Metals list will exclude cyanide and the nutrients calcium, magnesium, potassium, and sodium.

3. Project Screening Levels

   Metals concentrations in the Site 23 monitoring wells will be compared to the most stringent of the criteria below, as well as maximum background groundwater concentrations.

   - Groundwater Quality Standards (GWQS)
     N.J.A.C. 7:9C, July 2008
     http://www.state.nj.us/dep/wms//bwqsa/docs/njac79C.pdf or

   - National Primary Drinking Water Maximum Contaminant Levels (MCLs)
     CFR Title 40 Part 141, July 2002
     http://www.epa.gov/safewater/contaminants/index.html#primary

11.3 DELINEATION OF STUDY BOUNDARY

The boundary is delineated by the existing site monitoring wells 23MW01 and 23MW03 (see **Figure 10-3**). Monitoring well 23GW02 is located just outside the western fence line and is hydraulically upgradient of site wells 23GW01 and 23GW03; it will be included in the background data set. The depths of the site wells range from 24 feet to 27 feet bgs.

11.4 DEFINITION OF RULES FOR DECISION MAKING

If any TAL Metal concentration in a site-related groundwater sample exceeds the PSL and background UTL, then the project team (Navy, EPA, and NJDEP) will determine if further investigation or monitoring are required. If no TAL Metal concentration in a site-related groundwater sample exceeds the PSL, then the Navy recommends no further monitoring of groundwater.

If any TAL Metal background UTL exceeds the PSL, then the project team will determine the appropriate action for Site 23 groundwater. If no TAL Metal background UTL exceeds the PSL, then the Navy recommends no further investigation of background groundwater.

Statistical calculations will not be used to compare site-related concentrations to the PSLs. Simple comparisons of every analyte concentration from each sample will be compared to the corresponding PSL and background UTL. A 95% UTL will be calculated for each analyte in background using the historical background data combined with the new background data.

11.5 PERFORMANCE CRITERIA

Because this groundwater investigation includes sampling of existing site monitoring wells, probability limits for false positive and false negative decision errors were not established. Simple comparisons of measured concentrations to PSLs and background concentrations are being used. In order to limit uncertainty in the field and laboratory data, performance criteria for field collection and laboratory analysis will be measured. Performance criteria are described in Worksheet Nos. 12, 15, and 28.

11.6 PLANS FOR OBTAINING THE DATA

Groundwater from existing site monitoring wells 23GW01, 23GW02, and 23GW03 and background groundwater from existing wells BGGW01 and BGGW02 and will be collected and analyzed for TAL metals. Monitoring well 23GW02 is hydraulically upgradient of site wells 23GW01 and 23GW03 and it will be included in the background data set. Site maps and figures detailing the site location, boundaries, and sampling locations are included in Appendix C. The sampling design and rationale is described in more detail in Worksheet No. 17.

SAP Worksheet No. 12 -- Measurement Performance Criteria Table

(UFP-QAPP Manual Section 2.6.2)

Measurement Performance Criteria Table - Field QC Samples

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria (MPC)	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Field Blank	TAL Metals	One per source water	Accuracy / Bias Contamination	No target compounds ≥Method Detection Limit (MDL)	S & A
Equipment Rinsate Blank	TAL Metals	One per 20 samples per sampling equipment[1]	Accuracy / Bias Contamination	No target compounds ≥MDL	S & A
Filtered Rinsate Blank	TAL Metals	One per filter brand[2]	Accuracy / Bias Contamination	No target compounds ≥MDL	S & A
Matrix Spike (MS)	TAL Metals	One per Sample Delivery Group (SDG), or every 20 samples	Accuracy / Bias	75 - 125 percent recovery (%R)	A
Matrix Spike Duplicate (MSD) (may not be required if laboratory duplicate is used)	TAL Metals	One per SDG, or every 20 samples	Accuracy / Bias Precision	75 - 125 %R; Relative Percent Difference (RPD) of ≤20%	A
Laboratory Duplicate (may not be required if an MSD is used)	TAL Metals	One per SDG, or every 20 samples	Precision	RPD of ≤20%	A
Field Duplicate	TAL Metals	One per 10 field samples	Precision	Values >5X the MDL: RPD of ≤20% Values <5X MDL: absolute difference of values must be <MDL	S & A
Temperature Blank	TAL Metals	One per cooler	Representativeness	Temperature between 2 and 6 degrees Celsius (4 ± 2 °C)	S

1 - Rinsate blanks will be collected if non-dedicated submersible pumps are used.
2 - A filtered rinsate blank be collected, if filtered samples are collected.

SAP Worksheet No. 13 -- Secondary Data Criteria and Limitations Table
(UFP-QAPP Manual Section 2.7)

Secondary Data	Data Source (originating organization, report title and date)	Data Generator(s) (originating organization, data types, data generation / collection dates)	How Data Will Be Used	Limitations on Data Use
Remedial Investigation (RI) Analytical Data	RI Report for Naval Weapons Station Earle Colts Neck, New Jersey	Brown and Root Environmental, February 1996	Historical data may be compared to current concentrations to determine if the RI data accurately reflect site conditions. A 95% UTL will be calculated for each target analyte in background using the historical background data combined with the new background data.	Historical site-related data will not be used to make the decision, only current site-related groundwater data will be used.

SAP Worksheet No. 14 -- Summary of Project Tasks
(UFP-QAPP Manual Section 2.8.1)

14.1 FIELD INVESTIGATION TASK PLAN

The field activities include mobilization and demobilization, monitoring well development, and monitoring well sampling. The following sections provide additional details regarding these subtasks. Investigation-Derived Waste (IDW) management is described in Section 14.6.

14.1.1 Mobilization/Demobilization

Mobilization shall consist of the delivery of all equipment, materials, and supplies to the site, the complete assembly in satisfactory working order of all such equipment at the site, and the satisfactory storage at the site of all such materials and supplies. Site-specific Health and Safety Training to all Tetra Tech field subcontractors will be provided as part of the site mobilization.

Demobilization shall consist of the prompt and timely removal of all equipment, materials, and supplies from the site following completion of the work. Demobilization includes the cleanup and removal of waste generated during the conduct of the investigation (i.e., IDW).

14.1.2 Well Development

Prior to the measurement of groundwater levels and the collection of groundwater samples, the Site 23 monitoring wells and the facility background monitoring wells to be sampled along with the Site 23 wells, will be redeveloped. Redevelopment will consist of pumping the wells to rapidly draw down the water level then allowing the wells to recover. The alternate pumping and recovery will continue until the evidence of sediment in the discharge water is minimal and turbidity is minimized. The procedures for well development are located in Tetra Tech SOP GH-2.8 Section 5.4 in Appendix A.

14.1.3 Groundwater Level Measurement

Prior to the collection of groundwater samples, one round of water level measurements will be collected from all of the site wells to be sampled. All measurements will be collected within an 8-hour period of consistent weather conditions to minimize atmospheric or precipitation effects on groundwater levels. The water levels will also be obtained a minimum of 12 to 24 hours after a significant rainfall event in order to negate the effects of short-term fluctuations in hydraulic head. Water level measurement procedures are discussed in Tetra Tech SOP GH-1.2, included in Appendix A.

14.1.4 Monitoring Well Sampling

The three site monitoring wells (23GW01 through 23GW03) and two off-site background monitoring wells (BGGW01 and BGGW02) will be purged and sampled using low-flow sampling techniques in accordance with the NJDEP Field Sampling Procedures Manual. The monitoring wells will be purged and sampled using a peristaltic pump or a submersible pump, depending on the depth to water. In each well, the tubing or pump intake will be positioned at the middle of the submerged length of well screen. The pumping rate will be set at between 0.1 to 0.4 liters/minute. The purge water discharge will be monitored for pH, specific conductivity, turbidity, redox potential, and DO. When the levels of these parameters stabilize, and a minimum of two saturated screen volumes have been removed from the well, the purging is complete and sampling can begin. Parameter stabilization is defined as three successive readings (taken at least 5 minutes apart) within 0.1 standard units for pH, 3% for conductivity, 10% for turbidity and DO, and 10 millivolts (mV) for redox potential. If possible, the stabilized turbidity reading should be less than 10 NTU. A filtered sample will be obtained using a new, disposable in-line 0.45 micron (pore size) filter for dissolved metals analysis, in addition to the sample for total metals analysis. The pumping rate will be controlled and the water level at each well will be constantly monitored. The vertical location of the tubing or pump intake, the required minimum purge volume, and the calculations for parameter stabilization will be recorded on the groundwater sample log sheet. The NJDEP SOP for low-flow sampling will be followed for purging and sampling of monitoring wells. In the event that the NJDEP

procedure does not provide guidance for a specific sampling issue, then the procedures outlined in Tetra Tech SOP SA-1.1 will be followed. The field SOPs are included in Appendix A.

The locations of the site monitoring wells to be sampled are shown in **Figure 14-1**. The locations of the two off-site background wells to be sampled are shown in **Figure 14-2**. The sampling and analysis program is outlined in Worksheet No. 18, and the sampling requirements for each type of analyses (i.e., sample containers, preservation, holding time) are listed in Worksheet No. 19. Field and laboratory QC samples will also be collected as identified in Worksheet No. 20.

14.2 SAMPLE DESIGNATION SYSTEM

Each sample collected for analysis will be assigned a unique sample tracking number. This number will consist of a two-segment alphanumeric code that identifies the site, the sample type (sample medium or QC sample designation), and the sample location. Tetra Tech SOP CT-04 addresses sample nomenclature (Appendix A). The alphanumeric coding system to be used is as follows:

Site Identifier:

23 = Site 23
BG = Background Location

Sample Type:

GW = Groundwater

QA/QC Sample Designation:

FB = Field Blank
RB = Equipment Rinsate Blank

Sample ID:

The sample ID will consist of the monitoring well number followed by eight-digits indicating the date on which the sample was collected. For example, a sample collected from 23GW01 on July 1, 2009 will be assigned the sample designation 23GW01-20090701. If the sample is filtered, the suffix "-F" will be appended to the sample ID (ex. 23GW01-20090701-F).

QC Sample Number:

All QC samples will be assigned a sample number based on the sample date. For example, the rinsate blank collected on July 1, 2009 will be assigned the tracking number RB-20090701.

Duplicate samples will be submitted to the laboratory as blind duplicates. Therefore, duplicate codes will be reflective of the standard sample matrix code followed by a "DUP" tag and sequentially listed. Due to the blind nature of the duplicate samples, no sample depth or date will be listed with the duplicated sample. An example of a duplicate sample would be "23DUP-01". The sampling time recorded on the Chain-of-Custody form, labels, and tags for the field duplicate samples will be 0000. 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).

Samples to be used for matrix spikes and matrix spike duplicates will be labeled MS/MSD on the bottle label and noted on the chain-of-custody, as required in the laboratory QA Plan; however, "MS/MSD" will not be part of the unique sample identifier in order to maintain consistency with the project database. Additional information regarding protocol for sample labeling is contained in Tetra Tech SOP SA-6.3.

All pertinent information regarding sample identification will be recorded in the field logbooks and on sample log sheets where appropriate.

14.3 EQUIPMENT DECONTAMINATION PROCEDURE

Decontamination of non-dedicated sampling equipment (e.g., submersible pump) will be conducted prior to and between sampling at each location. The following sequence will be implemented:

- Detergent wash (Alconox)
- Tap water rinse
- Distilled water rinse
- Air dry

14.4 FIELD DOCUMENTATION PROCEDURES

Field documentation will be performed in accordance with SOP SA-6.3 presented in Appendix A.

A summary of all field activities will be properly recorded in a bound logbook with consecutively numbered pages that cannot be removed. Logbooks will be assigned to field personnel and will be stored in a secured area when not in use.

At a minimum, the following information will be recorded in the site logbook:

- Name of the person to whom the logbook is assigned.
- Project name.
- Project start date.
- Names and responsibilities of on-site project personnel including subcontractor personnel.
- Arrival/departure of site visitors.
- Arrival/departure of equipment.
- Sampling activities and sample log sheet references.
- Description of subcontractor activities.
- Sample pick-up information including chain-of-custody numbers, air bill numbers, carrier, time, and date.
- Description of borehole or monitoring well installation activities and operations.
- Health and safety issues.
- Description of photographs including date, time, photographer, roll and picture number, location, and compass direction of photograph.

All entries will be written in ink and no erasures will be made. If an incorrect entry is made, striking a single line through the incorrect information will make the correction; the person making the correction will initial and date the change.

14.6 INVESTIGATION-DERIVED WASTE MANAGEMENT

It is anticipated that the following waste materials will be generated during the field investigation:

- Decontamination fluid
- Used personal protective equipment
- Used sampling equipment
- Well development water
- Well purge water

Decontamination fluid, well development water, and well purge water will be containerized and disposed at the NWS Earle wastewater treatment plant. Used PPE and sampling equipment will be bagged and sealed prior to its disposal as general refuse.

14.7 ANALYTICAL TASKS

Chemical analysis of TAL Metals will be performed by Katahdin Analytical Services. Katahdin is currently a DoD Environmental Laboratory Approval Program (ELAP) approved laboratory. DoD ELAP accreditation documentation is included in Appendix B. Analyses will be performed in accordance with the analytical methods identified in Worksheet No. 30. Katahdin will meet the PALs specified in Worksheet No. 15, with the exceptions noted, and will perform the chemical analyses following laboratory-specific SOPs (Worksheet Nos. 19 and 23) that were developed based on the methods listed in Worksheet Nos. 19 and 30. Copies of Katahdin SOPs that will be utilized on this project are included in Appendix B.

14.8 DATA HANDLING AND MANAGEMENT

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

14.8.1 Data Tracking and Control

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

The labels will be reviewed for adherence to work plan 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.

When field sampling is underway, the FOL forwards the chain-of-custody forms to the PM or designee and the laboratory for each day that samples are collected. The PM or designee will confirm that the chain-of-custody forms provide the information required by the work plan.

This will allow for early detection of errors made in the field so that adjustments can be made while the field team is mobilized. After successful completion of all requested analyses, the laboratory will submit an electronic deliverable for every sample delivery group (SDG). When all electronic deliverables have been received from the laboratory, the PM or designee 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.

14.8.2 Sample Information

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

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

14.8.3 Project Data Compilation

The analytical laboratory will generate a PDF file of the analytical data packages, as well as electronic database deliverables. The electronic database will be checked against the PDF file provided by the laboratory and updated as required, based on data qualifier flags applied during the data validation process. The data generated during the implementation of the SAP will be incorporated into the NWS Earle database and Geographical Information System (GIS). All data, such as units of measure and chemical nomenclature, will be manipulated to maintain consistency with the project database.

14.8.4 Geographical Information System

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

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

14.8.4 Data Management

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

Project Documentation and Records

A summary of project documentation and records to be generated and stored in the project files is provided in Worksheet No. 29.

Data Package Deliverables

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

The laboratory will provide Contract Laboratory Program (CLP) equivalent data packages for all analyses. Additionally electronic deliverables, formatted according to the requirements stated in the laboratory subcontracts, will be provided by the laboratories for all analytical data. Worksheet No. 30 summarizes the analytical requirements.

Data Reporting Formats

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

Data Review

The internal data verification requirements for this project include the maintenance and periodic review of field documentation (i.e., site logbooks, instrument calibration logs, chain-of-custody forms, field summary reports, and field modification records) and laboratory analytical data packages. After receipt of analytical laboratory results, Tetra Tech will perform data validation according to the requirements outlined in the method, EPA Region 2 SOP HW-2 Rev. 13, Evaluation of Metals Data for the CLP Program (September 2006), and Tetra Tech SOP DV-04 to ensure that the analytical results meet the project quality objectives.

After the data are validated, a list of nonconformities will be generated. Nonconformities require data qualifiers, which are used to alert the data user to inaccurate or imprecise data. For situations in which several quality control criteria are out of specification with regard to the limits specified in the DoD QSM, the data validator may make professional judgments and/or comments on the validity of the overall data package. In situations where the validity of an entire data package is in question, it may be necessary for

the sample(s) to be reanalyzed. The reviewer will then prepare a technical memorandum presenting changes in the data, if necessary, and the rationale for making such changes.

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

Data validation reports for all parameters will be generated according to the procedures described in Tetra Tech SOP DV-04. 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.

SAP Worksheet No. 15 -- Reference Limits and Evaluation Table
(UFP-QAPP Manual Section 2.8.1)

Media: Groundwater
Analyte Group: TAL Metals

Analyte	CAS Number	PSL	PSL Reference [1,2,3]	Project QL Goal [4]	Laboratory QLs	Laboratory MDLs
SW-846 6010		**(µg/L)**		**(µg/L)**	**(µg/L)**	**(µg/L)**
Aluminum	7429-90-5	200	NJDEP GWQS	67	300 [6]	13 [6]
Barium	7440-39-3	2,000 (6,000)	MCL (NJDEP GWQS)	670	5	0.35
Chromium	7440-47-3	70	NJDEP GWQS	23	15	0.44
Cobalt	7440-48-4	100	NJDEP Int. GWQS	33	30	0.27
Copper	7440-50-8	1300	NJDEP GWQS	430	25	0.7
Iron	7439-89-6	300	NJDEP GWQS	100	100	5.2
Manganese	7439-96-5	50	NJDEP GWQS	17	5	0.67
Selenium	7782-49-2	40	NJDEP GWQS	13	10	3
Zinc	7440-66-6	2,000	NJDEP GWQS	670	25	1.7
SW-846 6020						
Antimony	7440-36-0	6	NJDEP GWQS	2	1	0.14
Arsenic	7440-38-2	3	NJDEP GWQS	1	5 [6]	1.5 [6]
Beryllium	7440-41-7	1	NJDEP GWQS	0.33	1 [5]	0.06
Cadmium	7440-43-9	4	NJDEP GWQS	1.3	1	0.045
Lead	7439-92-1	5	NJDEP GWQS	1.7	1	0.04
Nickel	7440-02-0	100	NJDEP GWQS	33	1	0.13
Silver	7440-22-4	40	NJDEP GWQS	13	1	0.054
Thallium	7440-28-0	0.5	NJDEP GWQS	0.17	2 [6]	0.11 [6]
Vanadium	7440-62-2	60	NJDEP GWQS	20	5	0.69
SW-846 7470						
Mercury	7439-97-6	2	NJDEP GWQS	0.67	0.2	0.021

[1] NJDEP GWQS – Groundwater Quality Standards (NJDEP, 2008) http://www.state.nj.us/dep/wms//bwqsa/docs/njac79C.pdf
[2] NJDEP Interim GWQS – Interim Groundwater Quality Standards (NJDEP, 2008) http://www.state.nj.us/dep/wms/bwqsa/gwqs_interim_criteria_table.htm
[3] National Primary Drinking Water Maximum Contaminant Levels (MCLs) (EPA, 2009) http://www.epa.gov/safewater/consumer/pdf/mcl.pdf
[4] Project Quantitation Limit Goals are determined as at least 3X lower than the PSL, if achievable.
[5] The laboratory QL does not meet the Project QL Goal; however, the QL is sufficiently low to meet the PSL.
[6] The laboratory QL does not meet the PSL; however, the MDL, is sufficiently low to meet the Project QL Goal, or at a minimum the PSL.

SAP Worksheet No. 16 -- Project Schedule / Timeline Table

(UFP-QAPP Manual Section 2.8.2)

Activities	Organization	Dates (MM/DD/YYYY)		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
Well Redevelopment	Tetra Tech	8/16/2010	8/20/2010	NA	NA
Groundwater Sampling and Groundwater Level Measurements	Tetra Tech	9/6/2010	9/10/2010	Site 23 Groundwater Sampling Report	11/15/2010 (draft) 12/15/2010 (final)

SAP Worksheet No. 17 -- Sampling Design and Rationale

(UFP-QAPP Manual Section 3.1.1)

Groundwater from existing site monitoring wells 23GW01, 23GW02, and 23GW03 and background groundwater from existing wells BGGW01 and BGGW02 and will be collected and analyzed for TAL metals. Monitoring well 23GW02 is hydraulically upgradient of site wells 23GW01 and 23GW03 and it will be included in the background data set. All samples will be analyzed for total TAL Metals (excluding calcium, magnesium, potassium, and sodium). Groundwater samples will be collected using a peristaltic pump or submersible pump and low-flow sampling techniques (see SOP SA-1.1 and NJDEP Sec. 6.9.2.2). If the turbidity is measured in the field to be greater than 10 NTU at any location, filtered samples will also be collected and analyzed for dissolved TAL Metals. Site maps and figures detailing the site location, boundaries, and sampling locations are included in Appendix C.

SAP Worksheet No. 18 -- Sampling Locations and Methods/SOP Requirements Table
(UFP-QAPP Manual Section 3.1.1)

Sampling Location / ID Number	Matrix	Depth (units)	Analytical Group	Number of Samples (identify field duplicates)	Sampling SOP Reference
23GW01-YYYYMMDD	Groundwater	NA	Total TAL metals (dissolved metals if necessary)	1	SA-1.1, Sec. 6.9.2.2
23GW02-YYYYMMDD	Groundwater	NA	Total TAL metals (dissolved metals if necessary)	1	SA-1.1, Sec. 6.9.2.2
23GW03-YYYYMMDD and DUP-01	Groundwater	NA	Total TAL metals (dissolved metals if necessary)	1 + Field Duplicate + MS/MSD[1]	SA-1.1, Sec. 6.9.2.2
BGGW01-YYYYMMDD	Groundwater	NA	Total TAL metals (dissolved metals if necessary)	1	SA-1.1, Sec. 6.9.2.2
BGGW02-YYYYMMDD	Groundwater	NA	Total TAL metals (dissolved metals if necessary)	1	SA-1.1, Sec. 6.9.2.2

[1] Triplicate volume will be collected for MS/MSD analyses.

SAP Worksheet No. 19 -- Analytical SOP Requirements Table

(UFP-QAPP Manual Section 3.1.1)

Matrix	**Analytical Group**	**Analytical and Preparation Method / SOP Reference**	**Containers** (number, size, and type)	**Sample volume** (units)	**Preservation Requirements** (chemical, temperature, light protected)	**Maximum Holding Time** (preparation / analysis)
Groundwater	Aluminum, barium, chromium, cobalt, copper, iron, manganese, selenium, and zinc	SW-846 3010A/6010B Katahdin CA-604/608	1 – 500 mL High Density Polypropylene bottle	100 mL minimum	Cool to 4°C ± 2°C Nitric Acid (HNO_3) to pH<2	180 days to analysis
	Antimony, arsenic, beryllium, cadmium, lead, nickel, silver, thallium, and vanadium	SW-846 3010A/6020 Katahdin CA-604/627				
	Mercury	SW-846 7470A Katahdin CA-615				28 days to analysis

SAP Worksheet No. 20 -- Field Quality Control Sample Summary Table
(UFP-QAPP Manual Section 3.1.1)

Matrix	Analytical Group	No. of Sampling Locations	No. of Field Duplicates	No. of MS/MSDs	No. of Field Blanks[2]	No. of Equip. Blanks[3]	No. of PT Samples	Total No. of Samples to Lab
Groundwater	Total TAL Metals	5	1	1/1	1	1	0	10
Groundwater	Dissolved TAL Metals	5	1	1/1	NA	1	0	9

[2] A field blank will be collected at the beginning of the sampling effort representing the source water used for decontamination, if a non-dedicated pump is used for sampling.

[3] An equipment rinsate blank will be collected if a non-dedicated submersible pump is used for sampling. A filtered rinsate blank will be collected by running analyte-free deionized or distilled water through a 0.45-micron filter.

SAP Worksheet No. 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
GH-1.2	Evaluation of Existing Monitoring Wells and Water Level Measurement Revision 2, September 2003	Tetra Tech	NA	N	
SA-1.1	Groundwater Sample Acquisition and Onsite Water Quality Testing Revision 7, April 2008	Tetra Tech	Peristaltic Pump	N	
Sec. 6.9.2.2	NJDEP Low-Flow Purging and Sampling March 2005	NJDEP	Peristaltic Pump	N	
CT-04	Sample Nomenclature Revision 2, March 2009	Tetra Tech	NA	Y	Samples will be identified according to Worksheet No. 18
GH-2.8	Groundwater Monitoring Well Installation Revision 3, September 2003	Tetra Tech	Pump or Bailer	Y	Only Section 5.4 will be used for well redevelopment
SA-6.1	Non-radiological Sample Handling Revision 3, February 2004	Tetra Tech	Sample Bottleware, Packaging Material, Shipping Materials	N	
SA-6.3	Field Documentation Revision 3, March 2009	Tetra Tech	NA	N	
SA-7.1	Decontamination of Field Equipment Revision 6, January 2009	Tetra Tech	Decontamination Equipment (scrub brushes, phosphate free detergent, de-ionized water)	Y	Decontamination is limited to detergent wash and deionized water rinse. Nitric acid will be removed from the procedure.

SAP Worksheet No. 22 -- Field Equipment Calibration, Maintenance, Testing, and Inspection Table
(UFP-QAPP Manual Section 3.1.2.4)

Field Equipment	Activity	Frequency	Acceptance Criteria	Corrective Action	Resp. Person	SOP Reference	Comments
Multi-Parameter Water Quality Meter	Visual Inspection, Calibration	Daily	Manufacturer's Guidance	Replace	FOL	SA-1.1, Sec. 6.9.2.2, and Manufacturer's Guidance	
Turbidity Meter	Visual Inspection, Calibration	Daily	Manufacturer's Guidance; Calibrations must bracket expected values. Initial Calibration Verification (ICV) must be <10 NTU	Replace	FOL	SA-1.1, Sec. 6.9.2.2, and Manufacturer's Guidance	To be used to determine the need to collect Dissolved TAL Metals samples (if >10 NTU)
Peristaltic Pump	Visual Inspection	Daily	Equipment Inspection Sheet Criteria	Replace	FOL	SA-1.1, Sec. 6.9.2.2	
Submersible Pump	Visual Inspection	Daily	Manufacturer's Guidance	Replace	FOL	SA-1.1, Sec. 6.9.2.2	
Water Level Indicator	Field Checks per Manufacturer's Guidance	Once, upon Receiving from Vendor	0.01-foot accuracy	Replace	FOL	GH-1.2	

SAP Worksheet No. 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)
CA-101	Equipment Maintenance 07/08, Revision 7	Definitive	Various	Various	Katahdin	N
CA-604	Acid Digestion of Aqueous Samples by EPA Method 3010 for ICP Analysis of Total or Dissolved Metals 05/09, Revision 4	Definitive	Groundwater / Metals Digestion	NA (sample preparation)	Katahdin	N
CA-608	Trace Metals Analysis by ICP-AES Using EPA Method 6010 02/09, Revision 8	Definitive	Groundwater / TAL Metals (except Mercury)	Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES)	Katahdin	N
CA-627	Trace Metals Analysis by ICP-MS Using EPA Method 6020 02/09, Revision 5	Definitive	Groundwater / TAL Metals (except Mercury)	Inductively Coupled Plasma-Mass Spectroscopy (ICP-MS)	Katahdin	N
CA-615	Digestion and Analysis of Aqueous Samples for Mercury by EPA Method 7470 03/09, Revision 3	Definitive	Groundwater / Mercury	Cold Vapor Atomic Absorption (CVAA)	Katahdin	N

SAP Worksheet No. 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	SOP Reference
ICP-AES	Initial calibration	At the beginning of each day or if QC is out of criteria.	One point calibration per manufacturer's guidelines	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards	Analyst, Supervisor	CA-608
	Continuing calibration	At the beginning and end of each run sequence and every 10 samples	90-110% of True Values	Check problem, recalibrate and reanalyze any samples not bracketed by passing CCVs.	Analyst, Supervisor	
	Initial Calibration Blank (ICB)	Immediately following Initial calibration	Absolute value < QL.	(1) Do not use result is ≥ QL and < 10x the CCB level. (2) Investigate and correct problem.	Analyst, Supervisor	
	Continuing Calibration Blank (CCB)	Immediately following each calibration verification	Absolute value < QL.	(1) Do not use result is ≥ QL and < 10x the CCB level. (2) Investigate and correct problem.	Analyst, Supervisor	
ICP-MS	Tune	Daily prior to calibration	Mass calibration within 0.1 amu of true value, Resolution < 0.9 amu at 10% peak height	Perform necessary equipment maintenance	Analyst, Supervisor	CA-627
	Initial calibration	Daily prior to sample analysis.	4 point calibration plus blank – correlation coefficient ≥ 0.995.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards	Analyst, Supervisor	
	Continuing calibration	At the beginning and end of each run sequence and every 10 samples	90-110% of True Values	Check problem, recalibrate and reanalyze any samples not bracketed by passing CCVs.	Analyst, Supervisor	
	ICB	Immediately following Initial calibration	Absolute value < QL.	(1) Do not use result is ≥ QL and < 10x the CCB level. (2) Investigate and correct problem.	Analyst, Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
	CCB	Immediately following each calibration verification	Absolute value < QL.	(1) Do not use result is ≥ QL and < 10x the CCB level. (2) Investigate and correct problem.	Analyst, Supervisor	
Mercury analyzer	Initial calibration	IC-instrument receipt, major instrument change, at the start of each day	Correlation coefficient ≥ 0.995.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards	Analyst, Supervisor	CA-611, CA-615
	Continuing calibration	CCV-at beginning and end of each run sequence and every 10 samples	80-120% of True Value	Check problem, recalibrate and reanalyze any samples not bracketed by passing CCVs.	Analyst, Supervisor	
	ICB	At beginning of run	Absolute value < QL.	Correct problem and recalibrate.	Analyst, Supervisor	
	CCB	After every 10 samples, and at the end of run	Absolute value < QL.	(1) Correct problem and recalibrate. (2) Reanalyze all sample analyzed since the last successful CCB.	Analyst, Supervisor	

SAP Worksheet No. 25 -- Analytical Instrument & Equipment Maintenance, Testing, & Inspection Table
(UFP-QAPP Manual Section 3.2.3)

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
ICP-AES	Clean torch assembly and spray chamber when discolored or when degradation in data quality is observed. Clean nebulizer, check argon, and replace peristaltic pump tubing as needed. Other maintenance specified in lab Equipment Maintenance SOP.	QC standards	Torch, nebulizer chamber, pump, pump tubing	Prior to initial calibration and as necessary	Acceptable calibration or CCV	Correct the problem and repeat calibration or CCV	Analyst, Department Manager	CA-608
ICP-MS	Clean torch assembly and spray chamber when discolored or when degradation in data quality is observed. Clean nebulizer, check argon, and replace peristaltic pump tubing as needed. Other maintenance specified in lab Equipment Maintenance SOP.	QC standards	Torch, nebulizer, spray chamber, pump tubing	Prior to initial calibration and as necessary	Acceptable calibration or CCV	Correct the problem and repeat calibration or CCV	Analyst, Department Manager	CA-627
Mercury Analyzer	Replace peristaltic pump tubing, replace mercury lamp, replace drying tube, clean optical cell and/or clean liquid/gas separator as needed. Other maintenance specified in lab Equipment Maintenance SOP.	QC standards	Tubing, sample probe, optical cell	Prior to initial calibration and as necessary	Acceptable calibration or CCV	Correct the problem and repeat calibration or CCV	Analyst, Department Manager	CA-615

SAP Worksheet No. 26 -- Sample Handling System
(UFP-QAPP Manual Appendix A)

Sample Handling System

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): Joseph Tomalavage/Tetra Tech
Sample Packaging (Personnel/Organization): Joseph Tomalavage/Tetra Tech
Coordination of Shipment (Personnel/Organization): Joseph Tomalavage/Tetra Tech
Type of Shipment/Carrier: Overnight courier service (Federal Express)
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Sample custodians/Katahdin
Sample Custody and Storage (Personnel/Organization): Sample custodians/Katahdin
Sample Preparation (Personnel/Organization): Preparation Laboratory Staff/Katahdin
Sample Determinative Analysis (Personnel/Organization): ICP-AES, ICP-MS, and CVAA chemists/Katahdin
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): 60 days from submittal of final report
Sample Extract/Digestate Storage (No. of days from extraction/digestion): 1 year from submittal of final report
Biological Sample Storage (No. of days from sample collection): Not Applicable
SAMPLE DISPOSAL
Personnel/Organization: Sample custodians/Katahdin
Number of Days from Analysis: 30 days from submittal of final report

SAP Worksheet No. 27 -- Sample Custody Requirements Table
(UFP-QAPP Manual Section 3.3.3)

Field Sample Custody Procedures (sample collection, packaging, shipment, and delivery to laboratory): Following sample collection into the appropriate bottleware, all samples will be immediately placed on ice in a cooler. The cooler will be secured using strapping tape along with a signed custody seal. Sample coolers will be delivered to a local courier location or picked up by a courier for priority overnight delivery to the selected laboratory for analysis. Samples will be preserved as appropriate based on the analytical method. Laboratories will provide pre-preserved sample containers for sample collection. Samples will be maintained at 4°C until delivery to the laboratories. Proper custody procedures will be followed throughout all phases of sample collection and handling. Chain-of-custody protocols will be used throughout sample handling to establish the evidentiary integrity of sample containers. These protocols will be used to demonstrate that the samples were handled and transferred in a manner that would eliminate possible tampering. Samples for the laboratory will be packaged and shipped in accordance with Tetra Tech SOP SA-6.1.

Laboratory Sample Custody Procedures (receipt of samples, archiving, disposal): Custody Seals are supplied with all bottle orders. They are affixed to the cooler after sampling. The presence or absence of Custody Seals is noted on the Sample Receipt Condition Report (see below).

Upon receipt of samples from the field, the laboratory sample management personnel will sign off on the chain-of-custody, open the sample cooler(s), verify sample integrity and conduct a check against the chain-of-custody. If there is a discrepancy or problem (i.e. broken sample containers) the laboratory will contact the field leader or other qualified personnel and resolve the issue. Additionally, the laboratory completes a Sample Receipt Condition Report (SRCR), which documents visual inspection of the samples and specific parameters such as cooler temperature, holding times, and preservation. Discrepancies or changes will be documented on the Sample Receipt Condition Report.

The laboratory sample management personnel assigns a unique laboratory work order number for the entire sample set listed on the chain of custody. The samples are then logged into our laboratory information system and a Login Chain of Custody Report is generated. Each sample within a work order is labeled numerically. Each container of a particular sample is uniquely identified by adding an alphabetical suffix to the sample number. The laboratory labels each sample container with a Laboratory Custody Label which will remain on the sample bottle for the duration of the laboratory sample storage. The laboratory also initiates the appropriate Internal Custody Record for the sample set. Personnel fill out the Internal Custody Records to document sample removal from and return to sample storage.

A laboratory data file is also initiated for the work order. This file includes the Login chain-of-custody, chain-of-custody and Sample Receipt Condition Report. The folder also includes a Login File Sheet which summarizes the analyses that the work order has been logged for. This sheet is used to track data completion.

Samples for a project may be batched or grouped together by the laboratory. A series of batched work orders is referred to as a SDG. The SDG includes those samples received on a chain of custody, duplicate samples, and field QA/QC samples, and can include samples of different media. QA/QC samples will be run at the frequency specified in the analytical methods. The sample delivery group is given a specific identification number.

Samples are stored at the laboratory in refrigerators prior to, during, and after analysis. Refrigerators at the laboratory are constantly monitored for temperature. Proper temperatures and lighting are maintained in the refrigerators to ensure sample integrity and preservation. Samples are retained by the laboratory for a period of 90 days after the data report is mailed to the client unless otherwise specified in a client contract. The laboratory then disposes of non-hazardous samples, following certified disposal practices. Hazardous samples are either returned to the client or disposed of through a licensed broker. Documentation of disposal is maintained by the laboratory.

Please refer to Katahdin Analytical Services SOPs SD-902 “Sample Receipt and Internal Control” and SD-903, “Sample Disposal” for more information.

SAP Worksheet No. 27 (continued)

Sample Identification Procedures: Sample nomenclature will be conducted in general accordance with Worksheet Nos. 14 and 18 and the procedures outlined in Tetra Tech SOP CT-04 (Sample Nomenclature). Sample nomenclature put forth for this field event has been selected based on historical usage.

Chain-of-custody Procedures: After recovery, each sample will be maintained in the sampler's custody until formally transferred to another party (e.g., Federal Express). For all samples recovered, custody records will document the date and time of sample collection, the sampler's name, and the names of all others who subsequently held custody of the sample. Specifications for chemical analyses will also be documented on the custody record. Attached SOP SA-6.3 (Field Documentation) provides further details on the chain-of-custody procedure. Chain-of-custody requirements are also documented with instructions contained in each shipment from the laboratory (Katahdin SOP SD-902).

SAP Worksheet No. 28 -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix	Groundwater
Analytical Group	Metals
Analytical Method/ SOP Reference	SW-846 6010B CA-608

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Preparation Blank	One per prep batch of 20 or fewer samples of similar matrix	Absolute value < QL.	(1) If blank value > QL report sample results if < QL or > 10 x the blank value; otherwise re-digest. (2) If blank value is less than negative QL, report sample results if > 10x the absolute value of the blank result, otherwise re-digest.	Analyst/Supervisor, QA Manager	Accuracy / Bias	Absolute value < QL .sample results if > 10x the absolute value of the blank result, otherwise re-digest.
ICB/CCB	Immediately following each calibration verification	Absolute value < QL.	(1) Do not use result is ≥ QL and < 10x the CCB level. (2) Investigate and correct problem.	Analyst/Supervisor, QA Manager	Accuracy / Bias	Absolute value < QL.
Laboratory Control Sample (LCS)	One per prep batch of 20 or fewer samples of similar matrix	Recovery within ± 20% of true value.	(1) If the LCS recoveries are > 120 % but the sample results are <QL narrate. (2) Re-digest and reanalyze all associated samples for affected analytes.	Analyst, Supervisor, QA Manager	Accuracy / Bias	80-120 % recovery.
Interference Check Sample (ICS)	Beginning and end of analytical run	For Al, Ca, Fe, Mg recovery ± 20% of true value. For analytes not spiked, ± QL, or if QL < 0.1 mg/L, ± 2xQL.	(1) Do not use result s for failing elements. (2) Investigate and correct problem.	Analyst, Supervisor, QA Manager	Accuracy / Bias	For Al, Ca, Fe, Mg recovery ± 20% of true value. For analytes not spiked, ± QL, or if QL < 0.1 mg/L, ± 2xQL.
MS	One per prep batch of 20 or fewer samples of similar matrix	Recovery ± 25 % of true value if sample < 4x spike value	Flag results for affected analytes for all associated samples with ”N”.	Analyst, Supervisor, QA Manager	Accuracy / Bias / Precision	Recovery ± 25 % of true value if sample < 4x spike value
MSD	One per prep batch of 20 or fewer samples of similar matrix	Recovery ± 25 % of true value if sample < 4x spike value RPD ≤ 20 %	Flag results for affected analytes for all associated samples with ”N”.	Analyst, Supervisor, QA Manager	Accuracy / Bias / Precision	Recovery ± 25 % of true value if sample < 4x spike value RPD ≤ 20 % (If a laboratory duplicate is performed instead of an MSD, only an RPD will be measured.)
ICP Serial Dilution	One per prep batch of 20 or fewer samples of similar matrix	If original sample result is at least 50x IDL, 5-fold dilution must agree within ± 10% of the original result.	Flag results for affected analytes for all associated samples with “E”.	Analyst, Supervisor, QA Manager	Accuracy / Bias / Precision	If original sample result is at least 50x IDL, 5-fold dilution must agree within ± 10% of the original result.

Matrix	Groundwater
Analytical Group	TAL Metals
Analytical Method / SOP Reference	SW-846 6020 CA-627

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Preparation Blank	One per prep batch of 20 or fewer samples of similar matrix	Absolute value < QL.	(1) Investigate source of contamination. (2) If blank value > QL report sample results if < QL or > 10 x the blank value; otherwise re-digest.	Analyst, Supervisor, Quality Assurance Manager	Accuracy / Bias	Absolute value < QL.
ICB/CCB	Immediately following each calibration verification	Absolute value < QL.	(1) Do not use result is ≥ QL and < 10x the CCB level. (2) Investigate and correct problem.	Analyst/Supervisor, QA Manager	Accuracy / Bias	Absolute value < QL.
LCS	One per prep batch of 20 or fewer samples of similar matrix	Recovery within ± 20% of true value.	(1) If the LCS recoveries are > 120 % but the sample results are <QL narrate. (2) Re-digest and reanalyze all associated samples for affected analytes.	Analyst, Supervisor, Quality Assurance Manager	Accuracy / Bias	80-120 % recovery.
ICS	Beginning and end of analytical run	Recovery ± 20% of true value.	Do not use sample results for failing analytes, unless ICSB > 120% and sample result <QL.	Analyst, Supervisor, Quality Assurance Manager	Accuracy / Bias	Recovery ± 20% of true value.
MS	One per prep batch of 20 or fewer samples of similar matrix	Recovery ± 25 % of true value if sample < 4x spike value	(1) Flag results for affected analytes for all associated samples with "N". (2) Perform post-digestion spike for all failing elements.	Analyst, Supervisor, Quality Assurance Manager	Accuracy / Bias / Precision	Recovery ± 25 % of true value if sample < 4x spike value
MSD	One per prep batch of 20 or fewer samples of similar matrix	Recovery ± 25 % of true value if sample < 4x spike value RPD ≤ 20%	(1) See above. (2) If RPD > 20%, flag results for affected analytes for all associated samples with "*".	Analyst, Supervisor, Quality Assurance Manager	Accuracy / Bias	RPD ≤ 20%
ICP Serial Dilution	One per prep batch of 20 or fewer samples of similar matrix	If original sample result is at least 50x IDL, 5-fold dilution must agree within ± 10% of the original result.	Flag results for affected analytes for all associated samples with "E".	Analyst, Supervisor, Quality Assurance Manager	Accuracy / Bias / Precision	If original sample result is at least 50x IDL, 5-fold dilution must agree within ± 10% of the original result.
Internal Standard (IS)	Appropriate IS required for all analytes in all samples. Mass of IS must be <50 amu different from that of analyte	For each sample, IS intensity must be within 30%-120% of that of initial calibration standard. For ICV, ICB, CCV, and CCB, IS intensity within 80%-120% of that in initial calibration standard.	Reanalyze affected samples	Analyst, Supervisor, Quality Assurance Manager	Accuracy	For each sample, IS intensity must be within 30%-120% of that of initial calibration standard. For ICV, ICB, CCV, and CCB, IS intensity within 80%-120% of that in initial calibration standard.

Matrix	Groundwater
Analytical Group	Mercury
Analytical Method / SOP Reference	SW-846 7470A CA-615

QC Sample:	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Preparation Blank	One per prep batch of 20 or fewer samples of similar matrix	Absolute value < QL.	(1) If blank value > QL report sample results if < QL or > 10 x the blank value; otherwise re-digest. (2) If blank value is less than negative QL, report sample results if > 10x the absolute value of the blank result, otherwise re-digest.	Analyst/Supervisor, QA Manager	Accuracy/bias-	Absolute value < QL sample results if > 10x the absolute value of the blank result, otherwise re-digest.
ICB/CCB	At beginning of run and after every 10 samples, and at the end of run	Absolute value < QL.	(1) Correct problem and recalibrate. (2) Reanalyze all sample analyzed since the last successful CCB.	Analyst/Supervisor, QA Manager	Accuracy/bias-	Absolute value < QL.
LCS	One per prep batch of 20 or fewer samples of similar matrix	Recovery within ± 20% of true value.	(1) If the LCS recoveries are > 120 % but the sample results are <QL narrate. (2) Re-digest and reanalyze all associated samples for affected analytes.	Analyst/Supervisor, QA Manager	Accuracy/Bias	± 20%
MS	One per prep batch of 20 or fewer samples of similar matrix	Recovery ± 25 % of true value if sample < 4x spike value	Flag results for affected analytes for all associated samples with ”N”.	Analyst/Supervisor, QA Manager	Accuracy/bias	Recovery ± 25 % of true value if sample < 4x spike value
MSD	One per prep batch of 20 or fewer samples of similar matrix	Recovery ± 25 % of true value if sample < 4x spike value RPD ± 20%	Flag results for affected analytes for all associated samples with “*”.	Analyst/Supervisor, QA Manager	Accuracy/Bias, Precision	Recovery ± 25 % of true value if sample < 4x spike value RPD ± 20%
Serial Dilution	One per prep batch of 20 or fewer samples of similar matrix	If original sample result is at least 50x IDL, 5-fold dilution must agree within ± 10% of the original result.	Flag results for affected analytes for all associated samples with “E”.	Analyst/Supervisor, QA Manager	Accuracy/Bias, Precision	If original sample result is at least 50x IDL, 5-fold dilution must agree within ± 10% of the original result.

SAP Worksheet No. 29 -- Project Documents and Records Table

(UFP-QAPP Manual Section 3.5.1)

Document	Where Maintained
Field Documents Field Logbook Field Sample Forms Chain-of-custody Records Air bills Sampling Instrument Calibration Logs Sampling Notes Photographs Field Task Modification Request Forms This SAP Health and Safety Plan	Field documents will be maintained in the project file located in the Tetra Tech King of Prussia office.
Laboratory Documents Sample receipt, custody, and tracking record Standards traceability logs Equipment calibration logs Sample preparation logs Analysis Run logs Equipment maintenance, testing, and inspection logs Corrective action forms Reported field sample results Reported results for standards, QC checks, and QC samples Sample storage and disposal records Telephone logs Extraction/clean-up records Raw data Data Completeness checklist	Laboratory documents will be included in the hardcopy and PDF deliverables from the laboratory. Laboratory data deliverables will be maintained in the Tetra Tech King of Prussia project file and in long-term data package storage at a third-party professional document storage firm. Electronic data results will be maintained in a database on a password protected Structured Query Language (SQL) server.
Assessment Findings Data Validation Memoranda (includes tabulated data summary forms)	All data validation memoranda will be maintained in the Tetra Tech King of Prussia project file.
Reports Site 23 Sampling Report	All reports for the Site 23 Groundwater Sampling will be stored in hardcopy in the Tetra Tech King of Prussia project file and electronically in the server library.

SAP Worksheet No. 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)
Groundwater	TAL Metals (except Mercury)	See Worksheet No. 18	SW-846 3010A/6010B Katahdin CA-604/608	21 days	Katahdin Analytical Services 600 Technology Way Scarborough, ME 04074 (207)874-2400 Contact: Kate Zaleski	NA
Groundwater	TAL Metals (except Mercury)	See Worksheet No. 18	SW-846 3010A/6020 Katahdin CA-604/627			
Groundwater	Mercury	See Worksheet No. 18	SW-846 7470A Katahdin CA-615			

SAP Worksheet No. 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 (title and organizational affiliation)	Person(s) Responsible for Monitoring Effectiveness of Corrective Actions (title and organizational affiliation)
Field Supervision	Daily during sampling events	Internal	Tetra Tech	Tetra Tech FOL	Tetra Tech Field Crew	Tetra Tech FOL and Field Crew	Tetra Tech FOL, PM, and CLEAN QAM
Project Supervision	Every sampling event	Internal	Tetra Tech	Tetra Tech PM	Tetra Tech FOL	Tetra Tech PM and FOL	Tetra Tech PM and FOL
Health and Safety Audit	1 per contract year	Internal	Tetra Tech	Tetra Tech Health and Safety Personnel	PM	Auditor and HSM	HSM
Laboratory Systems Audit	1 per contract year	Internal	Tetra Tech	Person assigned by Tetra Tech QAM	Tetra Tech PM and FOL	Tetra Tech Auditor and PM	Tetra Tech CLEAN QAM
Laboratory Systems Audit[1]	Every 18 months	External	DoD Environmental Laboratory Accreditation Program (ELAP)	DoD ELAP	Laboratory QA Manager or Laboratory Manager	Laboratory QAM or Laboratory Manager	DoD ELAP

[1] Katahdin has successfully completed the laboratory evaluation process required as part of the DoD ELAP and as described in the DoD QSM. The DoD ELAP certification letter is included in Appendix B.

SAP Worksheet No. 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
Health and Safety Audit	Audit checklist and written audit finding summary	Mary Mang, PM, Joseph Tomalavage, FOL, and John Trepanowski, Program Manager, Tetra Tech	Dependant on findings, if major, a stop work may be issued immediately, however, if minor, within 1 week of audit.	Written Memo	Matt Soltis, HSM, Tetra Tech Auditor and John Trepanowski, Program Manager, Tetra Tech	Within 48 hours of notification
Field Supervision	Site log book and sample collection logs	Mary Mang, PM, Joseph Tomalavage, FOL, Tetra Tech	Immediately	Entry in site log book	Mary Mang, PM, Joseph Tomalavage, FOL, Tetra Tech	24 hours
Project Supervision	Written report	John Trepanowski Program Manager; Garth Glenn Deputy Program Manager, Tetra Tech	Monthly	Written memo	John Trepanowski Program Manager; Garth Glenn Deputy Program Manager, Tetra Tech	Within a week of notification
Field Sampling System Audit[1]	Audit checklist (as per Navy Installation Restoration Chemical Data Quality Manuel [IRCDQM]) and written audit report	Mary Mang, PM, Joseph Tomalavage, FOL; John Trepanowski Program Manager; Garth Glenn, Deputy Program Manager, Tetra Tech	Dependent on the finding; if major a stop work may be issued immediately; however, if minor within 1 week of audit	Written memo	Mary Mang, PM, Joseph Tomalavage, FOL; John Trepanowski Program Manager; Garth Glenn, Deputy Program Manager, Tetra Tech	Within 48 hours of notification
Laboratory System Audit	Written audit report	Leslie Dimond QAM, Katahdin Analytical	Not specified by DoD ELAP	Letter	DoD ELAP	Specified by DoD ELAP

[1] Audits are scheduled at the Tetra Tech program level and may or may not include this project.

SAP Worksheet No. 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	One per SDG	within 1 month after receipt of data	Data Validator	Tetra Tech PM, project file
Major analysis problem identification (Internal Memorandum)	When persistent analysis problems are detected	Immediately	Tetra Tech QAM	Tetra Tech PM, project file
Project monthly progress report	Monthly for duration of project	Monthly	Tetra Tech PM	Tetra Tech PM, QAM, Program Manager; project file
Field progress report	Daily, oral, during the course of sampling	Every day that field sampling is occurring	Tetra Tech FOL	Tetra Tech PM
Laboratory QA report	When significant plan deviations results from unanticipated circumstances	Immediately	Katahdin PM	Tetra Tech PM, project file

SAP Worksheet No. 34 -- Verification (Step I) Process Table
(UFP-QAPP Manual Section 5.2.1)

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
Sample Tables (Worksheet No. 18)	Proposed samples verified to have been collected.	Internal	Tetra Tech FOL or designee
Chain-of-custody	Chain-of-custody records will be reviewed internally by the Project Manager or designee and compared against sample table (Worksheet No. 18) listing the proposed samples to verify that all planned samples have been collected.	Internal	Tetra Tech PM or designee
Sample Coordinates	Sample locations have been verified to be correct and in accordance with the SAP (overlay maps proposed locations against actual locations).	Internal	Tetra Tech FOL, PM, or designee
Electronic Data	Verify that all sample results are included in the Electronic Data Deliverable from the laboratory. Verify the samples against the chain-of-custody. Upload the data into the SQL database for the data validator.	Internal	Laboratory PM and Tetra Tech Sample Management Coordinator
Analytical Data Package	The analytical data will be verified for completeness by the laboratory performing the work. The laboratory shall complete an appropriate form documenting the organization and complete contents of each data package.	External	Laboratory Personnel
Data Package	Verify that the data package contains all the elements required by CLP format and the scope of work, this occurs as part of the data validation process.	Internal	Tetra Tech Data Validator
Sample Log Sheets	Log sheets completed as samples are collected in the field are verified for completeness and are maintained at the project office.	Internal	Tetra Tech PM or designee

SAP Worksheet No. 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)

[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]

Step IIa / IIb[1]	Validation Input	Description	Responsible for Validation (name, organization)
IIa	Field SOPs/Field Logs/Sample Collection	Ensure that all sampling SOPs were followed. Verify that deviations have been documented and MPCs have been achieved. Particular attention should be given to verify that samples were correctly identified, that sampling location coordinates are accurate, and that documentation establishes an unbroken trail of documented chain-of-custody from sample collection to report generation. Verify that the correct sampling and analytical methods/SOPs were applied. Verify that the sampling plan was implemented and carried out as written and that any deviations are documented.	Tetra Tech PM, FOL, or designee
IIa	Analytical SOPs	Ensure that all laboratory SOPs were followed. Verify that the correct analytical methods/SOPs were applied.	Katahdin Personnel
IIa	Documentation of Method QC Results	Establish that all method QC samples were analyzed and in control as listed in the analytical SOPs. If method QA is not in control, the laboratory will contact Tetra Tech for guidance prior to report preparation.	Katahdin Personnel
IIa	Chain-of-Custody	Ensure that the custody and integrity of the samples was maintained from collection to analysis and the custody records are complete and any deviations are recorded.	Tetra Tech Project Chemist or Data Validator
IIa	Holding Times	Review that the samples were shipped and stored at the required temperature and sample pH for chemically-preserved samples meet the requirements listed in Worksheet #19. Ensure that the analyses were performed within the holding times listed in Worksheet #19.	Tetra Tech Project Chemist or Data Validator
IIa/IIb	Laboratory Data Results for Accuracy	Ensure that the laboratory QC samples listed in Worksheet #28 were analyzed and that the MPC listed in Worksheet #12 were met for all field samples and QC analyses. Check that specified field QC samples were collected and analyzed and that the analytical QC criteria set up for this project were met.	Tetra Tech Project Chemist or Data Validator

Step IIa / IIb[1]	Validation Input	Description	Responsible for Validation (name, organization)
IIa/IIb	Laboratory Duplicate Analyses for Precision	Check the laboratory precision by reviewing the RPD or percent difference values from laboratory duplicate analyses; MS/MSDs; and LCS/LCSDs. Ensure compliance with the methods and project MPC accuracy goals listed in Worksheet #12.	Tetra Tech Project Chemist or Data Validator
IIa/IIb	Sample Results for Representativeness	Check 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.	Tetra Tech Project Chemist or Data Validator
IIa/IIb	PALs	Discuss the impact on matrix interferences or sample dilutions performed because of the high concentration of one or more contaminant, on the other target compounds reported as no-detected. Document this usability issue and inform the PM.	Tetra Tech Project Chemist or Data Validator
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 project data base qualified data depicting data qualifiers and data qualifiers 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.	Tetra Tech Project Chemist or Data Validator
IIa, IIb	SAP QC Sample Documentation	Ensure that all QC samples specified in the SAP were collected and analyzed and that the associated results were within prescribed SAP acceptance limits. Ensure that QC samples and standards prescribed in analytical SOPs were analyzed and within the prescribed control limits. If any significant QC deviations occur, the laboratory shall have contacted the Tetra Tech PM.	Tetra Tech PM or designee
IIa, IIb	Documentation of Analytical Reports for Completeness	Review the chain-of-custody form generated in the field to ensure that the required analytical samples have been collected, appropriate sample identifications have been used, and correct analytical methods have been applied. Validator will verify that required elements of the data package for validation are present, and if not, the laboratory will be contacted and the missing information will be requested. Validation will be performed as per Worksheet #36. Check that all data have been transferred correctly and completely to the final Structured Query Language (SQL) database.	Tetra Tech Project Chemist or Data Validator
IIa/IIb	PALs	Review and add PSLs to the laboratory EDD. Flag samples and notify PM of samples that exceed PSLs, as listed on Worksheet #15.	Tetra Tech PM or designee

Step IIa / IIb[1]	Validation Input	Description	Responsible for Validation (name, organization)
IIb	QLs for Sensitivity	Ensure that the QLs listed in Worksheet #15 were achieved.	Tetra Tech Project Chemist or Data Validator
IIb	Analytical Data Deviations	Determine the impact of any deviation from sampling or analytical methods and SOPs requirements and matrix interferences effect on the analytical results.	Tetra Tech Project Chemist or Data Validator

SAP Worksheet No. 36 -- Analytical 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	Total and Dissolved TAL Metals	Full data validation. Project-specific criteria for SW-846 6010B/6020/7470A listed in Worksheet Nos. 12, 15, 24 and 28. If not included in the aforementioned the logic outlined in Tetra Tech SOP DV-04 and the EPA Region 2 SOP HW-2 Rev. 13, Evaluation of Metals Data for the CLP Program (September 2006) should be used to apply qualifiers to data.	Tetra Tech Data Validator

SAP Worksheet No. 37 -- Usability Assessment

(UFP-QAPP 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 Tetra Tech PM will determine whether the deviations compromise the ability to meet project objectives. If they do, the Tetra Tech 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. 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 Tetra Tech PM and acting on behalf of the project team will determine whether the data are adequately representative of intended populations, both spatially and temporally. This will be accomplished by verifying that samples were collected and processed for analysis in accordance with the SAP, by reviewing spatial and temporal data variations, and by comparing these characteristics 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 evaluate deviations from planned sensitivity goals.

Project Assumptions and Data Outliers

The Tetra Tech Project Manager and designated team members will evaluate whether project assumptions are 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. Statistical tests for outliers will be conducted using standard statistical techniques appropriate for this task. Potential outliers will be removed if a review of the associated indicates that the results have an assignable cause the renders them inconsistent with the rest of the data. During this evaluation, the team will consider whether outliers could be indications of unanticipated site conditions. Consideration will be given to whether outliers represent an unanticipated site condition.

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. The project team members identified by the project manager will assess whether the data collectively support the attainment of project objectives. Data concentrations that were determined to be unusable during the data validation will be removed from the data set. 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. The average of the original and duplicate samples will be used to represent the concentration at a particular sampled location. MPCs for defining good duplicate precision are presented in Worksheet #12. Duplicates that are not within MPCs will be qualified during the data validation and these limitations will be considered during the usability assessment.

Identify the personnel responsible for performing the usability assessment:

The Tetra Tech Project Manager, Project Chemist, FOL, and Project Scientist will be responsible for conducting the listed data usability assessments. The data usability assessment will be reviewed with the Navy RPM, the EPA RPM, and the NJDEP PM. 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. 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, if necessary.

REFERENCES

Brown and Root Environmental, 1996. Remedial Investigation Report for Naval Weapons Station Earle, Colts Neck, New Jersey. July.

DoD (U.S. Department of the Defense), U.S. DoE, U.S. EPA, 2005. Interagency Data Quality Task Force (IDQTF) Uniform Federal Policy for Quality Assurance Project Plans. March.

DoD, 2006. Department of Defense Quality Systems Manual. January.

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

EPA, 2006. Validation of Metals for the Contract Laboratory Program based on ILM05.3. U.S. EPA Region 2 SOP #HW-2, Revision 13. September.

Foster Wheeler Environmental Corp, 1996. Action Memorandum Sites 22, 23, and 27 Paint Disposal Areas. September.

Foster Wheeler Environmental Corp, 1996. Engineering Evaluation/Cost Analysis Clean Up of Site 22 - Paint Chip Disposal Area, Site 23 – Paint Disposal Area and Site 27 – Projectile Refurnishing Area, Naval Weapons Station Earle, Installation Restoration Sites 22, 23 and 27. September.

Foster Wheeler Environmental Corp, 1997. Removal Actions at Sites 22, 23, 27, Naval Weapons Station Earle, Colts Neck, New Jersey. February.

NJDEP (New Jersey Department of Environmental Protection), 2006. Field Sampling Procedures Manual. August.

Tetra Tech (Tetra Tech NUS, Inc.), 1998. Proposed Plan Operable Unit 4 (OU-4) for Naval Weapons Station Earle, Colts Neck, New Jersey. April.

Tetra Tech, 1999. Record of Decision Operable Unit 4 (OU-4) Sites 14, 20, 22, 23, 24, 25, 27, and 29 Naval Weapons Station Earle, Colts Neck, New Jersey. August.

APPENDIX A

TETRA TECH NUS, INC.
STANDARD OPERATING PROCEDURES

CT-04	Sample Nomenclature
GH-1.2	Water Level Measurements
GH-2.8	Groundwater Monitoring Well Installation
SA-1.1	Groundwater Sample Acquisition
SA-6.1	Non-Radiological Sample Handling
SA-6.3	Field Documentation
SA-7.1	Decontamination of Field Equipment
6.9.2.2	NJDEP Field Sampling Procedures Manual 08/2005

TABLE OF CONTENTS

SECTION **PAGE**

1.0 PURPOSE **2**

2.0 SCOPE **2**

3.0 GLOSSARY **2**

4.0 RESPONSIBILITIES **2**

5.0 PROCEDURES **2**

5.1 INTRODUCTION 2
5.2 SAMPLE IDENTIFICATION FIELD REQUIREMENTS 3
5.3 EXAMPLE SAMPLE FIELD DESIGNATIONS 4
5.4 EXAMPLES OF SAMPLE NOMENCLATURE 5
5.5 FIELD QUALITY ASSURANCE/QUALITY CONTROL (QA/QC) SAMPLE NOMENCLATURE) 6
5.6 EXAMPLES OF FIELD QA/QC SAMPLE NOMENCLATURE 6

6.0 DEVIATIONS **6**

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.

TABLE OF CONTENTS

SECTION **PAGE**

1.0 PURPOSE **2**

2.0 SCOPE **2**

3.0 GLOSSARY **2**

4.0 RESPONSIBILITIES **2**

5.0 PROCEDURES **2**

5.1 PRELIMINARY EVALUATION 3
5.2 FIELD INSPECTION 3
5.3 WATER LEVEL (HYDRAULIC HEAD) MEASUREMENTS 4
5.3.1 General 4
5.3.2 Water Level Measuring Techniques 5
5.3.3 Methods 5
5.3.4 Water Level Measuring Devices 6
5.3.5 Data Recording 6
5.3.6 Specific Quality Control Procedures for Water Level Measuring Devices 7
5.4 EQUIPMENT DECONTAMINATION 7
5.5 HEALTH AND SAFETY CONSIDERATIONS 7

6.0 RECORDS **7**

ATTACHMENTS

A MONITOIRNG WELL INSPECTON SHEET 8
B GROUNDWATER LEVEL MEASUREMENT SHEET 9

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)

Page ____ of ____

TABLE OF CONTENTS

SECTION **PAGE**

1.0 PURPOSE **2**

2.0 SCOPE **2**

3.0 GLOSSARY **2**

4.0 RESPONSIBILITIES **2**

5.0 PROCEDURES **3**

5.1 EQUIPMENT/ITEMS NEEDED 3
5.2 WELL DESIGN 3
5.2.1 Well Depth, Diameter, and Monitored Interval 3
5.2.2 Riser Pipe and Screen Materials 5
5.2.3 Annular Materials 6
5.2.4 Protective Casing 6
5.3 MONITORING WELL INSTALLATION 7
5.3.1 Monitoring Wells in Unconsolidated Sediments 7
5.3.2 Confining Layer Monitoring Wells 7
5.3.3 Bedrock Monitoring Wells 8
5.3.4 Drive Points 8
5.3.5 Innovative Monitoring Well Installation Techniques 8
5.4 WELL DEVELOPMENT METHODS 8
5.4.1 Overpumping and Backwashing 8
5.4.2 Surging with a Surge Plunger 9
5.4.3 Compressed Air 9
5.4.4 High Velocity Jetting 9

6.0 RECORDS **9**

7.0 REFERENCES **10**

ATTACHMENTS

A RELATIVE COMPATIBILITY OF RIGID WELL-CASING MATERIAL (PERCENT) / RELATIVE COMPATIBILITY OF SEMI-RIGID OR ELASTOMERIC MATERIALS (PERCENT) 11
B COMPARISON OF STAINLESS STEEL AND PVC FOR MONITORING WELL CONSTRUCTION 12

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

Scalf, M. R., J. F. McNabb, W. J. Dunlap, R. L. Cosby, and J. Fryberger, 1981. Manual of Groundwater Sampling Procedures. R. S. Kerr Environmental Research Laboratory, Office of Research and Development, U.S. EPA, Ada, Oklahoma.

Barcelona, M. J., P. P. Gibb and R. A. Miller, 1983. A Guide to the selection of Materials for Monitoring Well Construction and Groundwater Sampling. ISWS Contract Report 327, Illinois State Water Survey, Champaign, Illinois.

U.S. EPA, 1980. Procedures Manual for Groundwater Monitoring of Solid Waste Disposal Facilities. Publication SW-611, Office of Solid Waste, U.S. EPA, Washington, D.C.

Driscoll, Fletcher G., 1986. Groundwater and Wells. Johnson Division, St. Paul, Minnesota, 1989.

ATTACHMENT A

RELATIVE COMPATIBILITY OF RIGID WELL CASING MATERIAL (PERCENT)

Potentially-Deteriorating Substance	Type of Casing Material						
	PVC 1	Galvanized Steel	Carbon Steel	Lo-carbon Steel	Stainless Steel 304	Stainless Steel 316	Teflon*
Buffered Weak Acid	100	56	51	59	97	100	100
Weak Acid	98	59	43	47	96	100	100
Mineral Acid/ High Solids Content	100	48	57	60	80	82	100
Aqueous/Organic Mixtures	64	69	73	73	98	100	100
Percent Overall Rating	91	58	56	59	93	96	100

Preliminary Ranking of Rigid Materials:

1 Teflon®
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.

TABLE OF CONTENTS

SECTION **PAGE**

1.0 PURPOSE **2**

2.0 SCOPE **2**

3.0 GLOSSARY **2**

4.0 RESPONSIBILITIES **2**

5.0 PROCEDURES **3**

5.1 GENERAL 3
5.2 SAMPLING, MONITORING, AND EVACUATION EQUIPMENT 4
5.3 CALCULATIONS OF WELL VOLUME 4
5.4 EVACUATION OF STATIC WATER (PURGING) 5
5.4.1 General 5
5.4.2 Evacuation Devices 5
5.5 ONSITE WATER QUALITY TESTING 6
5.5.1 Measurement of pH 7
5.5.2 Measurement of Specific Conductance 9
5.5.3 Measurement of Temperature 10
5.5.4 Measurement of Dissolved Oxygen 11
5.5.5 Measurement of Oxidation-Reduction Potential 12
5.5.6 Measurement of Turbidity 13
5.5.7 Measurement of Salinity 14
5.6 SAMPLING 15
5.6.1 Sampling Plan 15
5.6.2 Sampling Methods 16
5.7 LOW FLOW PURGING AND SAMPLING 17
5.7.1 Scope & Application 17
5.7.2 Equipment 17
5.7.3 Purging and Sampling Procedure 18

6.0 REFERENCES **20**

ATTACHMENTS

A PURGING EQUIPMENT SELECTION 21
B GROUNDWATER SAMPLE LOG SHEET 24
C LOW FLOW PURGE DATA SHEET 25

1.0 PURPOSE

The purpose of this procedure is to provide general reference information regarding the sampling of groundwater wells.

2.0 SCOPE

This procedure provides information on proper sampling equipment, onsite water quality testing, and techniques for groundwater sampling. 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 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, their total concentration, mobility, valence, and relative concentrations, and on temperature of measure. Conductivity is highly dependent on temperature and should be reported at a particular temperature, i.e., 20.2 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.

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 standard hydrogen electrode.

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.

Salinity – The measurement of dissolved salts in a given mass of solution. Note: most field meters determined salinity automatically from conductivity and temperature. The displayed value will be displayed in either parts per thousand (ppt) or % (e.g., 35 ppt will equal 3.5%).

Turbidity – Turbidity in water is caused by suspended matter, such as clay, silt, 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

Project Hydrogeologist - Responsible for selecting and detailing the specific groundwater sampling techniques, onsite water quality testing (type, frequency, and location), and equipment to be used, and providing detailed input in this regard to the project plan documents. The project hydrogeologist is also responsible for properly briefing and overseeing the performance of the site sampling personnel.

Project Geologist/Field Sample Technician - is primarily responsible for the proper acquisition of the groundwater samples. He/she is also responsible for the actual analyses of onsite water quality samples, as well as instrument calibration, care, and maintenance. When appropriate, such responsibilities may be performed by other qualified personnel (e.g., field technicians).

5.0 PROCEDURES

5.1 General

To be useful and accurate, a groundwater sample must be representative of the particular zone of the water being sampled. The physical, chemical, and bacteriological integrity of the sample must be maintained from the time of sampling to the time of analysis in order to keep any changes in water quality parameters to a minimum.

Methods for withdrawing samples from completed wells include the use of pumps, compressed air, bailers, and various types of samplers. The primary considerations in obtaining a representative sample of the groundwater are to avoid collection of stagnant (standing) water in the well and to avoid physical or chemical alteration of the water due to sampling techniques. 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 the groundwater due to normal flow patterns, but the well water above the screened section will remain isolated and become stagnant. To safeguard against collecting non-representative stagnant water in a sample, the following approach shall be followed prior to sample acquisition:

1. All monitoring wells shall be purged prior to obtaining a sample. Evacuation of three to five volumes is recommended prior to sampling. 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.

2. For wells that can be purged dry, the well shall be evacuated and allowed to recover to 75% full capacity prior to sample acquisition. If the recovery rate is fairly rapid, evacuation of more than one volume of water is required.

3. For high-yielding monitoring wells which cannot be evacuated to dryness, there is no absolute safeguard against contaminating the sample with stagnant water. One of the following techniques shall be used to minimize this possibility:

   - A submersible pump or the intake line of a surface pump or bailer shall be placed just below the water surface when removing the stagnant water and lowered as the water level drops. Three to five volumes of water shall be removed to provide reasonable assurance that all stagnant water has been evacuated. Once this is accomplished, a bailer or other approved device may be used to collect the sample for analysis.

   - The intake line of the sampling pump (or the submersible pump itself) unless otherwise directed shall be placed near the center of the screened section, and approximately one casing volume of water shall be pumped from the well at a low purge rate, equal to the well's recovery rate (low flow sampling).

Stratification of contaminants may exist in the aquifer. Concentration gradients as a result of mixing and dispersion processes, layers of variable permeability, and the presence of separate-phase product (i.e.,

floating hydrocarbons) may cause stratification. Excessive pumping or improper sampling methods can dilute or increase the contaminant concentrations in the recovered sample compared to what is representative of the integrated water column as it naturally occurs at that point, thus the result is the collection of a non-representative sample.

5.2 Sampling, Monitoring, and Evacuation Equipment

Sample containers shall conform with the guidelines expressed 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, ice, labels and chain-of-custody documents.
- Field tools and instrumentation - Multi-parameters water quality meter 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.
- Pumps
  - Shallow-well pumps: Centrifugal, bladder, suction, or peristaltic pumps with droplines, 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 and inert line with tripod-pulley assembly (if necessary).
- Pails - Plastic, graduated.
- Decontamination solutions - Deionized water, potable water, laboratory detergents, 10% nitric acid solution (as required), 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.

5.3 Calculations of Well Volume

To insure 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. 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 (see SOP SA-6.3):

- Obtain all available information on well construction (location, casing, screens, etc.).
- Determine well or inner casing diameter.
- Measure and record static water level (depth below ground level or top of casing reference point).
- Determine depth of well by sounding using a clean, decontaminated, weighted tape measure.

- Calculate number of linear feet of static water (total depth or length of well pipe minus the depth to static water level).

- Calculate one static well volume in gallons $V = (0.163)(T)(r^2)1$

where:	V	=	Static volume of well in gallons.
	T=		Thickness of water table in the well measured in feet (i.e., linear feet of static water).
	r	=	Inside radius of well casing in inches.
	0.163	=	A constant conversion factor which compensates for the conversion of the casing radius from inches to feet, the conversion of cubic feet to gallons, and pi.

- Per evacuation volumes discussed above, determine the minimum amount to be evacuated before sampling.

5.4 Evacuation of Static Water (Purging)

5.4.1 General

The amount of purging a well shall receive prior to sample collection 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 that 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 the 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, field notebook, or on standardized data sheets.

5.4.2 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. Note that all of these techniques involve equipment which is portable and readily available.

Bailers

Bailers are the simplest evacuation devices used and have many advantages. They generally consist of a length of pipe with a sealed bottom (bucket-type bailer) or, as is more useful and favored, with a ball check-valve at the bottom. An inert line is used to lower the bailer and retrieve the sample.

Advantages of bailers include:

- Few limitations on size and materials used for bailers.
- No external power source 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.
- Transfer of sample may cause aeration.
- Use of bailers is physically demanding, especially in warm temperatures at protection levels above Level D.

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 a flexible tubing, thereby creating suction. This tubing can be dedicated to a well to prevent cross contamination.

These 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 significant loss of dissolved gases and volatile organics.

Air-Lift Samplers

This group of pump samplers uses gas pressure either in the annulus of the well or in a venturi to force the water 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, leading to 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. The operation principles vary and the 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:

- They may have low delivery rates.
- Many models of these pumps are expensive.
- Compressed gas or electric power is needed.
- Sediment in water may cause clogging of the valves or eroding the impellers with some of these pumps.
- Decontamination of internal components can be difficult and time-consuming.

5.5 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
- Dissolved Oxygen (DO)
- Oxidation-Reduction Potential (ORP)

- Turbidity
- Salinity

This section is applicable for use in an onsite groundwater quality monitoring program to be conducted at a hazardous or nonhazardous site. The procedures and equipment described are applicable to groundwater samples and are not, in general, subject to solution interferences from color, turbidity, and colloidal material or suspended matter.

This section provides general information for measuring the parameters listed above with instruments and techniques in common use. Since 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 6.3 for example equipment calibration log.

5.5.1 Measurement of pH

5.5.1.1 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.

Two methods are given for pH measurement: the pH meter and pH indicator paper. The indicator paper is used when only a rough estimate of the pH is required, and the pH meter when a more accurate measurement is needed. The response of a pH meter can be affected to a slight degree by high levels of colloidal or suspended solids, but the effect is usually small and 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, 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.

5.5.1.2 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 pH range hydrion 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 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.

5.5.1.3 Equipment

The following equipment is needed for taking pH measurements:

- Stand-alone portable pH meter, or combination meter (e.g., Horiba U-10), or combination meters 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.
- pH indicator paper, to cover the pH range 2 through 12.
- Manufacturer's operation manual.

5.5.1.4 Measurement Techniques for Field Determination of pH

pH Meter

The following procedure is used for measuring pH with a pH meter (meter standardization is according to manufacturer's instructions):

- Inspect the instrument and batteries prior to initiation of the field effort.
- 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.
- 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).
- Calibrate on a daily use basis (or as recommended by manufacturer) following manufacturer's instructions. Record calibration data on an equipment calibration log sheet.
- 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. This must be clearly noted in the logbook.
- Read and record the pH of the sample. pH shall be recorded to the nearest 0.01 pH unit. Also record the sample temperature.
- Rinse the electrode(s) with deionized water.
- Store the electrode(s) in an appropriate manner when not in use.

Any visual observation of conditions which may interfere with pH measurement, such as oily materials, or turbidity, shall be noted.

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 adequately determined.

5.5.2 Measurement of Specific Conductance

5.5.2.1 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 the 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, since temperature changes, precipitation reactions, and absorption of carbon dioxide from the air all affect the specific conductance. Most conductivity meters in use today display specific conductance (SC); units of milliSiemens per centimeter, which is the conductivity normalized to temperature @ 25°C. This format (SC) is the required units recorded on the groundwater sample log field form (Attachment B).

5.5.2.2 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, while the negatively charged ions migrate toward the positive electrode. Most inorganic acids, bases and salts (such as hydrochloric acid, sodium carbonate, or 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 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.

5.5.2.3 Equipment

The following equipment is needed for taking specific conductance (SC) measurements:

- Stand alone portable conductivity meter, or combination meter (e.g., Horiba U-10), or combination meters 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 which may also be used to monitor salinity and temperature. Probe types and cable lengths vary, so equipment must be obtained to meet the specific requirement of the sampling program.

5.5.2.4 Measurement Techniques for Specific Conductance

The steps involved in taking specific conductance measurements are listed below (standardization is according to manufacturer's instructions):

- Check batteries and calibrate instrument before going into the field.
- Calibrate on a daily use basis (or as recommended by manufacturer), according to the manufacturer's instructions and record all pertinent information on an equipment calibration log sheet. Potassium chloride solutions with a SC closest to the values expected in the field shall be used for calibration.
- Rinse the cell with one or more portions of the sample to be tested or with deionized water.
- Immerse the electrode in the sample and measure the conductivity.
- Read and record the results in a field logbook or sample log sheet.
- Rinse the electrode with deionized water.

If the specific conductance measurements become erratic, recalibrate the instrument and see the manufacturer's instructions for details.

5.5.3 Measurement of Temperature

5.5.3.1 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. Collected water samples may rapidly equilibrate with the temperature of their surroundings.

5.5.3.2 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 dissolved oxygen meters, which 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.

5.5.3.3 Measurement Techniques for Water Temperature

If a thermometer is used to determine the temperature for a water sample:

- Immerse the thermometer in the sample until temperature equilibrium is obtained (1-3 minutes). To avoid the possibility of cross-contamination, the thermometer shall not be inserted into samples which will undergo subsequent chemical analysis.
- Record values in a field logbook or sample log sheet.

If a temperature meter or probe is used, the instrument shall be calibrated according to manufacturer's recommendations.

5.5.4 Measurement of Dissolved Oxygen

5.5.4.1 General

Dissolved oxygen (DO) levels in natural water and wastewater depend on the physical, chemical and biochemical activities in the water body. Conversely, the growth of many aquatic organisms as well as the rate of corrosivity, are dependent on the dissolved oxygen concentration. Thus, analysis for dissolved oxygen is a key test in water pollution and waste treatment process control. If at all possible, DO measurements shall be taken in-situ, since concentration 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 dissolved oxygen meters only. 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, colloidal material or suspended matter.

5.5.4.2 Principles of Equipment Operation

Dissolved oxygen 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.

Since 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, while leaving the surface of the solution undisturbed.

Dissolved oxygen 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, which are not easily depolarized from the indicating electrode. If a gaseous interference is suspected, it shall be noted in the field log book and checked if possible. Temperature variations can also cause interference because probes exhibit temperature sensitivity. Automatic temperature compensation is normally provided by the manufacturer.

5.5.4.3 Equipment

The following equipment is needed to measure dissolved oxygen concentration:

- Stand alone portable dissolved oxygen meter, or combination meter (e.g., Horiba U-10), or combination meters 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.

5.5.4.4 Measurement Techniques for Dissolved Oxygen Determination

Probes differ as to specifics of use. Follow the manufacturer's instructions to obtain an accurate reading. The following general steps shall be used to measure the dissolved oxygen concentration:

- The equipment shall be calibrated and have its batteries checked before going to the field.
- The probe shall be conditioned 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.
- The instrument shall be calibrated in the field according to manufacturer's recommendations or in a freshly air-saturated water sample of known temperature.
- Record all pertinent information on an equipment calibration sheet.
- Rinse the probe with deionized water.
- 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 can be moved up and down.
- Record the dissolved oxygen content and temperature of the sample in a field logbook or sample log sheet.
- Rinse the probe with deionized water.
- 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, since sample handling is not involved. This however, may not always be practical.

Special care shall be taken during sample collection to avoid turbulence which can lead to increased oxygen solubilization and positive test interferences.

5.5.5 Measurement of Oxidation-Reduction Potential

5.5.5.1 General

The oxidation-reduction potential (ORP) provides a measure of the tendency of organic or inorganic compounds 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 oxidized to reduced species in the sample.

5.5.5.2 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 dissolved oxygen, may be correlated with ORP to provide a knowledge of the quality of the solution, water, or wastewater.

5.5.5.3 Equipment

The following equipment is needed for measuring the oxidation-reduction potential of a solution:

- Combination meters 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.

5.5.5.4 Measurement Techniques for Oxidation-Reduction Potential

The following procedure is used for measuring oxidation-reduction potential:

- The equipment shall be checked using the manufacturer's recommended reference solution and have its batteries checked before going to the field.

- Thoroughly rinse the electrode with deionized water.

- If the probe does not respond properly to the recommended reference solution, then verify the sensitivity of the electrodes by noting the change in millivolt reading 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 if the test solution pH is increased. Place the sample in a clean container and agitate the sample. Insert the electrodes and note the ORP drops sharply when the caustic is added (i.e., pH is raised) thus indicating 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.

- Record all pertinent information on an equipment calibration log sheet.

5.5.6 Measurement of Turbidity

5.5.6.1 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, finely divided organic and inorganic matter, soluble colored organic compounds, and microscopic organisms, including plankton.

It is important to obtain a turbidity reading immediately after taking a sample, since irreversible changes in turbidity may occur if the sample is stored too long.

5.5.6.2 Principles of Equipment Operation

Turbidity is measured by the Nephelometric Method. This method 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 turbidmeter. Therefore, nephelometric turbidity units (NTU) based on the formazin preparation will approximate units derived from the candle turbidimeter but will not be identical to them.

5.5.6.3 Equipment

The following equipment is needed for turbidity measurement:

- Light meter (e.g., LaMotte 2020) which calibrates easily using test cells with standards of 0.0 NTUs, and 10 NTUs, or combination meter (e.g., Horiba U-10), 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.

5.5.6.4 Measurement Techniques for Turbidity

The steps involved in taking turbidity measurements utilizing an electrode (e) or light meter (l) are listed below (standardization is according to manufacturer's instructions):

- Check batteries and calibrate instrument before going into the field.
- Check the expiration date (etc.) of the solutions used for field calibration.
- Calibrate on a daily use basis, according to the manufacturer's instructions and record all pertinent information on an equipment calibration log sheet.
- Rinse the electrode with one or more portions of the sample to be tested or with deionized water (applies to "e").
- Fill the light meters glass test cell with ~5 ml of sample, screw on cap, wipe off glass, place test cell in light meter and close the lid (applies to "l").
- Immerse the electrode in the sample and measure the turbidity (applies to "e").
- The reading must be taken immediately as suspended solids will settle over time resulting in a lower, inaccurate turbidity reading.
- Read and record the results in a field logbook or sample log sheet. Include a physical description of the sample, including color, qualitative estimate of turbidity, etc.
- Rinse the electrode or test cell with deionized water.

5.5.7 Measurement of Salinity

5.5.7.1 General

Salinity is a unitless property of industrial and natural waters. It is the measurement of dissolved salts in a given mass of solution. Note: Most field meters determined salinity automatically from conductivity and

temperature. The displayed value will be displayed in either parts per thousand (ppt) or % (e.g., 35 ppt will equal 3.5%).

5.5.7.2 Principles of Equipment Operation

Salinity is determined automatically from the meter's conductivity and temperature readings according to algorithms (found in *Standard methods for the Examination of Water and Wastewater*). Depending on the meter, the results are displayed in either ppt or %. The salinity measurements are carried out in reference to the conductivity of standard seawater (*corrected to S = 35*).

5.5.7.3 Equipment

The following equipment is needed for Salinity measurements:

- Multi-parameter water quality meter capable of measuring conductive, temperature and converting them to salinity (e.g., Horiba U-10 or YSI 600 series).
- Calibration Solution, as specified by the manufacturer.
- Manufacturer's operation manual.

5.5.7.4 Measurement Techniques for Salinity

The steps involved in taking Salinity measurements are listed below (standardization is according to manufacturer's instructions):

- Check batteries and calibrate before going into the field.
- Check the expiration date (etc.) of the solutions used for field calibration.
- Calibrate on a daily use basis, according to the manufacturer's instructions and record all pertinent information on an equipment calibration log sheet.
- Rinse the cell with the sample to be tested.
- Immerse the multi-probe in the sample and measure the salinity. Read and record the results in a field logbook or sample log sheet.
- Rinse the probes with deionized water.

5.6 Sampling

5.6.1 Sampling Plan

The sampling approach consisting of the following, shall be developed as part of the project plan documents which are 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 degrees of contamination between wells is unknown or insignificant, a sampling sequence which 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 as a result of the sampling procedures.

- 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, requirement for split samples, access problems, location of keys, etc.

5.6.2 Sampling Methods

The collection of a groundwater sample consists of the following steps:

1. The site Health & Safety Officer (or designee) will first open the well cap and use volatile organic detection equipment (PID or FID) on the escaping gases at the well head to determine the need for respiratory protection.

2. 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 (see Attachment B); then calculate the fluid volume in the well pipe (as previously described in this SOP).

3. Calculate well volume to be removed as stated in Section 5.3.

4. Select the appropriate purging equipment (see Attachment A). If an electric submersible pump with packer is chosen, go to Step 10.

5. Lower the purging equipment or intake into the well to a short distance below the water level and begin water removal. Collect the purged water and dispose of it in an acceptable manner (as applicable). Lower the purging device, as required, to maintain submergence.

6. Measure the rate of discharge frequently. A graduated bucket or cylinder and stopwatch are most commonly used.

7. Observe the peristaltic pump 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.

8. Purge a minimum of three to five casing volumes before sampling. In low-permeability strata (i.e., if the well is pumped to dryness), one volume will suffice. Purged water shall be collected in a designated container and disposed in an acceptable manner.

9. If sampling using a pump, lower the pump intake to midscreen (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.

10. (For pump and packer assembly 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.

11. In the event that 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.

12. Fill sample containers (preserve and label as described in SOP SA-6.1).

13. Replace the well cap and lock as appropriate. Make sure the well is readily identifiable as the source of the samples.

14. Process sample containers as described in SOP SA-6.1.

15. Decontaminate equipment as described in SOP SA-7.1.

5.7 Low Flow Purging and Sampling

5.7.1 Scope & Application

Low flow purging and sampling techniques are sometimes 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. The minimum stress procedure emphasizes negligible water level drawdown and low pumping rates in order 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, or open interval, length of ten feet or less. Samples obtained are suitable for analyses of common types of groundwater contaminants (volatile and semi-volatile organic compounds, pesticides, PCBs, metals and other inorganic ions [cyanide, chloride, sulfate, etc.]). This procedure is not designed to collect non-aqueous phase liquids samples from wells containing light or dense non-aqueous phase liquids (LNAPLs or DNAPLs), using the low flow pumps.

The 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 NTU and to achieve a water level drawdown of less than 0.3 feet during purging and sampling. If these goals cannot be achieved, sample collection can take place provided the remaining criteria in this procedure are met.

5.7.2 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 may be used to check for and obtain samples of LNAPLs or DNAPLs.

- Tubing - Teflon, Teflon-lined polyethylene, polyethylene, 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, 0.01 foot accuracy, (electronic devices are preferred for tracking water level drawdown during all pumping operations).
- Interface probe, if needed.
- 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 dissolved oxygen, flow-through cell is required. Standards to perform field calibration of instruments.
- Decontamination supplies.
- Logbook(s), and other forms (see Attachments B and C).
- 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 VOCs (volatile organic compounds).

5.7.3 Purging and Sampling Procedure

Open monitoring well, measure head space gases using PID/FID. If there is an indication of off gassing when opening the well, wait 3-5 minutes to permit water level an opportunity to reach equilibrium.

Measure and record the water level immediately prior to placing the pump in the well.

Lower 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 two 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 three feet or less of standing water in the well.

Start with the initial pump rate set at approximately 0.1 liters/minute. Use a graduated cylinder and stopwatch to measure the pumping rate. Adjust pumping rates as necessary to prevent drawdown from exceeding 0.3 feet during purging. If no drawdown is noted, the pump rate may be increased (to a max of 0.4 liters/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 will cease or the well will be pumped to dryness and the well will be allowed to recover before purging continues. Slow recovering wells will be identified and purged at the beginning of the workday. If possible, samples will be colleted from these wells within the same workday and no later than 24 hours after the start of purging.

Measure the well water level using the water level meter every 5 to 10 minutes. Record the well water level on the Low-Flow Purge Data Form (Attachment C).

Record on the Low-Flow Purge Data Form every 5 to 10 minutes the water quality parameters (pH, specific conductance, temperature, turbidity, oxidation-reduction potential, dissolved oxygen and salinity or as specified by the approved site specific work plan) 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.

Measure the flow rate using a graduated cylinder. Remeasure the flow rate any time the pump rate is adjusted.

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.

After stabilization is achieved, sampling can begin when 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:

- pH ±0.2 standard units
- Specific conductance ±10%
- Temperature ±10%
- Turbidity less than 10 NTUs
- Dissolved oxygen ±10%

If the above conditions have still 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.

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.

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: (1) Collect the non-VOCs samples first, then increase the flow rate incrementally until the water column completely fills the tubing, collect the sample and record the new flow rate; (2) 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; (3) 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, collect sample from the narrow diameter tubing.

Prepare samples for shipping as per SOP SA-6.1.

6.0 REFERENCES

American Public Health Association, 1989. Standard Methods for the Examination of Water and Wastewater, 17th Edition, APHA, Washington, D.C.

Barcelona, M. J., J. P. Gibb and R. A. Miller, 1983. A guide to the Selection of Materials for Monitoring Well Construction and Groundwater Sampling. ISWS Contract Report 327, Illinois State Water Survey, Champaign, Illinois.

Johnson Division, UOP, Inc. 1975. Ground Water and Wells, A Reference Book for the Water Well Industry. Johnson Division, UOP, Inc., Saint Paul, Minnesota.

Nielsen, D. M. and G. L. Yeates, 1985. A Comparison of Sampling Mechanisms Available for Small-Diameter Ground Water Monitoring Wells. Ground Water Monitoring Review 5:83-98.

Scalf, M. R., J. F. McNabb, W. J. Dunlap, R. L. Crosby and J. Fryberger, 1981. Manual of Ground Water Sampling Procedures. R. S. Kerr Environmental Research Laboratory, Office of Research and Development, U.S. EPA, Ada, Oklahoma.

U.S. EPA, 1979. Methods for Chemical Analysis of Water and Wastes. EPA-600/4-79-020.

U.S. EPA, 1980. Procedures Manual for Ground Water Monitoring at Solid Waste Disposal Facilities. Office of Solid Waste, United States Environmental Protection Agency, Washington, D.C.

U.S. EPA, 1994. Groundwater Sampling Procedure - Low Flow Purge and Sampling (Draft Final). U.S. Environmental Protection Agency, Region I.

U.S. Geological Survey, 1984. National Handbook of Recommended Methods for Water Data Acquisition, Chapter 5: Chemical and Physical Quality of Water and Sediment. U.S. Department of the Interior, Reston, Virginia.

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:

Date:	Color	pH	S.C.	Temp.	Turbidity	DO	ORP	Other
Time:	Visual	Standard	mS/cm	Degrees C	NTU	mg/l	mV	NA
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
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___

TABLE OF CONTENTS

SECTION **PAGE**

1.0 PURPOSE **2**

2.0 SCOPE **2**

3.0 GLOSSARY **2**

4.0 RESPONSIBILITIES **3**

5.0 PROCEDURES **3**

5.1 SAMPLE CONTAINERS 3
5.2 SAMPLE PRESERVATION 3
5.2.1 Overview 4
5.2.2 Preparation and Addition of Reagents 4
5.3 FIELD FILTRATION 5
5.4 SAMPLE PACKAGING AND SHIPPING 6
5.4.1 Environmental Samples 6

6.0 REFERENCES **7**

ATTACHMENTS

A GENERAL SAMPLE CONTAINER AND PRESERVATION REQUIREMENTS 8
B ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES 9

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 (~~y~~261.30 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 (~~y~~261.20 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 SVOCs and pesticide/PCBs)	(Low	Amber glass	2x2 L or 4x1 L	Cool to 4°C	7 days to extraction; 40 days after extraction
	Extractables SVOCs and pesticide/PCBs)	(Medium	Amber glass	2x2 L or 4x1 L	None	7 days to extraction; 40 days after extraction
Inorganics	Metals	Low	High-density polyethylene	1 L	HNO_3 to pH ≤2	6 months (Hg-28 days)
		Medium	Wide-mouth glass	16 oz.	None	6 months
	Cyanide	Low	High-density polyethylene	1 L	NaOH to pH>12	14 days
	Cyanide	Medium	Wide-mouth glass	16 oz.	None	14 days
Organic/ Inorganic	High Hazard		Wide-mouth glass	8 oz.	None	14 days
SOIL						
Organics (GC&GC/MS)	VOC		EnCore Sampler	(3) 5 g Samplers	Cool to 4°C	48 hours to lab preservation
	Extractables SVOCs and pesticides/PCBs)	(Low	Wide-mouth glass	8 oz.	Cool to 4°C	14 days to extraction; 40 days after extraction
	Extractables SVOCs and pesticides/PCBs)	(Medium	Wide-mouth glass	8 oz.	Cool to 4°C	14 days to extraction; 40 days after extraction
Inorganics	Low/Medium		Wide-mouth glass	8 oz.	Cool to 4°C	6 months (Hg - 28 days) Cyanide (14 days)
Organic/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% $Na_2S_2O_3$.

TABLE OF CONTENTS

SECTION **PAGE**

1.0 PURPOSE **2**

2.0 SCOPE **2**

3.0 GLOSSARY **2**

4.0 RESPONSIBILITIES **2**

5.0 PROCEDURES **2**

5.1 SITE LOGBOOK 2
5.1.1 General 2
5.1.2 Photographs 3
5.2 FIELD NOTEBOOKS 3
5.3 FIELD FORMS 4
5.3.1 Sample Collection, Labeling, Shipment, Request for Analysis, and Field Test Results .. 4
5.3.2 Hydrogeological and Geotechnical Forms 5
5.3.3 Equipment Calibration and Maintenance Form 6
5.4 FIELD REPORTS 6
5.4.1 Daily Activities Report 6
5.4.2 Weekly Status Reports 7

6.0 LISTING OF TETRA TECH NUS FIELD FORMS FOUND ON THE TTNUS INTRANET SITE. HTTP://INTRANET.TTNUS.COM CLICK ON FIELD LOG SHEETS **7**

ATTACHMENTS

A TYPICAL SITE LOGBOOK ENTRY **9**
B SAMPLE LABEL **10**
C CHAIN-OF-CUSTODY RECORD FORM **11**
D CHAIN-OF-CUSTODY SEAL **12**

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

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	CONTAINER TYPE PLASTIC (P) or GLASS (G) / PRESERVATIVE USED / TYPE OF ANALYSIS								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**

TABLE OF CONTENTS

SECTION **PAGE**

1.0 PURPOSE **2**

2.0 SCOPE **2**

3.0 GLOSSARY **2**

4.0 RESPONSIBILITIES **3**

5.0 PROCEDURES **3**

5.1 DECONTAMINATION DESIGN/CONSTRUCTIONS CONSIDERATIONS 3
5.1.1 Temporary Decontamination Pads 3
5.1.2 Decontamination Activities at Drill Rigs/DPT Units 4
5.1.3 Decontamination Activities at Remote Sample Locations 5
5.2 EQUIPMENT DECONTAMINATION PROCEDURES 5
5.2.1 Monitoring Well Sampling Equipment 5
5.2.2 Down-Hole Drilling Equipment 6
5.2.3 Soil/Sediment Sampling Equipment 6
5.3 CONTACT WASTE/MATERIALS 7
5.3.1 Decontamination Solutions 7
5.4 DECONTAMINATION EVALUATION 7

1.0 PURPOSE

Decontamination is the process of removing and/or neutralizing site contaminants that have contacted and/or accumulated on equipment. The objective/purpose of this SOP is intended to protect site personnel, general public, and the sample integrity through the prevention of cross contamination onto unaffected persons or areas. It is further intended through this procedure to provide guidelines regarding the appropriate procedures to be followed when decontaminating drilling equipment, monitoring well materials, chemical sampling equipment and field analytical equipment.

2.0 SCOPE

This procedure applies to all equipment including drilling equipment, heavy equipment, monitoring well materials, as well as chemical sampling and field analytical equipment decontamination that may be used to provide access/acquire environmental samples. Where technologically and economically feasible, single use sealed disposable equipment will be employed to minimize the potential for cross contamination. This procedure also provides general reference information on the control of contaminated materials.

3.0 GLOSSARY

Acid - For decontamination of equipment when sampling for trace levels of inorganics, a 10% solution of nitric acid in deionized water should be used. Due to the leaching ability of nitric acid, it should not be used on stainless steel.

Alconox/Liquinox - A brand of phosphate-free laboratory-grade detergent.

Decontamination Solution - Is a solution selected/identified within 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) - Deionized water is 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 CAP and NCCLS specifications for reagent grade, Type I water.

Potable Water - Tap water used from any municipal water treatment system. Use of an untreated potable water supply is not an acceptable substitute for tap water.

Pressure Washing - Employs high pressure pumps and nozzle configuration to create a high pressure spray of potable water. High pressure spray is employed to remove solids.

Solvent - The solvent of choice is pesticide-grade Isopropanol. Use of other solvents (methanol, acetone, pesticide-grade hexane, or petroleum ether) may be required for particular projects or for a particular purpose (e.g. for the removal of concentrated waste) and must be justified in the project planning documents. As an 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 - This method employs a high pressure spray of heated potable water. This method through the application of heat provides for the removal of various organic/inorganic compounds.

4.0 RESPONSIBILITIES

Project Manager - Responsible for ensuring that all field activities are conducted in accordance with approved project plan(s) requirements.

Field Operations Leader (FOL) - Responsible for the onsite verification that all field activities are performed in compliance with approved Standards Operating Procedures or as otherwise dictated by the approved project plan(s).

Site Health and Safety Officer (SHSO) - The SHSO exercises shared responsibility with the FOL concerning decontamination effectiveness. All equipment arriving on-site (as part of the equipment inspection), leaving the site, moving between locations are 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. Failure to meet these objectives are 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.

5.0 PROCEDURES

The process of decontamination is accomplished through the removal of contaminants, neutralization of contaminants, or the isolation of contaminants. In order to accomplish this activity a level of preparation is required. This includes site preparation, equipment selection, and evaluation of the process. Site contaminant types, concentrations, media types, are primary drivers in the selection of the types of decontamination as well as where it will be conducted. For purposes of this SOP discussion will be provided concerning general environmental investigation procedures.

The decontamination processes are typically employed at:

- Temporary Decontamination Pads/Facilities
- Sample Locations
- Centralized Decontamination Pad/Facilities
- Combination of some or all of the above

The following discussion represents recommended site preparation in support of the decontamination process.

5.1 Decontamination Design/Constructions Considerations

5.1.1 Temporary Decontamination Pads

Temporary decontamination pads are constructed at satellite locations in support of temporary work sites. 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 soils generated during decontamination procedures. Therefore, construction of these pads should take into account the following considerations

- Site Location – The site selected should be within a reasonable distance from the work site but should avoid:

  - Pedestrian/Vehicle thoroughfares
  - Areas where control/custody cannot be maintained
  - Areas where a potential releases may be compounded through access to storm water transport systems, streams or other potentially sensitive areas.
  - Areas potentially contaminated.

- Pad – The pad should be constructed to provide 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.

  - Slope – An adequate slope will be constructed to permit the collection of the water and potentially contaminated soils 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.

  - Sidewalls – The sidewalls should be a minimum of 6-inches in height to provide adequate containment for wash waters and soils. If splash represents a potential problem, splash guards should be constructed to control overspray. Sidewalls maybe constructed of wood, inflatables, sand bags, etc. to permit containment.

  - Liner – Depending on the types of equipment and the decontamination method 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. Achieving the desired thickness maybe achieved through layering lighter constructed materials. 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 maybe applied to provide traction as necessary.

  - Wash/drying Racks – Auger flights, drill/drive rods require racks positioned off of the ground to permit these articles to be washed, drained, and dried while secured from falling during this process. A minimum ground clearance of 2-feet is recommended.

  - Maintenance – The work area should be periodically cleared of standing water, soils, and debris. This action will aid in eliminating slip, trip, and fall hazards. In addition, these articles will reduce potential backsplash and cross contamination. Hoses should be gathered when not in use to eliminate potential tripping hazards.

5.1.2 Decontamination Activities at Drill Rigs/DPT Units

During subsurface sampling activities including drilling and direct push activities decontamination of drive rods, Macro Core Samplers, split spoons, etc. are 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 media. Drying racks will be employed as directed for temporary pads to permit parts to dry and be evaluated prior to use/re-use.

5.1.3 Decontamination Activities at Remote Sample Locations

When sampling at remote locations sampling devices such as trowels, 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.

5.2 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.

5.2.1 Monitoring Well Sampling Equipment

5.2.1.1 Groundwater sampling pumps – This includes pumps inserted into the monitoring well such as Bladder pumps, Whale pumps, Redi-Flo, reusable bailers, etc.

1) Evacuate to the extent possible, any purge water within the pump.

2) Scrub using soap and water and/or steam clean the outside of the pump and tubing, where applicable.

3) Insert the pump and tubing into a clean container of soapy water. Pump a sufficient amount of soapy water through the pump to flush any residual purge water. Once flushed, circulate soapy water through the pump to ensure the internal components are thoroughly flushed.

4) Remove the pump and tubing from the container, rinse external components using tap water. Insert the pump and tubing into a clean container of tap water. Pump a sufficient amount of tap water through the pump to evacuate all of the soapy water (until clear).

5) Rinse equipment with pesticide grade isopropanol

6) Repeat item #4 using deionized water through the hose to flush out the tap water and solvent residue as applicable*.

7) Drain residual deionized water to the extent possible, allow components to air dry.

8) Wrap pump in aluminum foil or a clear clean plastic bag for storage.

5.2.1.2 Electronic Water Level Indicators/Sounders/Tapes

During water level measurements, rinsing with the extracted tape and probe with deionized water and wiping the surface of the extracted tape is acceptable. However, periodic full decontamination should be conducted as indicated below.

---

* - The solvent should be employed when samples contain oil, grease, PAHs, PCBs, and other hard to remove materials. If these are not of primary concern, the solvent step may be omitted. In addition, do not rinse PE, PVC, and associated tubing with solvents.

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.

5.2.1.3 Miscellaneous Equipment

Miscellaneous equipment including analytical equipment (water quality testing equipment) should be cleaned per manufacturer's instructions. This generally includes wiping down the sensor housing and rinsing with tap and deionized water.

Coolers/Shipping Containers employed to ship samples are received from the lab in a variety of conditions from marginal to extremely poor. Coolers should be evaluated prior to use for

- Structural integrity – Coolers missing handles or having breaks within the outer housing should be removed and not used. Notify the laboratory that the risk of shipping samples will not be attempted 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 associated with noticeable odors it should be decontaminated prior to use.

  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 notify the laboratory to provide a replacement unit.

5.2.2 Down-Hole 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. This procedure is to be employed prior to initiating the drilling/sampling activity, then between locations.

1) Remove all soils to the extent possible using shovels, scrapers, etc. to remove loose soils.
2) Through a combination of scrubbing using soap and water and/or steam cleaning remove visible dirt/soils.
3) Rinse with tap water.
4) Rinse equipment with pesticide grade isopropanol
5) To the extent possible allow components to air dry.
6) Wrap or cover equipment in clear plastic until it is time to be used.

5.2.3 Soil/Sediment Sampling Equipment

This consists of 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 soils to the extent possible.

2) Through a combination of scrubbing using soap and water and/or steam cleaning remove visible dirt/soils.

3) Rinse with tap water.

4) Rinse equipment with pesticide grade isopropanol

5) Rinse with deionized water

6) To the extent possible allow components to air dry.

7) If the device is to be used immediately, screen with a PID/FID to insure all solvents (if they were used) and trace contaminants have been adequately removed.

8) Once these devices have been dried wrap in aluminum foil for storage until it is time to be used.

5.3 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.) broken sample containers.

With the exception of the broken glass, single use articles should be cleaned (washed and rinsed) of visible materials and disposed of as normal refuse. The exception to this rule is that extremely soiled materials that cannot be cleaned should be containerized for disposal in accordance with applicable federal state and local regulations.

5.3.1 Decontamination Solutions

All waste decontamination solutions and rinses must be assumed to contain the hazardous chemicals associated with the site unless there are analytical or other data to the contrary. The waste solution volumes could vary from a few gallons to several hundred gallons in cases where large equipment required cleaning.

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. These containers must be appropriately labeled.

5.4 Decontamination Evaluation

Determining the effectiveness of the decontamination process will be accomplished in the following manner

- Visual Evaluation – A visual evaluation will be conducted to insure the removal of particulate matter. This will be done to insure that the washing/rinsing process is working as intended.

- Instrument Screening – A PID and/or an FID should be used to evaluate the presence of the contaminants or solvents used in the cleaning process. The air intake of the instrument should be passed over the article to be evaluated. A positive detection requires a repeat the decontamination process. It should be noted that the instrument scan is only viable if the contaminants are detectable within the instruments capabilities.

- 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:

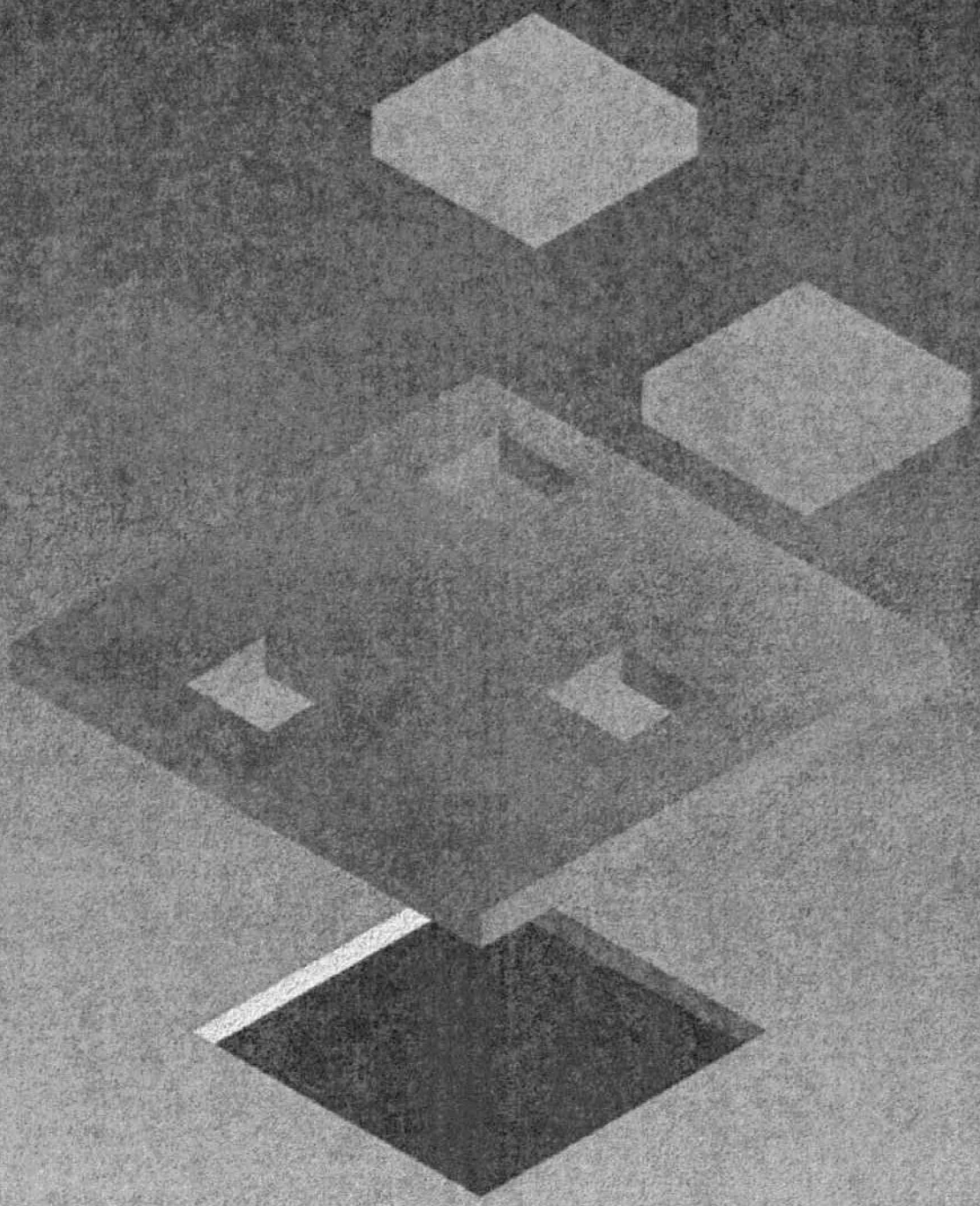
- Per decontamination method
- Per disposable article/Batch number of disposable articles

It is recommended that an initial rinsate sample be collected early in the project to ensure that the decontamination process is functioning properly and in an effort to avoid using a contaminated batch of single use articles. It is recommended that a follow up sample be collected during the execution of the project to insure those conditions do not change. Lastly, rinsate samples collection may be driven by types of and/or contaminant levels. Hard 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.

New Jersey
Department of Environmental Protection

Field Sampling Procedures Manual

August 2005



Temporary well points and direct push samplers typically have short screens. Therefore, the sampler will focus on a narrow zone in the aquifer. Examples of the use of temporary well points are those used to characterize a groundwater contaminant plume through vertical profiling using screening-level data. They can also be used to construct "transects" whereby temporary well points are placed at selected intervals perpendicular to the direction of plume movement. This focused approach allows for refined decision making when placing permanent monitor wells and plume delineation. In addition, advancement in direct push technology now allows for the generation of extended geophysical and hydrogeological data once strictly associated with monitor well installation and observation.

The American Society for Testing Materials discusses general technique issues in ASTM D6001-96, *Direct Push Water Sampling for Geoenvironmental Investigations*. Additional information can be found on the Internet at the following USEPA and vendor URLs:

http://www.epa.gov/superfund/programs/dfa/dirtech.htm,

http://www.epa.gov/swerust1/pubs/esa-ch5.pdf

http://www.geoprobe.com/products/tools/tools_menu.htm, and

http://www.geoinsightonline.com

http://www.ams-samplers.com/main.shtm?PageName=welcome.shtm

6.9.2.2 Low-Flow Purging and Sampling

6.9.2.2.1 Method Summary and Application

The purpose of Low-Flow Purging and Sampling (LFPS) is to collect groundwater samples from monitor wells that are representative of ambient groundwater conditions in the aquifer. This is accomplished by setting the intake velocity of the sampling pump to a flow rate that limits drawdown inside the well. LFPS has three primary benefits. First, it minimizes disturbance of sediment in the bottom of the well, thereby producing a sample with low turbidity. Second, LFPS minimizes aeration of the groundwater during sample collection. Third, the amount of groundwater purged from a well is usually reduced as compared to conventional groundwater purging and sampling methods.

Because the method allows collection of groundwater samples with low turbidity, it was originally used for collecting samples for inorganics analysis. The method typically allows the collection of samples for total metals analysis and eliminates the need to filter the samples for dissolved metals analysis. In addition, since the method minimizes aeration of the samples, it can be used to collect samples for analysis of volatile and semi-volatile organic compounds (VOCs and SVOCs), provided that appropriate pumps are used in sample collection, as discussed below.

Advantages of LFPS are:

- Groundwater samples tend to be more representative of actual aquifer conditions with respect to mobile contaminants and turbidity
- It causes minimal disturbance of the formation adjacent to the screened interval
- It is generally less prone to sampling variability compared to other groundwater sampling techniques (e.g., bailers)
- Smaller purge volumes and associated disposal expense

- Increased sample consistency from dedicated systems and reproducibility of data due to reduced operator variability

Disadvantages of LFPS are:

- Misconceptions regarding reduced purging and sampling time
- Sampling from non-dedicated systems requires greater set-up time
- Sampling from dedicated systems requires higher initial capital expenses
- Increased technical complexity
- Increased training needs for sampling personnel
- Attractiveness of advantages may lead to improper and inconsistent application
- Typically not a "first round" sampling option
- Not recommended for wells with long screen intervals unless multiple samples are collected

6.9.2.2.2 Introduction

The following procedures are specific to LFPS of monitor wells in New Jersey. These procedures were developed in consideration of the USEPA-Region I guidance document dated July 30, 1996 (http://www.epa.gov/region01/measure/well/lowflow8.pdf) and the USEPA-Region II guidance document dated March 16, 1998 (http://www.epa.gov/Region2/desa/hsw/lowflow.txt). In addition, the U.S. Geological Survey's (USGS) Techniques of Water-Resources Investigations, Book 9, National Field Manual for the Collection of Water-Quality Data was consulted (http://water.usgs.gov/owq/FieldManual/). The reader is encouraged to review these guidance documents prior to performing LFPS. The procedures provided in the USEPA and USGS guidance must be followed except where they differ from the information provided below. Finally, three forms are provided herein to assist the sampler in recording low-flow stabilization data, calibration information and pump intake depth placement. They can be found on pages 109, 110, and 111 respectively.

6.9.2.2.3 Low Flow Policy

In the event that a responsible party is conducting a Remedial Investigation without Departmental oversight, submittal of a sampling plan is not required. However, it is *highly recommended* that the responsible party seek approval for any deviations from this guidance prior to conducting LFPS. In the event that a responsible party decides to use LFPS without submitting a sampling plan and receiving approval, it must be recognized that any deviations from this guidance may result in rejection of the data. In addition, when submitting the results of the LFPS event, the responsible party must include specific details of the LFPS techniques used which demonstrate that they were consistent with the guidance specified below. The responsible party shall also provide adequate rationale justifying any deviations from this guidance whether or not they were previously approved by the Department.

It is also Departmental policy that LFPS is not an acceptable method for any wells with screened or open borehole intervals greater than 5 feet in length **unless:** 1) multiple locations at five-foot intervals along the screen/borehole are sampled, or 2) the data quality objectives (DQOs) warrant sampling a specific zone (e.g., the shallow water table to investigate the potential for vapor intrusion inside a building) or

specific zones where sufficient geophysical (e.g., heat-pulse flowmeter, caliper and temperature logs, etc.) and hydrogeological information (e.g., tracer tests) or other evidence (e.g., stained soils or fractures noted on boring logs) that **clearly** identifies the depth(s) at which contaminants are entering the well screen or open borehole.

Once the collection of multiple samples (vertical profiling) in a well has been completed, long-term sampling of the well may require LFPS at fewer depth intervals, or even just one depth interval, depending on the data quality objectives of the sampling and the types of contamination present in the groundwater (e.g., LNAPL, DNAPL, etc).

6.9.2.2.4 Laboratory Certification (N.J.A.C. 7:18)

N.J.A.C. 7:18 requires that any environmental laboratory* submitting analytical data to the Department, regardless of quality level, must be certified by the Office of Quality Assurance. This applies to those firms using LFPS instruments associated with the "analyze immediately" category of water quality indicator parameters (WQIPs) including pH, temperature, and dissolved oxygen. Regardless of whether or not the equipment in question is rented or privately owned the requirement for certification can not be ignored. All certification documentation must accompany the instrument into the field and accompany all WQIP data submitted to the Department. (*Environmental laboratory is defined as any laboratory, facility, consulting firm, government or private agency, business entity or other person that the Department has authorized, pursuant to N.J.A.C. 7:18, to perform analysis in accordance with the procedures of a given analytical method using a particular technique as set forth in a certain methods reference document and to report the results from the analysis of environmental samples in compliance with a Departmental regulatory program).

6.9.2.2.5 Specific LFPS Considerations

6.9.2.2.5.1 Pump Intake Location

When LFPS is performed correctly, the data being collected should be a snapshot of a narrow zone along a length of well screen or fracture in an open borehole. For these reasons, it is important to place the pump intake in the zone of highest contaminant concentration or contaminant flux along the screened/open-hole interval. This is particularly important in wells constructed with more than 5 feet of well screen.

Information to be considered when selecting the pump intake depth should include: 1) evidence of soil/sediment contamination from boring logs; 2) soil/sediment sampling analytical results; 3) vertical profiles of groundwater and soil contamination developed from direct-push sampling and field-screening techniques; and; 4) lithology/stratigraphy, particularly the permeability of the aquifer materials.

Typically, the most permeable zones are selected for the pump intake location since the majority of contaminant mass will be transported through them, particularly as the plume migrates downgradient of the source area. Identification of these zones may be made from borehole geophysical data, (e.g., resistivity, fluid conductance, or natural gamma logging, etc.) and hydraulic conductivity data or grain-size analyses. The use of a series of passive-diffusion-bag samplers in a well may also help to identify the zone of highest VOC contamination. The physical/chemical behavior of the contaminants of concern should be considered when determining

the pump intake depth. For example, gasoline-related contaminants may be present near the water table while chlorinated VOCs may be present deeper in the aquifer. If a well is contaminated by both types of contaminants, both may need to be sampled, each from a discrete sampling interval.

As discussed above, LFPS is not an option in wells with screened intervals that exceed 5 feet in length, **unless** multiple sample locations at five-foot intervals along the screen/borehole are investigated. Monitor wells screened across zones of significant geologic heterogeneity or open boreholes in fractured rock may be subject to significant vertical flow. Under those conditions, use of packers to isolate specific zones should be considered.

6.9.2.2.5.2 Water Quality Indicator Parameters (WQIPs)

For groundwater investigations in New Jersey utilizing LFPS, the following parameters must be measured in order to determine when well stability has been achieved prior to sampling. Their respective measurements must fall within the stated range for three consecutive readings. If the anticipated "third" reading of any individual parameter does not fall within the stated range, then the process to achieve three consecutive readings for that parameter must be restarted. If, after four hours, stability has not been achieved for the parameters listed below, follow the recommendations in Section 3 below.

Water Level Drawdown ..... < 0.3 ft*
pH ..... ± 0.1 unit
Specific Conductance ..... ± 3%
Temperature ..... ± 3%
Dissolved Oxygen ..... ± 10%
Turbidity ..... ± 10% for values greater than 1 NTU
ORP/Eh ..... ± 10 millivolts

* During pump start-up, drawdown may exceed the 0.3-ft target and then recover as flow-rate adjustments are made.

In wells with short screens (i.e., 5 to 10 ft long) or when sampling for gasoline constituents at the water table, it is much more important to limit the drawdown to less than 0.3 ft, for example, than a well with 15 ft of screen being sampled for metals only with the pump intake set in a permeable zone 5 ft or more below the water table. When sampling groundwater for VOCs and SVOCs, aerating the water by allowing it to cascade down the inside of the well should be avoided. Therefore, drawdown should not expose the screen more than 0.3 ft below the static water level in the well.

Measurements should be taken once every 5 to 6 minutes. This interval is based upon the time it takes for purge water to replace one flow-through-cell volume (generally 250 ml) and the time it takes to measure and record the data. If the purge rate decreases or if the flow cell volume is increased, the time required for purge water replacement will increase. Forms at the end of this document should be used to record drawdown and the WQIPs.

WQIP measurements must be collected in a manner that will insure integrity of the data being collected. To insure consistency of the data, consideration of the following must be made: 1) tubing diameter, length, and material of construction;

2) flow-through cell design, capacity, decontamination, and "purge-train" set-up; 3) pump selection and plumbing fittings; 4) calibration of flow-through cell probes; 5) purge rate; and, 6) water-level-measurement technique.

6.9.2.2.5.3 Purge Volume vs. Stabilization Time

In some cases, it may take considerable time to achieve stabilization of the WQIPs. In other cases, they may never stabilize. However, as provided in USEPA guidance, the following options are available if stability has not been achieved after **FOUR** hours of purging: 1) continue purging until stabilization occurs, no matter how long it takes; 2) discontinue purging, do not collect a sample and document the attempts to reach stabilization; or 3) discontinue purging, collect a sample and document the attempts to reach stabilization. In situations where WQIPs do not stabilize, the sampler must document that LFPS could not be performed and document in the report how the samples were collected.

While every effort should be taken to assure that all of the WQIPs stabilize prior to sample collection, one should keep in mind that the stabilization of some WQIPs may be more difficult to achieve than others. Also, achieving stabilization of some WQIPs may be more important with respect to some contaminant types (e.g., metals versus VOCs, etc.) than others. For example, total metals concentrations tend to increase with increasing turbidity due to sorption of metals on solids in the water. Similarly, VOC concentrations may be affected by dissolved oxygen (DO) concentrations (i.e., whether the groundwater is aerobic or anaerobic). In addition to providing information on the effectiveness of LFPS, collection of accurate DO data also aids in the evaluation of monitored natural attenuation (MNA) of VOC plumes. Similarly, temperature data can provide useful information regarding the sampling method. For example, temperature increases resulting from dissipation of heat generated by the submersible pump or from exposure of the tubing to excessive heat at the ground surface can have a significant impact on VOC concentrations in water samples.

If, for whatever reason, a WQIP is not accurately measured during the monitoring process or a certain WQIP does not stabilize, and that particular WQIP **is not** significant with respect to the type of contaminant of concern, sample collection may still proceed. For example, if DO data do not stabilize but all of the other WQIPs including drawdown and turbidity stabilize and samples will be collected for metals only, then the samples may be collected. However, any WQIPs that are affected by field conditions or instrument malfunction, must be discussed in the text of the report in order to alert the end-user of potential data bias. If questions arise regarding when stabilization occurs, the sampler should contact the Department's assigned case manager for the site, if any, either prior to (preferably) or when performing LFPS.

6.9.2.2.5.4 Tubing

The inside diameter (ID) of tubing should be no greater than three-eighths of an inch (3/8-in). Quarter-inch (1/4-in) tubing is preferred. Larger tubing diameters reduce flow velocity resulting in a corresponding increase of pump speeds to maintain flow. Increased pump speed will, in turn, elevate the potential for turbulent flow across the screened interval and this may affect the quality of the water being sampled. Conversely, any reduction in flow velocity may allow air to become

trapped in the tubing, which may ultimately affect air-sensitive parameters or allow particulates to settle, which may affect turbidity values.

The length of tubing, from the top of the well casing to the flow-through chamber, should be the shortest length manageable. Attention to this detail will help ensure that: 1) exposure to ambient temperature, direct sunlight, and bubble formation are kept to a minimum, and 2) deposited solids or air bubbles will less likely be trapped in tubing bends and re-mobilized after accidental movement. Occurrence of any one or combination of these factors can cause variations in WQIP measurements, which could increase stabilization time. Therefore, tubing must be completely full of water at all times.

If the sampling plan calls for multiple sample locations within the well screen, sampling should proceed from the top location to the bottom location. This will require that additional tubing be coiled at the surface to allow for pump relocation to the next deeper sampling location. In these instances, the coiled tubing must be protected from ambient conditions and the ground surface, in order to avoid impact to the WQIPs and sample data.

The tubing's material of construction must be either Teflon® or Teflon®-lined polyethylene up to the flow-through cell. This is consistent with collection of any groundwater sample. Tubing downstream of the flow cell may be constructed of a lower-quality, more flexible material. However, when sampling for metals analysis only, the tubing may be constructed of flexible polypropylene or polyethylene.

Tubing "reuse" is not recommended when sampling well to well since decontamination of tubing is difficult and time consuming. If tubing is to be reused, it must undergo a rigorous decontamination procedure, which must include a hot water wash/hot air drying process. In addition to the hot water wash/hot air drying, separate decontamination solutions of acetone and nitric acid may have to be pumped through the tubing for 15 minutes, followed by copious amounts of distilled, deionized water rinses. The cost of labor associated with decontamination, including the special handling of cleaning solvents and acid, often exceeds the cost of simply discarding the old tubing and using new tubing for each well. If a decision is made to reuse tubing, then one of the following requirements in the USGS, *Water-Quality National Field Manual,* must be considered: 1) Collect additional field blanks if VOC concentrations in the last sample collected through the tubing are greater than 500 µg/L, or 2) The tubing should be replaced, rather than cleaned, if VOC concentrations in the last sample exceed 700 µg/L.

6.9.2.2.5.5 Flow-Through Cell

Typical flow-through cell design is not complicated and almost all on the market today have common shared features. Cells should be transparent in order to "see" the physical condition of the purge water or air bubbles passing through the system. Highly turbid or iron bacteria-laden water can be visually monitored for change as the purge progresses. The cell must be sealed against unwanted exposure to the atmosphere, thus insuring accurate measurement of air-sensitive parameters (dissolved oxygen, pH, etc.). The total capacity of the cell must be small (300-1,000 ml) in order to maintain a desirable turnover rate of water coming into the cell to ensure real-time data integrity. The in-line design must allow for purge

water to enter the flow cell from a bottom port and exit at the top. The discharge may be fitted with a check valve.

Upon initial pump startup, it is good practice to not connect the pump discharge line to the flow-through cell. This will allow the sampler time to monitor drawdown, stabilize the flow rate and prevent fowling of probes by bacteria, sediment, or NAPL. Once drawdown measurements indicate that the flow rate has been controlled and a few minutes (<10) have been allowed to clear any unwanted material, the pump discharge line can then be connected to the flow cell.

Flow cell decontamination is important, not only to reduce the potential for cross contamination, but also to ensure data integrity and consistent instrument performance. The cell and probes should be rinsed with distilled/deionized water between each monitor well as accumulation of suspended material may impact probe performance. If they are exposed to contaminants, use a mild detergent or laboratory glassware cleaning solution. Flow cell exposure to high levels of contamination may damage probes and require their repair by the manufacturer. Since LFPS is NOT normally a first-round sampling option, knowledge of contaminant levels will generally be known prior to the cell's exposure to purge water.

The location of the flow cell or cells in relation to the sample port is critical. Samples for turbidity measurement, general chemistry and laboratory analysis must be collected ahead of the flow cell. When two cells are used in series, the dissolved oxygen probe must be located in the first cell.

Set up the flow-through cell in a location which will cause minimal fluctuation of the flow rate due to elevation changes in the sample tubing as the tubing is disconnected from the cell prior to sample collection. It is also important to locate the flow-through cell as close as possible to the well head in order to minimize the length of tubing needed between the well head and flow-through cell. The flow-through cell must be protected from ambient conditions and the ground surface. See Figure 6.6.

6.9.2.2.5.6 Pump Selection

Pumps used for monitoring WQIPs must be submersible, positive-displacement pumps. Examples of acceptable positive-displacement pumps include bladder, variable-speed submersible-centrifugal, reciprocating-piston, progressive-cavity, and gear pumps. The pump discharge must be fitted appropriately to receive either 1/4 or 3/8-inch inside-diameter (ID) Teflon® or Teflon®-lined polyethylene tubing.

Peristaltic pumps are suction-lift pumps, which can create a negative pressure gradient. Therefore, their use is not appropriate when collecting groundwater samples for analysis of organic compounds. However, peristaltic pumps may be used for the collection of groundwater samples for analysis of inorganic compounds. It should be kept in mind, however, that sampling with peristaltic pumps may affect the stabilization of some WQIPs including dissolved oxygen, pH and redox potential. Since these WQIPs can be affected by the peristaltic pump, this pump should not be used when these data are to be used to evaluate the effectiveness of Monitored Natural Attenuation of groundwater.

Figure 6.6 Illustration of Flow Cell with stand. (Photograph by J. Schoenleber)

Two basic collection scenarios have a bearing on pump selection. These include: 1) a permanently installed pump system, or 2) a portable (well-to-well) pump installation. Bladder pumps can be used for either scenario, however, only those with disposable bladders and easily cleaned parts are suitable when sampling on a well-to-well basis. Variable-speed submersible-centrifugal pumps, gear or progressive-cavity pumps can be used for either scenario as long as they are constructed of easy to clean stainless steel/Teflon® parts. Pumps constructed with impellers, helicoils, or gears, which are difficult to clean or are constructed of unacceptable plastic parts, are not suitable for sampling. In addition, when conducting LFPS on a portable basis, the power or gas supply line should be isolated from the sample tubing. Power supply and sample tubing lines that form a single unit do not allow for easy decontamination and are not recommended.

6.9.2.2.5.7 Plumbing Fittings

A check valve should be incorporated into the tubing train or flow cell discharge to eliminate accidental drainage and subsequent aeration of the flow cell. More importantly, a check valve will prevent a back-surge of purged water being reintroduced at the screen interval of the well should the power source or pump experience mechanical failure. A back-surge of purge water into the screened interval of the well may result in variability of the WQIPs and create analytical bias. In order to avoid the need to decontaminate the check valve, it may be placed on the discharge side of the flow cell or installed immediately above the pump discharge. Some flow-through cells have check valves built into the unit. By design, bladder pumps also have a check valve built into their construction.

A ¼- or 3/8-inch ID barbed "T" or "Y" fitting, placed ahead of the flow cell, may be used to establish the line which will receive a needle valve for turbidity, general

chemistry and analytical sample collection. The "T" or "Y" fitting used should be constructed of Teflon® or stainless steel and decontaminated between each use, if used for analytical samples. The fitting may be constructed of polyethylene and decontaminated between each use if it is only used to sample for turbidity and general chemistry parameters. If analytical samples are collected through the "T" or "Y" fitting and needle valve, then those parts must be incorporated into the field blank collection technique.

When collecting a sample at the port ahead of the flow cell, a flow control valve (stainless-steel needle valve [preferred] or stainless steel/Teflon ball valve [optional]) must be used to prevent backpressure and air bubbles from forming in the tubing (see http://water.usgs.gov/owq/FieldManual/chap4_rpt.pdf, page 84). The "needle valve" offers versatility as it can be used for collection of turbidity, general chemistry **and** analytical samples. It can be used with Teflon® tubing and can be used to control sample flow rate because the design significantly reduces any backpressure gradient. Like all other sampling equipment, the "needle valve" must be decontaminated before use at any well. See Figure 6.7.

Figure 6.7 Closeup of Needle Valve. (Photograph by D. Dibblee)

6.9.2.2.5.8 Calibration of Probes

> Calibration of the probes used to monitor water quality indicator parameters must take place **in the field prior** to the day's events. The Office of Quality Assurance must certify the *environmental laboratory* (see Section 6.9.2.2.4) using probes for pH, dissolved oxygen and temperature measurement.

There are no exceptions to these rules. Probe calibration is critical to the accurate and precise measurement of WQIPs.

For warranty purposes, **all** manufacturers' instructions for proper care and calibration must be followed. Solutions for probe calibration must be held to the temperature of the liquid (groundwater) being measured as temperature correlation is critical in calculating conductivity, dissolved oxygen and pH. Tables and equations to compensate for the difference between ambient groundwater and calibration solution temperature are sometimes provided in the operating manuals or with the calibration solutions. Some instruments are designed with internal features to

compensate for this difference in temperature. The respective difference between calibration of conductivity and specific conductivity requires compensation for groundwater temperature at the time of calibration vs. solution temperature adjusted to 25°C at the time of calibration. For dissolved oxygen, the flow cell itself must be maintained at the temperature of groundwater during calibration. All efforts made to account for proper temperature control of solutions during calibration must be reported to the end user. All steps must be recorded in the field notes. No sampling shall commence until all instruments are calibrated and operating properly. See the "Tips" section below for further discussion on Temperature of Calibration Solutions.

6.9.2.2.5.9 Water Level Measurements

The depth to the top of the water column must be recorded prior to pump installation and/or prior to purging. If the **total** depth of the well needs to be determined (e.g., to verify the correct well designation and/or to determine if silt has accumulated in the bottom of a well), it should be measured at least 48 hours prior to sample collection or after the sample has been collected and the pump removed. Total depth measurements must never be taken immediately before purging as this may cause the re-suspension of solids in the well and prolong the purge time.

Once the initial water-level measurement has been recorded and the pump installed, suspend the water-level probe in the well at the point at which drawdown is equivalent to a 0.3-foot drop. Record water levels simultaneously with WQIP measurements once every five minutes.

Water-level-measurement devices, which may impart some disturbance to the water column (i.e., stainless steel "popper" or coated tape), are not acceptable.

6.9.2.2.5.10 Pump Installation

LFPS pump installation can be divided into two general collection scenarios: permanent and portable (well to well). Permanent pump installation is the most desirable. Among other advantages are improved consistency in data acquisition and reduced long-term labor, preparation and material costs. However, permanent installation is more typically associated with long-term monitoring due to the high initial capital investment required.

The more common practice is to use a pump on a portable or well-to-well basis. While initial capital investment is comparatively less than that of a permanent installation, this practice requires close attention to quality control aspects of pump selection, preparation and decontamination.

Once pumps have been properly decontaminated and fitted with appropriate tubing, installation of the pump can begin. Ideally, pumps should be installed 24 to 48 hours prior to initiation of purging. However, this is not always practical, especially when site security can not be guaranteed. In addition, wells constructed with flush-mount casing are difficult to protect from storm water or infiltration of other contaminants during the extended period monitor wells are open.

Pumps must be installed in such a manner as to insure any disturbance in the well is kept to an absolute minimum. Once pumps reach the top of the water column, their descent should proceed very slowly through the water column. The actual

level where the pump intake is to be suspended must be predetermined. Under no circumstance should the pump make contact with, or be "bounced" off, the bottom of the well.

One helpful method to insure proper intake location is to accurately measure and pre-cut the tubing for each individual well prior to site activity. A mark can be made on the tubing, which coincides, with the top of the well. Cutting the tubing off-site in a controlled setting is most desirable. Tubing can be wiped down with paper towels, moistened with distilled/ deionized water, labeled and then sealed into plastic bags until needed. If this practice is used, be sure to allow enough tubing to account for the distance from the top of the well casing to the flow cell.

6.9.2.2.5.11 Purge Rates

Control over the purge rate is one of the most critical aspects of this technique. Once the pump is set within the screened interval at the desired location, a clean electronic water-level-monitoring device is lowered approximately 0.3 ft into the water column. Start the pump at a speed that results in a flow rate in the range of 100 to 500 ml/min. Pump the initial purge water to waste in order to prevent any fouling of the flow-through cell. *With the pump running,* connect the tubing to the cell. Make sure that all air is purged from the tubing and flow cell as the system fills with purge water. For LFPS, the pump speed must remain constant such that flow rates never exceed 500 ml/min and, once stabilized, the flow rate must not be varied, even during sample collection. If drawdown continues to exceed 0.3 ft., reduce the pump speed until the drawdown has stabilized but do not adjust pump speed to a flow rate below 100 ml/min. Flow rates below this level may induce pump stalling and undo the effort to reach stabilization. If drawdown does not come under control at 100 ml/min, then a field decision should be rendered as to how far to allow drawdown to continue until sample collection. At no time should evacuation allow any portion of the well screen to be exposed (for wells screened below the water table) or bring the well to dryness.

Adjustments to pump speed are best made during the first 15 minutes. Once a "feel" for the purge rate is obtained, begin recording well stabilization indicators. Any significant change to purge rates after this time may negatively impact well stabilization measurements.

Purge rates are best monitored by measuring the flow from the discharge side of the flow cell with a graduated cylinder. Record all of the required WQIPs once every 5 minutes. Once stability has been attained and recorded, begin sample collection

6.9.2.2.5.12 Sampling

Once WQIPs have stabilized, or a 4-hour time decision has been rendered, sampling can proceed. Do not adjust the flow rate; maintain the same pumping rate during sampling that was used to purge the well. Collect the sample directly from the needle valve at the sample port. The needle valve allows for sample collection with significantly reduced backpressure and turbulence and offers the best means for sample collection without affecting water quality. It also allows for monitoring using the flow-through cell during sample collection, thereby allowing a final WQIP measurement to be recorded immediately after sample collection. This is the

preferred method, especially if volatile organic compounds are the parameters of concern. Any exceptions to this technique must first be approved in writing from the NJDEP on a case-by-case basis before commencing sampling operations.

If higher than expected water temperatures are being observed, evaluate whether the submersible pump is overheating. If the pump motor is not suspected, check the system for any exposure to direct sunlight, especially during warmer periods of the year.

6.9.2.2.5.13 Pump Decontamination

The pump forms one of the two key elements of sampling equipment (tubing is the other). The importance of proper pump decontamination is especially true when pumps are rented and utilized on a well-to-well basis. Never assume that rented pumps have been thoroughly cleaned. **Pumps constructed with plastic parts, or sealed inner workings that are inaccessible to direct handling are not an option for LFPS well-to-well consideration because of their limited ability to be decontaminated thoroughly**.

Most bladder pumps can not be easily decontaminated in the field due to their unique construction. For that reason, bladder pumps are not employed on a well-to-well basis **unless** they are constructed with easy to clean parts and *disposable* bladders. Bladder pumps are best suited for dedicated (permanently installed) scenarios. Another popular pump, the variable-speed, 2-inch diameter submersible, is more adaptable for well-to-well sampling; however, close attention to decontamination is warranted. One manufacturer, Grundfos®, clearly states in the operational handbook that the pump must be completely disassembled, including removal of the motor shaft from the stator housing, and all components within the impeller housing (See Figure 6.8). Care must be taken upon reassembly to insure that the cavity housing the motor shaft is *completely* refilled with distilled/deionized water. Care must also be taken with this pump during periods of cold weather to avoid freezing of the coolant water. Proper decontamination not only helps to ensure more reliable data; it also prolongs the life of any pump.

6.9.2.2.5.14 Field Blank Collection

When employing LFPS techniques, collection of the field blank must follow the same general rules for all groundwater sampling equipment. This includes the requirement that "all" sampling equipment, which comes in contact with the sample, must also come into contact with the field blank water. To overcome some of the difficulties that manual field blank collection through the inside of a pumping system creates, the following procedure is strongly recommended. Fill a 1000-ml decontaminated, graduated glass cylinder with method blank water supplied by the laboratory performing the analysis. Place a properly decontaminated pump into the graduated cylinder with sample tubing and plumbing fittings attached. Activate the pump and collect the required field blank samples. As the water is removed from the cylinder, replace it with additional method blank water. This procedure will require that the laboratory supply larger volumes of field blank water i.e., bulk water in liter or 4-liter containers. The traditional requirement that field blank water be supplied in the same identical containers as the sample being collected can not be practically satisfied when using LFSP. The identical bottle-to-bottle field blank requirement is waived for this sampling technique procedure only.

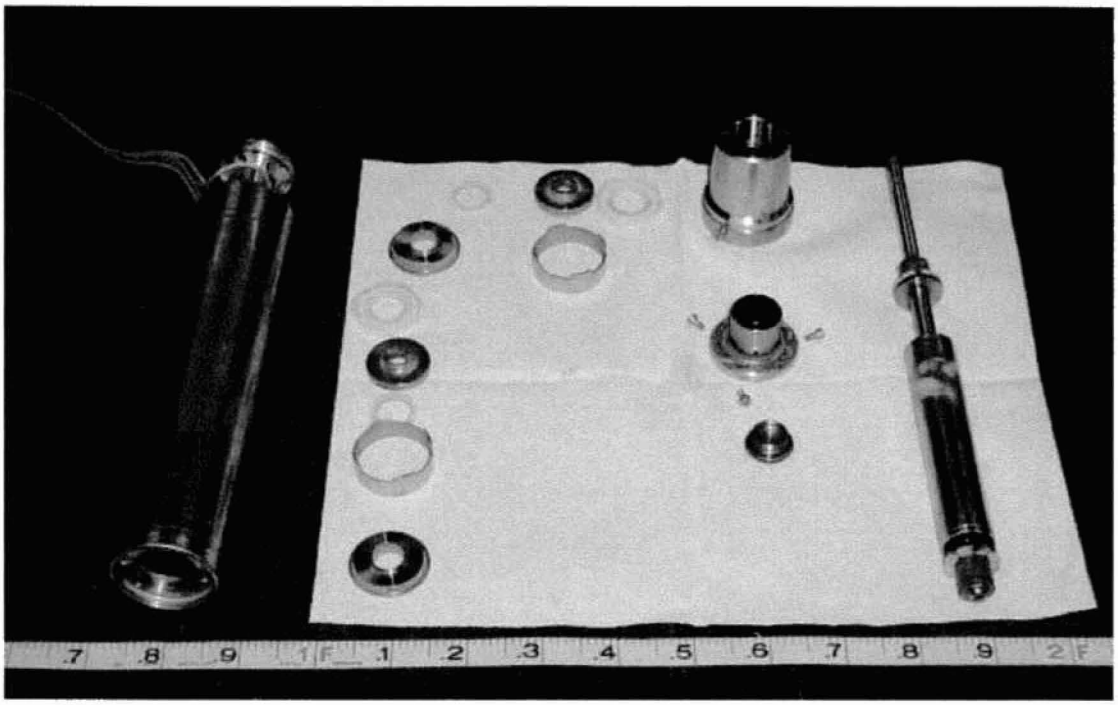


Figure 6.8 Grundfos® Pump being prepared for decontamination (Photograph by J. Schoenleber)

6.9.2.2.6 Tips

6.9.2.2.6.1 Temperature Measurement and Submersible Pumps

Variable-speed submersible pumps such as the Grundfos Redi Flo 2[a] pump use water to cool the motor during operation. Sometimes, reduced flow rates may result in insufficient cooling of the motor and may elevate the temperature of the water to a point where it may begin to affect sample integrity. If the pump is used in low-yielding, two (2)- or four (4)- inch-diameter wells, temperature increases that do not stabilize may result. If this is observed, a field decision must be made to either discontinue or continue with LFPS. If all other WQIPs have stabilized, then collecting the sample and qualifying the water-quality data accordingly may be acceptable. If the temperature increase continues and eventually exceeds 40% of the initial recorded temperature (Celsius) and other WQIPs have not stabilized, sampling should be discontinued. Turning the pump off and on to control overheating is not acceptable. Always keep in mind that elevated temperature has a direct relationship with dissolved oxygen, specific conductance and, to a lesser degree, pH measurement. Higher temperatures may also reduce the concentrations of volatile organic compounds in groundwater samples due to their relatively high Henry's Law constants. If sampling with submersible pumps continues to result in elevated water temperature, other sampling alternatives should be discussed with the appropriate regulatory program.

When using some submersible pumps in large-diameter wells (six inch and greater), overheating of the motor, followed by mechanical shutdown and possible motor damage, may occur. This is the result of water being drawn to the pump intake in a more horizontal flow pattern which diminishes the design feature that

normally moves cool water vertically across the motor (stator) housing. The use of specially designed shrouds may overcome this condition.

6.9.2.2.6.2 Control of Pump Speed

In order to achieve the high turning speeds, low-speed startup torque is generally lacking in some submersible pumps including the Grundfos[a] Redi Flo 2 pump. When attempting to control initial drawdown and/or sample flow rates, it is possible for the pump to cease pumping. Then, if a check valve has been installed, the pump may not have enough torque to overcome the head pressure when attempting to restart it. Sometimes, turning the pump to the highest speeds will overcome this situation or sometimes the pump may have to be pulled from the well and reinstalled. Neither of these corrective measures is conducive to LFPS. To avoid this scenario, make sure the control box comes equipped with a "ten turn pot" frequency adjustment knob. This will allow significantly greater control over pump speeds and the risk of losing pump flow will be reduced.

6.9.2.2.6.3 pH

Monitoring for stabilization of pH in groundwater is relatively straightforward and rarely requires serious troubleshooting. When calibrating for pH, do a two-point calibration, at a minimum. The calibration range should bracket the anticipated pH. If the pH is unknown, then a three-point calibration must be made. The temperature of the buffer solutions should be as close to the temperature of the groundwater as possible. If the probe does not calibrate properly, check to make sure that the probe's electrical contact points are dry. As with preventative maintenance of any probe, make sure that the pH probe is rinsed with distilled/deionized water between use and cleaned periodically per the manufacturer's specifications. Overnight storage generally requires placement of the probe into a 2-molar (M) solution of potassium chloride. This solution may cause an unwanted build up of salt, therefore, frequent rinsing may be necessary.

6.9.2.2.6.4 Temperature of Calibration Solutions

Correct field measurement of dissolved oxygen, conductivity and pH requires tight control over calibration solution temperature. Proper calibration calls for solution temperatures of these parameters to be the same as the groundwater being measured (http://water.usgs.gov/owq/FieldManual/Chapter6/6.2.1.html#HDR6.2.1.CAL1). This may be difficult to achieve when field sampling well to well as groundwater temperature can vary between wells based on depth, local setting (asphalt vs. open field) and other atmospheric and hydrogeological factors. In addition, it is logistically difficult to bring solutions to groundwater temperature at the point of pump intake without first installing the pump, collecting purge water and allowing sufficient time to bring calibration solutions to appropriate temperatures.

For the purposes of LFPS in New Jersey, calibration solution temperatures and the flow-through cell itself must be maintained at approximately 54° F (12° C ± 2° C) during calibration. When ambient conditions warrant, this will require the suspension of the solutions and flow-through cell in a container/bucket of water at the aforementioned temperature. When calibrating for dissolved oxygen, always make sure the cell is vented to the atmosphere by attaching short pieces of tubing to the inlet and outlet fittings while the cell is submerged.

LOW FLOW SAMPLING
DATA SHEET

SHEET ____ OF ____

SITE: _____
DATE: _____
WEATHER: _____

CONSULTING FIRM: _____
FIELD PERSONNEL: _____

MONITOR WELL #: _____ WELL DEPTH: _____
WELL PERMIT #: _____ WELL DIAMETER: _____ inches

SCREENED/OPEN INTERVAL: _____

PID/FID READINGS (ppm):
BACKGROUND: _____
BENEATH OUTER CAP: _____
BENEATH INNER CAP: _____

PUMP INTAKE DEPTH: _____ ft below TOC
DEPTH TO WATER BEFORE PUMP INSTALLATION: _____ ft below TOC
MAKE/MODEL OF PUMP: _____

TIME	PURGING	SAMPLING	pH (pH units)		SPECIFIC CONDUCTIVITY (mS/cm)		REDOX POTENTIAL (mv)		DISSOLVED OXYGEN (mg/l)		TURBIDITY (NTU)		TEMPERATURE (degrees C)		PUMPING RATE (ml/min)	DEPTH TO WATER (ft below TOC)
			READING	CHANGE*	READING	CHANGE*	READING	CHANGE*	READING	CHANGE*	READING	CHANGE*	READING	CHANGE*		
				NA		NA		NA		NA		NA		NA		

COMMENTS:

*INDICATOR PARAMETERS HAVE STABILIZED WHEN 3 CONSECUTIVE READINGS ARE WITHIN: ±0.1 for pH; ±3% for Specific Conductivity and Temperature; ±10 mv for Redox Potential; and ±10% for Dissolved Oxygen and Turbidity.

Field Instrument and Calibration Data Sheet

Site: _____

Field Personnel: _____

Date: _____ Start Time: _____ Stop: _____

	Meter (make/model)	Probe
DO		
pH		
Spec. Cond.		
ORP		
Turbidity		

Dissolved Oxygen		Turbidity Standard	Turbidity Reading	ORP	
Water Temp		D.I. Water		Standard Temp.	
Baro. Pres.				Standard Conc.	
Saturation				Initial Reading	
Init. Mtr. Rd.				Meter reset to	
Mtr. reset to					
O_2 Satur. %					

Specific Conductance	Conc.	Initial Reading	Reset to	Temperature	Lot # and Exp. Date
Standard #1					
Standard #2					
Standard #3					
Standard #4					

pH Calibration Buffer	Temperature	Initial Reading	mV	Meter Reset To	Lot # and Exp. Date
4					
7					
10					

Monitor Well Information in Support of Pump Intake Depth Placement

Sheet _____ of _____

Monitor Well	Screened/Open Interval			Intake Depth(s) (ft TOC)	Rationale for Pump Intake Depth(s)*
	Top (ft TOC)	Bottom (ft TOC)	Length (ft)		

ft = feet

ft TOC = feet below Top of Casing

* If necessary, attach supporting documentation (e.g., boring logs, construction diagrams, soil sampling data, etc.).

During the purge phase, record the difference between the stabilized temperature and the temperature of the calibration solutions. This information must be presented to the end user. If the sampling event is extended for two or more days, appropriate adjustments can then be made to more accurately reflect the groundwater temperature during calibration.

6.9.2.3 Low-flow Purging and Sampling for Low-Yielding Wells

The principal focus of water supply well installation is well-yield. In contrast, the principal focus of monitor well installation is water quality; well-yield is of secondary importance. In an attempt to locate and delineate ground water contamination, monitor wells are frequently installed in low-yielding water-bearing zones.

Low-yield wells present challenges with respect to representative ground water sample collection. The removal of water by bailers draws down the water-level in the well in slug-type increments. Peristaltic pumps draw water out of the well by vacuum (negative pressure) which may result in degassing and VOC loss. The operation of variable-speed submersible pumps at low-flow rates may result in heating of the sample as it flows around and through the pump, which may also lead to degassing and VOC loss.

Wells that yield less than 0.1 Lpm (100ml/min) frequently incur significant drawdown during well purging. If drawdown occurs across the screened interval or open borehole of a well, VOC loss may result. The increased stress on a well caused by significant drawdown may also result in an increase in well water turbidity. NJDEP recognizes that the use of sampling methods for LFPS discussed above may be impractical if drawdown cannot be limited. In an effort to facilitate the collection of a representative ground water sample from low-yielding wells, NJDEP will allow special sampling procedures to be used. This may include sample collection without regard to monitoring water quality indicator parameters associated with well stabilization.

At a minimum, water quality data, well construction data, water-level data, and accurate well-yield data for each low yielding well will need to be submitted to the Department prior to the formulation of an acceptable sampling procedure. Since sample collection may begin almost as soon as purging is initiated, it is imperative that the exact interval where the sample will be collected along the screen be predetermined. Aside from the considerations for monitoring drawdown and WQIP, all other aspects of LFPS may be found in the section above. The owner of the well shall also propose possible explanations for the low-yield of the well(s). Once the aforementioned information has been received, the Department will work with the well owner to formulate an acceptable sampling plan. The sampling plans will be approved on a case-by-case basis and will be well specific. Implementation of any special sampling procedure shall not be undertaken without prior NJDEP approval.

6.9.2.4 Volume-Averaged Purging and Sample Collection

Application of water supply well construction practices to remedial investigations often resulted in monitor wells with open boreholes or screens of up to twenty-five feet in length. Previous NJDEP guidance specified that the standard well purging procedure was to calculate, in gallons, one volume of standing water within a monitor well and purge three to five times that amount. Over the past decade, much information has shown that this procedure may have produced data of questionable value. Today, the general consensus is that ground water contaminants in a heterogeneous subsurface often flow within thin or narrow zones of higher permeability. Purging large volumes of water from wells with long screens situated in a heterogeneous aquifer

APPENDIX B

LABORATORY STANDARD OPERATING PROCEDURES

LABORATORY ACCREDITATION BUREAU

Certificate Number L2223

Certificate of Accreditation

Accredited to DoD ELAP and ISO/IEC 17025:2005

Katahdin Analytical Services, Inc.

600 Technology Way
Scarborough, ME 04074

has met the requirements set forth in L-A-B's policies and procedures, all requirements of ISO/IEC 17025:2005 "General Requirements for the competence of Testing and Calibration Laboratories" and the U.S. Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP).*

The accredited lab has demonstrated technical competence to a defined "Scope of Accreditation" and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated 8 January 2009).

Accreditation Granted through: November 4, 2012

R. Douglas Leonard, Jr., Managing Director
Laboratory Accreditation Bureau
Presented the 4th of November, 2009

*See the laboratory's Scope of Accreditation for details of the DoD ELAP requirements
Laboratory Accreditation Bureau is found to be in compliance with ISO/IEC 17011:2004 and recognized by ILAC (International Laboratory Accreditation Cooperation) and NACLA (National Cooperation for Laboratory Accreditation).

LABORATORY ACCREDITATION BUREAU

Certificate # L2223

Scope of Accreditation
For
Katahdin Analytical Services

600 Technology Way
Scarborough, ME 04074
Leslie Dimond
1- 207-874-2400

In recognition of a successful assessment to ISO/IEC 17025:2005 and the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in the DoD Quality Systems Manual for Environmental Laboratories (DoD QSM v4.1) based on the National Environmental Laboratory Accreditation Conference Chapter 5 Quality Systems Standard (NELAC Voted Revision June 5, 2003), accreditation is granted to Katahdin Analytical Services to perform the following tests:

Accreditation granted through: **November 4, 2012**

Testing - Environmental

Non-Potable Water

Technology	Method	Analyte
GC/ECD	608 / 8081A,B/ SOM01.2	4 4`-DDD
GC/ECD	608 / 8081A,B/ SOM01.2	4 4`-DDE
GC/ECD	608 / 8081A,B / SOM01.2	4 4`-DDT
GC/ECD	608 / 8081A,B / SOM01.2	Aldrin
GC/ECD	608 / 8081A,B / SOM01.2	alpha-BHC (alpha-Hexachlorocyclohexane)
GC/ECD	8081A,B / SOM01.2	Alpha-Chlordane
GC/ECD	608 / 8081A,B / SOM01.2	beta-BHC (beta-Hexachlorocyclohexane)
GC/ECD	608 /8081A,B	Chlordane (tech.)
GC/ECD	608 / 8081A,B / SOM01.2	delta-BHC
GC/ECD	608 / 8081A,B / SOM01.2	Dieldrin
GC/ECD	608 / 8081A,B / SOM01.2	Endosulfan I
GC/ECD	608 / 8081A,B / SOM01.2	Endosulfan II
GC/ECD	608 / 8081A,B / SOM01.2	Endosulfan sulfate
GC/ECD	608 / 8081A,B / SOM01.2	Endrin
GC/ECD	608 / 8081A,B / SOM01.2	Endrin aldehyde
GC/ECD	8081A,B / SOM01.2	Endrin Ketone
GC/ECD	8081A,B / SOM01.2	gamma-BHC (Lindane gamma-Hexachlorocyclohexane)
GC/ECD	608 / 8081A,B / SOM01.2	Heptachlor
GC/ECD	608 / 8081A,B / SOM01.2	Heptachlor epoxide

LABORATORY ACCREDITATION BUREAU

Certificate # L2223

Non-Potable Water

Technology	Method	Analyte
GC/ECD	8081A,B / SOM01.2	Methoxychlor
GC/ECD	608 / 8081A,B / SOM01.2	Toxaphene (Chlorinated camphene)
GC/ECD	608 / 8082/8082A / SOM01.2	Aroclor-1221 (PCB-1221)
GC/ECD	608 / 8082/8082A / SOM01.2	Aroclor-1232 (PCB-1232)
GC/ECD	608 / 8082/8082A / SOM01.2	Aroclor-1242 (PCB-1242)
GC/ECD	608 / 8082/8082A / SOM01.2	Aroclor-1248 (PCB-1248)
GC/ECD	608 / 8082/8082A / SOM01.2	Aroclor-1254 (PCB-1254)
GC/ECD	608 / 8082/8082A / SOM01.2	Aroclor-1260 (PCB-1260)
GC/ECD	8082/8082A	Aroclor-1262 (PCB-1262)
GC/ECD	8082/8082A	Aroclor-1268 (PCB-1268)
GC/ECD	8082/8082A	2 2' 3 3' 4 4' 5 5' 6-Nonachlorobiphenyl (BZ 206)
GC/ECD	8082/8082A	2 2' 3 3' 4 4' 5 6-Octachlorobiphenyl (BZ 195)
GC/ECD	8082/8082A	2 2' 3 3' 4 4' 5-Heptachlorobiphenyl (BZ 170)
GC/ECD	8082/8082A	2 2' 3 3' 4 4'-Hexachlorobiphenyl (BZ 128)
GC/ECD	8082/8082A	2 2' 3 4 4' 5 5'-Heptachlorobiphenyl (BZ 180)
GC/ECD	8082/8082A	2 2' 3 4 4' 5' 6-Heptachlorobiphenyl (BZ 183)
GC/ECD	8082/8082A	2 2' 3 4 4' 5'-Hexachlorobiphenyl (BZ 138)
GC/ECD	8082/8082A	2 2' 3 4 4' 6 6'-Heptachlorobiphenyl (BZ 184)
GC/ECD	8082/8082A	2 2' 3 4' 5 5' 6-Heptachlorobiphenyl (BZ 187)
GC/ECD	8082/8082A	2 2' 3 4 5'-Pentachlorobiphenyl (BZ 87)
GC/ECD	8082/8082A	2 2' 3 5'-Tetrachlorobiphenyl (BZ 44)
GC/ECD	8082/8082A	2 2' 4 4' 5 5'-Hexachlorobiphenyl (BZ 153)
GC/ECD	8082/8082A	2 2' 4 5 5'-Pentachlorobiphenyl (BZ 101)
GC/ECD	8082/8082A	2 2' 4' 5-Tetrachlorobiphenyl (BZ 49)
GC/ECD	8082/8082A	2 2' 5 5'-Tetrachlorobiphenyl (BZ 52)
GC/ECD	8082/8082A	2 2' 5-Trichlorobiphenyl (BZ 18)
GC/ECD	8082/8082A	2 3 3' 4 4' 5-Hexachlorobiphenyl (BZ 156)
GC/ECD	8082/8082A	2 3 3' 4 4' 5'-Hexachlorobiphenyl (BZ 157)
GC/ECD	8082/8082A	2 3 3' 4 4'-Pentachlorobiphenyl (BZ 105)
GC/ECD	8082/8082A	2 3 3’ 4 4’ 5 5’-Heptachlorobiphenyl (BZ 189)
GC/ECD	8082/8082A	2 3' 4 4' 5 5'-Hexachlorobiphenyl (BZ 167)
GC/ECD	8082/8082A	2 3' 4 4' 5-Pentachlorobiphenyl (BZ 118)
GC/ECD	8082/8082A	2 3' 4 4'5-Pentachlorobiphenyl (BZ 123)
GC/ECD	8082/8082A	2 3' 4 4'-Tetrachlorobiphenyl (BZ 66)
GC/ECD	8082/8082A	2 3’ 4 4’ 5-Pentachlorobiphenyl (BZ 114)
GC/ECD	8082/8082A	2 4 4'-Trichlorobiphenyl (BZ 28)
GC/ECD	8082/8082A	2 4'-Dichlorobiphenyl (BZ 8)
GC/ECD	8082/8082A	3 3' 4 4' 5 5'-Hexachlorobiphenyl (BZ 169)
GC/ECD	8082/8082A	3 3' 4 4' 5-Pentachlorobiphenyl (BZ 126)
GC/ECD	8082/8082A	3 3' 4 4'-Tetrachlorobiphenyl (BZ 77)
GC/ECD	8082/8082A	3 4 4’ 5-Tetrachlorobiphenyl (BZ 81)
GC/ECD	8082/8082A	Decachlorobiphenyl (BZ 209)
GC/ECD	8151A	2 4 5-T
GC/ECD	8151A	2 4-D
GC/ECD	8151A	2 4-DB

LABORATORY ACCREDITATION BUREAU

Certificate # L2223

Non-Potable Water

Technology	Method	Analyte
GC/ECD	8151A	Dalapon
GC/ECD	8151A	Dicamba
GC/ECD	8151A	Dichloroprop
GC/ECD	8151A	DInoseb
GC/ECD	8151A	MCPA
GC/ECD	8151A	MCPP
GC/ECD	8151A	Pentachlorophenol
GC/ECD	8151A	Silvex (2 4 5-TP)
GC/FID	8015B/C	Diesel range organics (DRO)
GC/FID	8015B/C	Gasoline range organics (GRO)
GC/FID	8011 / 504	1 2-Dibromoethane (EDB)
GC/FID	8011 / 504	1 2-Dibromo-3-chloropropane
GC/FID	RSK-175	Methane Ethane Ethene
GC/MS	8260B,C / 524.2	1 1 1 2-Tetrachloroethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 1 1-Trichloroethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 1 2 2-Tetrachloroethane
GC/MS	SOM01.2	1 1 2-Trichloro-1 2 2-trifluoroethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 1 2-Trichloroethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 1-Dichloroethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 1-Dichloroethene
GC/MS	8260B,C / 524.2	1 1-Dichloropropene
GC/MS	8260B,C / SOM01.2 / 524.2	1 2 3-Trichlorobenzene
GC/MS	8260B,C / 524.2	1 2 3-Trichloropropane
GC/MS	8260B,C / SOM01.2 / 524.2	1 2 4-Trichlorobenzene
GC/MS	8260B,C / 524.2	1 2 4-Trimethylbenzene
GC/MS	8260B,C / SOM01.2 / 524.2	1 2-Dibromo-3-chloropropane
GC/MS	8260B,C / SOM01.2 / 524.2	1 2-Dibromoethane (EDB)
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 2-Dichlorobenzene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 2-Dichloroethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 2-Dichloropropane
GC/MS	8260B,C / 524.2	1 3 5-Trimethylbenzene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 3-Dichlorobenzene
GC/MS	8260B,C / 524.2	1 3-Dichloropropane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	1 4-Dichlorobenzene
GC/MS	8260B,C / SOM01.2	1 4-Dioxane
GC/MS	8260B,C / 524.2	2 2-Dichloropropane

LABORATORY ACCREDITATION BUREAU

Certificate # L2223

Non-Potable Water

Technology	Method	Analyte
GC/MS	8260B,C / SOM01.2 / 524.2	2-Butanone
GC/MS	624 / 8260B,C	2-Chloroethyl vinyl ether
GC/MS	8260B,C / 524.2	2-Chlorotoluene
GC/MS	8260B,C / SOM01.2 / 524.2	2-Hexanone
GC/MS	8260B,C / 524.2	4-Chlorotoluene
GC/MS	8260B,C / SOM01.2 / 524.2	4-Methyl-2-pentanone
GC/MS	8260B,C / SOM01.2 / 524.2	Acetone
GC/MS	8260B,C	Acetonitrile
GC/MS	624 / 8260B,C	Acrolein
GC/MS	624 / 8260B,C / 524.2	Acrylonitrile
GC/MS	8260B,C / 524.2	Allyl chloride
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Benzene
GC/MS	8260B,C / 524.2	Bromobenzene
GC/MS	8260B,C / SOM01.2 / 524.2	Bromochloromethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Bromodichloromethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Bromoform
GC/MS	8260B,C / SOM01.2 / 524.2	Carbon disulfide
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Carbon tetrachloride
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Chlorobenzene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Chloroethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Chloroform
GC/MS	8260B,C	Chloroprene
GC/MS	8260B,C / SOM01.2 / 524.2	cis-1 2-Dichloroethene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	cis-1 3-Dichloropropene
GC/MS	SOM01.2	Cyclohexane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Dibromochloromethane
GC/MS	8260B,C / 524.2	Dibromomethane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Dichlorodifluoromethane
GC/MS	8260B,C / 524.2	Diethyl ether
GC/MS	8260B,C	Di-isopropylether
GC/MS	8260B,C / 524.2	Ethyl methacrylate
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Ethylbenzene
GC/MS	8260B,C	Ethyl-t-butylether
GC/MS	8260B,C / 524.2	Hexachlorobutadiene
GC/MS	8260B,C	Iodomethane
GC/MS	8260B,C	Isobutyl alcohol

Non-Potable Water

Technology	Method	Analyte
GC/MS	8260B,C / SOM01.2 / 524.2	Isopropyl benzene
GC/MS	8260B,C / SOM01.2 / 524.2	m p-xylenes
GC/MS	8260B,C / 524.2	Methacrylonitrile
GC/MS	SOM01.2	Methyl acetate
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Methyl bromide (Bromomethane)
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Methyl chloride (Chloromethane)
GC/MS	8260B,C / 524.2	Methyl methacrylate
GC/MS	8260B,C / SOM01.2 / 524.2	Methyl tert-butyl ether
GC/MS	SOM01.2	Methylcyclohexane
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Methylene chloride
GC/MS	8260B,C / 524.2	Naphthalene
GC/MS	8260B,C / 524.2	n-Butylbenzene
Gc/ms	8260B,C / 524.2	n-Propylbenzene
GC/MS	8260B,C / SOM01.2 / 524.2	o-Xylene
GC/MS	8260B,C / 524.2	p-Isopropyltoluene
GC/MS	8260B,C / 524.2	Propionitrile
GC/MS	8260B,C / 524.2	sec-butylbenzene
GC/MS	8260B,C / SOM01.2 / 524.2	Styrene
GC/MS	8260B,C	t-Amylmethylether
GC/MS	8260B,C / 524.2	tert-Butyl alcohol
GC/MS	8260B,C	tert-Butylbenzene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Tetrachloroethene (Perchloroethylene)
GC/MS	8260B,C / 524.2	Tetrahydrofuran
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Toluene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	trans-1 2-Dichloroethylene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	trans-1 3-Dichloropropylene
GC/MS	8260B,C / 524.2	trans-1 4-Dichloro-2-butuene
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Trichloroethene (Trichloroethylene)
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Trichlorofluoromethane
GC/MS	8260B,C	Vinyl acetate
GC/MS	624 / 8260B,C / SOM01.2 / 524.2	Vinyl chloride
GC/MS	624 8260B,C	Xylene
GC/MS	8270C,D / SOM01.2	1 2 4 5-Tetrachlorobenzene
GC/MS	625 / 8270C,D / SOM01.2	1 2 4-Trichlorobenzene
GC/MS	625 / 8270C,D / SOM01.2	1 2-Dichlorobenzene
GC/MS	8270C,D	1 2-Diphenylhydrazine

LABORATORY ACCREDITATION BUREAU

Certificate # L2223

Non-Potable Water

Technology	Method	Analyte
GC/MS	8270C,D	1 3 5-Trinitrobenzene
GC/MS	625 / 8270C,D / SOM01.2	1 3-Dichlorobenzene
GC/MS	8270C,D	1 3-Dinitrobenzene
GC/MS	625 / 8270C,D / SOM01.2	1 4-Dichlorobenzene
GC/MS	8270C,D	1 4-Dioxane
GC/MS	8270C,D	1 4-Naphthoquinone
GC/MS	8270C,D	1 4-Phenylenediamine
GC/MS	8270C,D	1-Naphthylamine
GC/MS	8270C,D / SOM01.2	2 3 4 6-Tetrachlorophenol
GC/MS	8270C,D / SOM01.2	2 4 5-Trochlorophenol
GC/MS	625 / 8270C,D / SOM01.2	2 4 6-Trichlorophenol
GC/MS	625 / 8270C,D / SOM01.2	2 4-Dichlorophenol
GC/MS	625 / 8270C,D / SOM01.2	2 4-Dimethylphenol
GC/MS	625 / 8270C,D / SOM01.2	2 4-Dinitrophenol
GC/MS	625 / 8270C,D / SOM01.2	2 4-Dinitrotoluene (2 4-DNT)
GC/MS	8270C,D	2 6-Dichlorophenol
GC/MS	625 / 8270C,D / SOM01.2	2 6-Dinitrotoluene (2 6-DNT)
GC/MS	8270C,D	2-Acetylaminofluorene
GC/MS	625 / 8270C,D / SOM01.2	2-Chloronaphthalene
GC/MS	625 / 8270C,D / SOM01.2	2-Chlorophenol
GC/MS	625 / 8270C,D / SOM01.2	2-Methyl-4 6-dinitrophenol
GC/MS	8270C,D / SOM01.2	2-Methylnaphthalene
GC/MS	8270C,D / SOM01.2	2-Methylphenol
GC/MS	8270C,D	2-Naphthylamine
GC/MS	8270C,D	2-Nitroaniline
GC/MS	625 / 8270C,D / SOM01.2	2-Nitrophenol
GC/MS	8270C,D	2-Picoline
GC/MS	625 / 8270C,D / SOM01.2	3 3'-Dichlorobenzidine
GC/MS	8270C,D	3 3'-Dimethylbenzidine
GC/MS	8270C,D	3-Methylcholanthrene
GC/MS	8270C,D / SOM01.2	3-Nitroaniline
GC/MS	8270C,D	4-Aminobiphenyl
GC/MS	625 / 8270C,D / SOM01.2	4-Bromophenyl phenyl ether
GC/MS	625 / 8270C,D / SOM01.2	4-Chloro-3-methylphenol
GC/MS	8270C,D / SOM01.2	4-Chloroaniline
GC/MS	625 / 8270C,D / SOM01.2	4-Chlorophenyl phenylether
GC/MS	8270C,D	4-Dimethyl aminoazobenzene
GC/MS	8270C,D / SOM01.2	4-Methylphenol
GC/MS	8270C,D / SOM01.2	4-Nitroaniline
GC/MS	625 / 8270C,D / SOM01.2	4-Nitrophenol
GC/MS	8270C,D	5-Nitro-o-toluidine
GC/MS	8270C,D	7,12-Dimethylphenethylamine
GC/MS	8270C,D	a a-Dimethylphenethylamine
GC/MS	625 / 8270C,D / SOM01.2	Acenaphthene
GC/MS	625 / 8270C,D / SOM01.2	Acenaphthylene

Non-Potable Water

Technology	Method	Analyte
GC/MS	8270C,D / SOM01.2	Acetophenone
GC/MS	8270C,D	Aniline
GC/MS	625 / 8270C,D / SOM01.2	Anthracene
GC/MS	8270C,D	Aramite
GC/MS	8270C,D / SOM01.2	Atrazine
GC/MS	SOM01.2	Benzaldehyde
GC/MS	625 / 8270C,D	Benzidine
GC/MS	625 / 8270C,D / SOM01.2	Benzo(a)anthracene
GC/MS	625 / 8270C,D / SOM01.2	Benzo(a)pyrene
GC/MS	625 / 8270C,D / SOM01.2	Benzo(b)fluoranthene
GC/MS	625 / 8270C,D / SOM01.2	Benzo(g h i)perylene
GC/MS	625 / 8270C,D / SOM01.2	Benzo(k)fluoranthene
GC/MS	8270C,D	Benzoic Acid
GC/MS	8270C,D	Benzyl alcohol
GC/MS	8270C,D / SOM01.2	Biphenyl
GC/MS	625 / 8270C,D / SOM01.2	bis(2-Chloroethoxy)methane
GC/MS	625 / 8270C,D / SOM01.2	bis(2-Chloroethyl) ether
GC/MS	625 / 8270C,D / SOM01.2	bis(2-Chloroisopropyl) ether (2 2`-Oxybis(1-chloropropane))
GC/MS	625 / 8270C,D / SOM01.2	bis(2-Ethylhexyl) phthalate (DEHP)
GC/MS	625 / 8270C,D / SOM01.2	Butyl benzyl phthalate
GC/MS	SOM01.2	Caprolactam
GC/MS	8270C,D / SOM01.2	Carbazole
GC/MS	8270C,D	Chlorobenzilate
GC/MS	625 / 8270C,D / SOM01.2	Chrysene
GC/MS	8270C,D	Diallate
GC/MS	625 / 8270C,D / SOM01.2	Dibenz(a h)anthracene
GC/MS	8270C,D / SOM01.2	Dibenzofuran
GC/MS	625 / 8270C,D / SOM01.2	Diethyl phthalate
GC/MS	8270C,D	Dimethoate
GC/MS	625 / 8270C,D / SOM01.2	Dimethyl phthalate
GC/MS	625 / 8270C,D / SOM01.2	Di-n-butyl phthalate
GC/MS	625 / 8270C,D / SOM01.2	Di-n-octyl phthalate
GC/MS	8270C,D	Ethyl methanesulfonate
GC/MS	8270C,D	Famfur
GC/MS	625 / 8270C,D / SOM01.2	Fluoranthene
GC/MS	625 / 8270C,D / SOM01.2	Fluorene
GC/MS	625 / 8270C,D / SOM01.2	Hexachlorobenzene
GC/MS	625 / 8270C,D / SOM01.2	Hexachlorobutadiene
GC/MS	625 / 8270C,D / SOM01.2	Hexachlorocyclopentadiene
GC/MS	625 / 8270C,D / SOM01.2	Hexachloroethane
GC/MS	8270C,D	Hexachloropropene
GC/MS	625 / 8270C,D / SOM01.2	Indeno(1 2 3-cd)pyrene
GC/MS	8270C,D	Isodrin
GC/MS	625 / 8270C,D / SOM01.2	Isophorone

Non-Potable Water

Technology	Method	Analyte
GC/MS	8270C,D	Isosafrole
GC/MS	8270C,D	Methapyriline
GC/MS	8270C,D	Methy methanesulfonate
GC/MS	8270C,D	Methyl parathion
GC/MS	625 / 8270C,D / SOM01.2	Naphthalene
GC/MS	625 / 8270C,D / SOM01.2	Nitrobenzene
GC/MS	8270C,D	Nitroquinoline-1-oxide
GC/MS	8270C,D	n-Nitrosodiethylamine
GC/MS	625 / 8270C,D / SOM01.2	n-Nitrosodimethylamine
GC/MS	8270C,D	n-Nitroso-di-n-butylamine
GC/MS	625 / 8270C,D / SOM01.2	n-Nitrosodi-n-propylamine
GC/MS	625 / 8270C,D / SOM01.2	n-Nitrosodiphenylamine
GC/MS	8270C,D	n-Nitrosomethylethylamine
GC/MS	8270C,D	n-Nitrosomorpholine
GC/MS	8270C,D	n-Nitrosopiperidine
GC/MS	8270C,D	n-Nitrosopyrrolidine
GC/MS	8270C,D	o o o-Triethyl phosphorothioate
GC/MS	8270C,D	o-Toluidine
GC/MS	8270C,D	Pentachlorobenzene
GC/MS	8270C,D	Pentachloronitrobenzene
GC/MS	625 / 8270C,D / SOM01.2	Pentachlorophenol
GC/MS	8270C,D	Phenacetin
GC/MS	625 / 8270C,D / SOM01.2	Phenanthrene
GC/MS	625 / 8270C,D / SOM01.2	Phenol
GC/MS	8270C,D	Phorate
GC/MS	8270C,D	Pronamide
GC/MS	625 / 8270C,D / SOM01.2	Pyrene
GC/MS	8270C,D	Pyrididne
GC/MS	8270C,D	Safrole
GC/MS	8270C,D	Thionazin
HPLC	8330/8330A/8330B	1 3 5-Trinitrobenzene
HPLC	8330/8330A/8330B	1 3-Dinitrobenzene
HPLC	8330/8330A/8330B	2 4 6-Trinitrotoluene
HPLC	8330/8330A/8330B	2 4-Dinitrotoluene
HPLC	8330/8330A/8330B	2 6-Dinitrotoluene
HPLC	8330/8330A/8330B	2-Amino-4 6 -dinitrotoluene
HPLC	8330/8330A/8330B	2-Nitrotoluene
HPLC	8330/8330A/8330B	3-Nitrotoluene
HPLC	8330/8330A/8330B	4-Amino-2,3-dinitrotoluene
HPLC	8330/8330A/8330B	4-Nitrotoluene
HPLC	8330/8330A/8330B	Hexahydro-1 3 5-trinitro-1 3 5-triazine (RDX)
HPLC	8330/8330A/8330B	Nitrobenzene
HPLC	8330/8330A/8330B	Nitroglycerin
HPLC	8330/8330A/8330B	Octahydro-1 3 5 7-tetrazocine (HMX)
HPLC	8330/8330A/8330B	Tetryl

LABORATORY ACCREDITATION BUREAU

Certificate # L2223

Non-Potable Water

Technology	Method	Analyte
CVAA	245.1 / 7470A / ILM05.3	Mercury
CVAF	1631E	Low Level Mercury
ICP	200.7 / 6010B,C / ILM05.3	Aluminum
ICP	200.7 / 6010B,C / ILM05.3	Antimony
ICP	200.7 / 6010B,C / ILM05.3	Arsenic
ICP	200.7 / 6010B,C / ILM05.3	Barium
ICP	200.7 / 6010B,C / ILM05.3	Beryllium
ICP	200.7 / 6010B,C	Boron
ICP	200.7 / 6010B,C / ILM05.3	Cadmium
ICP	200.7 / 6010B,C / ILM05.3	Calcium
ICP	200.7 / 6010B,C / ILM05.3	Chromium
ICP	200.7 / 6010B,C / ILM05.3	Cobalt
ICP	200.7 / 6010B,C / ILM05.3	Copper
ICP	200.7 / 6010B,C / ILM05.3	Iron
ICP	200.7 / 6010B,C / ILM05.3	Lead
ICP	200.7 / 6010B,C / ILM05.3	Magnesium
ICP	200.7 / 6010B,C / ILM05.3	Manganese
ICP	200.7 / 6010B,C	Molybdenum
ICP	200.7 / 6010B,C / ILM05.3	Nickel
ICP	200.7 / 6010B,C / ILM05.3	Potassium
ICP	200.7 / 6010B,C / ILM05.3	Selenium
ICP	200.7	Silicon
ICP	200.7 / 6010B,C / ILM05.3	Silver
ICP	200.7 / 6010B,C / ILM05.3	Sodium
ICP	6010B,C	Strontium
ICP	200.7 / 6010B,C / ILM05.3	Thallium
ICP	200.7 / 6010B,C	Tin
ICP	200.7 / 6010B,C	Titanium
ICP	200.7 / 6010B,C / ILM05.3	Vanadium
ICP	200.7 / 6010B,C / ILM05.3	Zinc
ICP/MS	200.8 / 6020/6020A / ILM05.3	Aluminum
ICP/MS	200.8 / 6020/6020A / ILM05.3	Antimony
ICP/MS	200.8 / 6020/6020A / ILM05.3	Arsenic
ICP/MS	200.8 / 6020/6020A / ILM05.3	Barium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Beryllium
ICP/MS	200.8 / 6020/6020A	Boron
ICP/MS	200.8 / 6020/6020A / ILM05.3	Cadmium
ICP/MS	200.8 / 6020/6020A	Calcium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Chromium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Cobalt
ICP/MS	200.8 / 6020/6020A / ILM05.3	Copper
ICP/MS	200.8 / 6020/6020A	Iron
ICP/MS	200.8 / 6020/6020A / ILM05.3	Lead
ICP/MS	200.8 / 6020/6020A / ILM05.3	Magnesium
ICP/MS	200.8 / 6020/6020A	Manganese

LABORATORY ACCREDITATION BUREAU

Certificate # L2223

Non-Potable Water

Technology	Method	Analyte
ICP/MS	200.8 / 6020/6020A	Molybdenum
ICP/MS	200.8 / 6020/6020A / ILM05.3	Nickel
ICP/MS	200.8 / 6020/6020A	Potassium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Selenium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Silicon
ICP/MS	200.8 / 6020/6020A / ILM05.3	Silver
ICP/MS	200.8 / 6020/6020A	Sodium
ICP/MS	6020/6020A	Strontium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Thallium
ICP/MS	200.8 / 6020/6020A	Tin
ICP/MS	200.8 / 6020/6020A	Titanium
ICP/MS	200.8	Uranium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Vanadium
ICP/MS	200.8 / 6020/6020A / ILM05.3	Zinc
IC	300.0 / 9056/9056A	Bromide
IC	300.0 / 9056/9056A	Chloride
IC	300.0 / 9056/9056A	Nitrate as N
IC	300.0 / 9056/9056A	Nitrite as N
IC	300.0 / 9056/9056A	Nitrate + Nitrite
IC	300.0 / 9056/9056A	Orthophosphate as P
IC	300.0 / 9056/9056A	Sulfate
Titration	310.2 / 2320B	Alkalinity
Caculation	2340C	Hardness
Gravimetric	1664A	Oil and Grease
Gravimetric	2540 B, C, D	Solids
ISE	120.1 / 2510 B	Conductivity
ISE	2520B	Practical Salinity
ISE	4500F- C	Fluoride
ISE	4500H+ B	pH
ISE	5210B	TBOD / CBOD
Physical	1010 A	Ignitability
Physical	9040C	pH
Titration	2340B	Hardness
Titration	4500SO_3 B	Sulfite
Titration	9034 / 4500S^{2-} E	Sulfide
Titration	Chap. 7.3.4	Reactive Sulfide
TOC	9060A / 5310B	Total organic carbon
Turbidimetric	180.1 / 2130B	Turbidity
Turbidimetric	9038 / ASTM 516-02	Sulfate
UV/VIS	335.4 / 9012B / 4500-CN G	Amenable cyanide
UV/VIS	350.1 / 4500NH3 H	Ammonia as N
UV/VIS	3500Fe D	Ferrous Iron
UV/VIS	351.2	Kjeldahl nitrogen - total
UV/VIS	353.2 / 4500NO3 F	Nitrate + Nitrite

LABORATORY ACCREDITATION BUREAU

Certificate # L2223

Non-Potable Water

Technology	Method	Analyte
UV/VIS	353.2 / 4500NO3 F	Nitrate as N
UV/VIS	353.2 / 4500NO3 F	Nitrite as N
UV/VIS	365.1 / 4500P E	Orthophosphate as P
UV/VIS	365.4	Phosphorus total
UV/VIS	376.3	AVS-SEM
UV/VIS	410.4	COD
UV/VIS	420.1 / 9065	Total Phenolics
UV/VIS	4500Cl G	Total Residual Chlorine
UV/VIS	5540C	MBAS
UV/VIS	7196A / 3500-Cr D	Chromium VI
UV/VIS	9012B / ILM05.3/ 335.4	Total Cyanide
UV/VIS	9251 / 4500Cl E	Chloride
UV/VIS	Chap. 7.3.4	Reactive Cyanide

Preparation	Method	Type
Cleanup Methods	3640A	Gel Permeation Clean-up
Cleanup Methods	3630C	Silica Gel
Cleanup Methods	3660B	Sulfur Clean-Up
Cleanup Methods	3665A	Sulfuric Acid Clean-Up
Organic Preparation	3510C	Separatory Funnel Extraction
Organic Preparation	3520C	Continuous Liquid-Liquid Extraction
Inorganic Preparation	3010A	Hotblock
Volatile Organic Preparation	5030B,C	Purge and Trap

Solid and Chemical Waste

Technology	Method	Analyte
GC/ECD	8081A,B/ SOM01.2	4 4`-DDD
GC/ECD	8081A,B / SOM01.2	4 4`-DDE
GC/ECD	8081A,B / SOM01.2	4 4`-DDT
GC/ECD	8081A,B / SOM01.2	Aldrin
GC/ECD	8081A,B / SOM01.2	alpha-BHC (alpha-Hexachlorocyclohexane)
GC/ECD	8081A,B / SOM01.2	Alpha-Chlordane
GC/ECD	8081A,B / SOM01.2	beta-BHC (beta-Hexachlorocyclohexane)
GC/ECD	608 /8081A,B	Chlordane (tech.)
GC/ECD	8081A,B / SOM01.2	delta-BHC
GC/ECD	8081A,B / SOM01.2	Dieldrin
GC/ECD	8081A,B / SOM01.2	Endosulfan I
GC/ECD	8081A,B / SOM01.2	Endosulfan II
GC/ECD	8081A,B / SOM01.2	Endosulfan sulfate
GC/ECD	8081A,B / SOM01.2	Endrin
GC/ECD	8081A,B / SOM01.2	Endrin aldehyde
GC/ECD	8081A,B / SOM01.2	Endrin Ketone

LABORATORY ACCREDITATION BUREAU

Certificate # L2223

Solid and Chemical Waste

Technology	Method	Analyte
GC/ECD	8081A,B / SOM01.2	gamma-BHC (Lindane gamma-Hexachlorocyclohexane)
GC/ECD	8081A,B / SOM01.2	Heptachlor
GC/ECD	8081A,B / SOM01.2	Heptachlor epoxide
GC/ECD	8081A,B / SOM01.2	Methoxychlor
GC/ECD	8081A,B / SOM01.2	Toxaphene (Chlorinated camphene)
GC/ECD	8082/8082A/ SOM01.2	Aroclor-1016 (PCB-1016)
GC/ECD	8082/8082A/ SOM01.2	Aroclor-1221 (PCB-1221)
GC/ECD	8082/8082A/ SOM01.2	Aroclor-1232 (PCB-1232)
GC/ECD	8082/8082A/ SOM01.2	Aroclor-1242 (PCB-1242)
GC/ECD	8082/8082A/ SOM01.2	Aroclor-1248 (PCB-1248)
GC/ECD	8082/8082A/ SOM01.2	Aroclor-1254 (PCB-1254)
GC/ECD	8082/8082A/ SOM01.2	Aroclor-1260 (PCB-1260)
GC/ECD	8082/8082A	Aroclor-1262 (PCB-1262)
GC/ECD	8082/8082A	Aroclor-1268 (PCB-1268)
GC/ECD	8082/8082A	2 2' 3 3' 4 4' 5 5' 6-Nonachlorobiphenyl (BZ 206)
GC/ECD	8082/8082A	2 2' 3 3' 4 4' 5 6-Octachlorobiphenyl (BZ 195)
GC/ECD	8082/8082A	2 2' 3 3' 4 4' 5-Heptachlorobiphenyl (BZ 170)
GC/ECD	8082/8082A	2 2' 3 3' 4 4'-Hexachlorobiphenyl (BZ 128)
GC/ECD	8082/8082A	2 2' 3 4 4' 5 5'-Heptachlorobiphenyl (BZ 180)
GC/ECD	8082/8082A	2 2' 3 4 4' 5' 6-Heptachlorobiphenyl (BZ 183)
GC/ECD	8082/8082A	2 2' 3 4 4' 5'-Hexachlorobiphenyl (BZ 138)
GC/ECD	8082/8082A	2 2' 3 4 4' 6 6'-Heptachlorobiphenyl (BZ 184)
GC/ECD	8082/8082A	2 2' 3 4' 5 5' 6-Heptachlorobiphenyl (BZ 187)
GC/ECD	8082/8082A	2 2' 3 4 5'-Pentachlorobiphenyl (BZ 87)
GC/ECD	8082/8082A	2 2' 3 5'-Tetrachlorobiphenyl (BZ 44)
GC/ECD	8082/8082A	2 2' 4 4' 5 5'-Hexachlorobiphenyl (BZ 153)
GC/ECD	8082/8082A	2 2' 4 5 5'-Pentachlorobiphenyl (BZ 101)
GC/ECD	8082/8082A	2 2' 4' 5-Tetrachlorobiphenyl (BZ 49)
GC/ECD	8082/8082A	2 2' 5 5'-Tetrachlorobiphenyl (BZ 52)
GC/ECD	8082/8082A	2 2' 5-Trichlorobiphenyl (BZ 18)
GC/ECD	8082/8082A	2 3 3' 4 4' 5-Hexachlorobiphenyl (BZ 156)
GC/ECD	8082/8082A	2 3 3' 4 4' 5'-Hexachlorobiphenyl (BZ 157)
GC/ECD	8082/8082A	2 3 3' 4 4'-Pentachlorobiphenyl (BZ 105)
GC/ECD	8082/8082A	2 3 3’ 4 4’ 5 5’-Heptachlorobiphenyl (BZ 189)
GC/ECD	8082/8082A	2 3' 4 4' 5 5'-Hexachlorobiphenyl (BZ 167)
GC/ECD	8082/8082A	2 3' 4 4' 5-Pentachlorobiphenyl (BZ 118)
GC/ECD	8082/8082A	2 3' 4 4'5-Pentachlorobiphenyl (BZ 123)
GC/ECD	8082/8082A	2 3' 4 4'-Tetrachlorobiphenyl (BZ 66)
GC/ECD	8082/8082A	2 3’ 4 4’ 5-Pentachlorobiphenyl (BZ 114)
GC/ECD	8082/8082A	2 4 4'-Trichlorobiphenyl (BZ 28)
GC/ECD	8082/8082A	2 4'-Dichlorobiphenyl (BZ 8)
GC/ECD	8082/8082A	3 3' 4 4' 5 5'-Hexachlorobiphenyl (BZ 169)
GC/ECD	8082/8082A	3 3' 4 4' 5-Pentachlorobiphenyl (BZ 126)
GC/ECD	8082/8082A	3 3' 4 4'-Tetrachlorobiphenyl (BZ 77)

Solid and Chemical Waste

Technology	Method	Analyte
GC/ECD	8082/8082A	3 4 4' 5-Tetrachlorobiphenyl (BZ 81)
GC/ECD	8082/8082A	Decachlorobiphenyl (BZ 209)
GC/ECD	8151A	2 4 5-T
GC/ECD	8151A	2 4-D
GC/ECD	8151A	2 4-DB
GC/ECD	8151A	Dalapon
GC/ECD	8151A	Dicamba
GC/ECD	8151A	Dichloroprop
GC/ECD	8151A	DInoseb
GC/ECD	8151A	MCPA
GC/ECD	8151A	MCPP
GC/ECD	8151A	Pentachlorophenol
GC/ECD	8151A	Silvex (2 4 5-TP)
GC/FID	8015B,C	Diesel range organics (DRO)
GC/FID	8015B,C	Gasoline range organics (GRO)
GC/FID	8011	EDB
GC/FID	8011	1 2-Dibromo-3-chloropropane
GC/MS	8260B,C	1 1 1 2-Tetrachloroethane
GC/MS	8260B,C / SOM01.2	1 1 1-Trichloroethane
GC/MS	8260B,C / SOM01.2	1 1 2 2-Tetrachloroethane
GC/MS	SOM01.2	1 1 2-Trichloro-1 2 2-trifluoroethane
GC/MS	8260B,C / SOM01.2	1 1 2-Trichloroethane
GC/MS	8260B,C / SOM01.2	1 1-Dichloroethane
GC/MS	8260B,C / SOM01.2	1 1-Dichloroethylene
GC/MS	8260B,C	1 1-Dichloropropene
GC/MS	8260B,C / SOM01.2	1 2 3-Trichlorobenzene
GC/MS	8260B,C	1 2 3-Trichloropropane
GC/MS	8260B,C / SOM01.2	1 2 4-Trichlorobenzene
GC/MS	8260B,C	1 2 4-Trimethylbenzene
GC/MS	8260B,C / SOM01.2	1 2-Dibromo-3-chloropropane
GC/MS	8260B,C / SOM01.2	1 2-Dichlorobenzene
GC/MS	8260B,C / SOM01.2	1 2-Dichloroethane
GC/MS	8260B,C / SOM01.2	1 2-Dichloropropane
GC/MS	8260B,C	1 3 5-Trimethylbenzene
GC/MS	8260B,C / SOM01.2	1 3-Dichlorobenzene
GC/MS	8260B,C	1 3-Dichloropropane
GC/MS	8260B,C / SOM01.2	1 4-Dichlorobenzene
GC/MS	8260B,C / SOM01.2	1 4-Dioxane
GC/MS	8260B,C	2 2-Dichloropropane
GC/MS	8260B,C / SOM01.2	2-Butanone
GC/MS	8260B,C	2-Chloroethyl vinyl ether
GC/MS	8260B,C	2-Chlorotoluene
GC/MS	8260B,C / SOM01.2	2-Hexanone
GC/MS	8260B,C	4-Chlorotoluene
GC/MS	8260B,C / SOM01.2	4-Methyl-2-pentanone

Solid and Chemical Waste

Technology	Method	Analyte
GC/MS	8260B,C / SOM01.2	Acetone
GC/MS	8260B,C	Acetonitrile
GC/MS	8260B,C	Acrolein
GC/MS	8260B,C	Acrylonitrile
GC/MS	8260B,C	Allyl chloride
GC/MS	8260B,C / SOM01.2	Benzene
GC/MS	8260B,C	Bromobenzene
GC/MS	8260B,C / SOM01.2	Bromochloromethane
GC/MS	8260B,C / SOM01.2	Bromodichloromethane
GC/MS	8260B,C / SOM01.2	Bromoform
GC/MS	8260B,C / SOM01.2	Carbon disulfide
GC/MS	8260B,C / SOM01.2	Carbon tetrachloride
GC/MS	8260B,C / SOM01.2	Chlorobenzene
GC/MS	8260B,C / SOM01.2	Chloroethane
GC/MS	8260B,C / SOM01.2	Chloroform
GC/MS	8260B,C	Chloroprene
GC/MS	8260B,C / SOM01.2	cis-1 2-Dichloroethene
GC/MS	8260B,C / SOM01.2	cis-1 3-Dichloropropene
GC/MS	SOM01.2	Cyclohexane
GC/MS	8260B,C / SOM01.2	Dibromochloromethane
GC/MS	8260B,C	Dibromomethane
GC/MS	624 / 8260B,C / SOM01.2	Dichlorodifluoromethane
GC/MS	8260B,C	Diethyl ether
GC/MS	8260B,C	Di-isopropylether
GC/MS	8260B,C / SOM01.2	EDB
GC/MS	8260B,C	Ethyl methacrylate
GC/MS	8260B,C / SOM01.2	Ethylbenzene
GC/MS	8260B,C	Ethyl-t-butylether
GC/MS	8260B,C	Hexachlorobutadiene
GC/MS	8260B,C	Iodomethane
GC/MS	8260B,C	Isobutyl alcohol
GC/MS	8260B,C / SOM01.2	Isopropyl benzene
GC/MS	SOM01.2	m p-xylenes
GC/MS	8260B,C	Methacrylonitrile
GC/MS	SOM01.2	Methyl acetate
GC/MS	8260B,C / SOM01.2	Methyl bromide (Bromomethane)
GC/MS	8260B,C / SOM01.2	Methyl chloride (Chloromethane)
GC/MS	8260B,C	Methyl methacrylate
GC/MS	8260B,C / SOM01.2	Methyl tert-butyl ether
GC/MS	SOM01.2	Methylcyclohexane
GC/MS	8260B,C / SOM01.2	Methylene chloride
GC/MS	8260B,C	Naphthalene
GC/MS	8260B,C	n-Butylbenzene
GC/MS	8260B,C	n-proplybenzene
GC/MS	8260B,C	o-Xylene

Solid and Chemical Waste

Technology	Method	Analyte
GC/MS	8260B,C	p-Isopropyltoluene
GC/MS	8260B,C	Propionitrile
GC/MS	8260B,C	sec-butylbenzene
GC/MS	8260B,C / SOM01.2	Styrene
GC/MS	8260B,C	t-Amylmethylether
GC/MS	8260B,C	tert-Butyl alcohol
GC/MS	8260B,C	tert-Butylbenzene
GC/MS	8260B,C / SOM01.2	Tetrachloroethylene (Perchloroethylene)
GC/MS	8260B,C	Tetrahydrofuran
GC/MS	8260B,C / SOM01.2	Toluene
GC/MS	8260B,C / SOM01.2	trans-1 2-Dichloroethylene
GC/MS	8260B,C / SOM01.2	trans-1 3-Dichloropropylene
GC/MS	8260B,C	Trans-1 4-Dichloro-2-butuene
GC/MS	8260B,C / SOM01.2	Trichloroethene (Trichloroethylene)
GC/MS	8260B,C / SOM01.2	Trichlorofluoromethane
GC/MS	8260B,C	Vinyl acetate
GC/MS	8260B,C / SOM01.2	Vinyl chloride
GC/MS	8260B,C	Xylene
GC/MS	8270C,D	1-Naphthylamine
GC/MS	8270C,D	2-Acetylaminofluorene
GC/MS	8270C,D / SOM01.2	2-Chloronaphthalene
GC/MS	8270C,D / SOM01.2	2-Chlorophenol
GC/MS	8270C,D / SOM01.2	2-Methylnaphthalene
GC/MS	8270C,D / SOM01.2	2-Methylphenol
GC/MS	8270C,D	2-Naphthylamine
GC/MS	8270C,D	2-Nitroaniline
GC/MS	8270C,D / SOM01.2	2-Nitrophenol
GC/MS	8270C,D	2-Picoline
GC/MS	8270C,D	3-Methylcholanthrene
GC/MS	8270C,D / SOM01.2	3-Nitroaniline
GC/MS	8270C,D	4-Aminobiphenyl
GC/MS	8270C,D / SOM01.2	4-Bromophenyl phenyl ether
GC/MS	8270C,D / SOM01.2	4-Chloro-3-methylphenol
GC/MS	8270C,D / SOM01.2	4-Chloroaniline
GC/MS	8270C,D / SOM01.2	4-Chlorophenyl phenylether
GC/MS	8270C,D	4-Dimethyl aminoazobenzene
GC/MS	8270C,D / SOM01.2	4-Methylphenol
GC/MS	8270C,D / SOM01.2	4-Nitroaniline
GC/MS	8270C,D / SOM01.2	4-Nitrophenol
GC/MS	8270C,D	5-Nitro-o-toluidine
GC/MS	8270C,D	a a-Dimethylphenethylamine
GC/MS	8270C,D / SOM01.2	Acenaphthene
GC/MS	8270C,D / SOM01.2	Acenaphthylene
GC/MS	8270C,D / SOM01.2	Acetophenone
GC/MS	8270C,D	Aniline

Solid and Chemical Waste

Technology	Method	Analyte
GC/MS	8270C,D / SOM01.2	Anthracene
GC/MS	8270C,D	Aramite
GC/MS	8270C,D / SOM01.2	Atrazine
GC/MS	SOM01.2	Benzaldehyde
GC/MS	8270C,D	Benzidine
GC/MS	8270C,D / SOM01.2	Benzo(a)anthracene
GC/MS	8270C,D / SOM01.2	Benzo(a)pyrene
GC/MS	8270C,D / SOM01.2	Benzo(b)fluoranthene
GC/MS	8270C,D / SOM01.2	Benzo(g h i)perylene
GC/MS	8270C,D / SOM01.2	Benzo(k)fluoranthene
GC/MS	8270C,D	Benzoic Acid
GC/MS	8270C,D	Benzyl alcohol
GC/MS	8270C,D / SOM01.2	Biphenyl
GC/MS	8270C,D / SOM01.2	bis(2-Chloroethoxy)methane
GC/MS	8270C,D / SOM01.2	bis(2-Chloroethyl) ether
GC/MS	8270C,D / SOM01.2	bis(2-Ethylhexyl) phthalate (DEHP)
GC/MS	8270C,D / SOM01.2	Butyl benzyl phthalate
GC/MS	SOM01.2	Caprolactam
GC/MS	8270C,D / SOM01.2	Carbazole
GC/MS	8270C,D	Chlorobenzilate
GC/MS	8270C,D / SOM01.2	Chrysene
GC/MS	8270C,D	Diallate
GC/MS	8270C,D / SOM01.2	Dibenz(a h)anthracene
GC/MS	8270C,D / SOM01.2	Dibenzofuran
GC/MS	8270C,D / SOM01.2	Diethyl phthalate
GC/MS	8270C,D	Dimethoate
GC/MS	8270C,D / SOM01.2	Dimethyl phthalate
GC/MS	8270C,D / SOM01.2	Di-n-butyl phthalate
GC/MS	8270C,D / SOM01.2	Di-n-octyl phthalate
GC/MS	8270C,D	Ethyl methanesulfonate
GC/MS	8270C,D	Famfur
GC/MS	8270C,D / SOM01.2	Fluoranthene
GC/MS	8270C,D / SOM01.2	Fluorene
GC/MS	8270C,D / SOM01.2	Hexachlorobenzene
GC/MS	8270C,D / SOM01.2	Hexachlorobutadiene
GC/MS	8270C,D / SOM01.2	Hexachlorocyclopentadiene
GC/MS	8270C,D / SOM01.2	Hexachloroethane
GC/MS	8270C,D	Hexachloropropene
GC/MS	8270C,D	Isodrin
GC/MS	8270C,D / SOM01.2	Isophorone
GC/MS	8270C,D	Isosafrole
GC/MS	8270C,D	Methapyriline
GC/MS	8270C,D	Methyl methanesulfonate
GC/MS	8270C,D	Methyl parathion
GC/MS	8270C,D / SOM01.2	Naphthalene

Solid and Chemical Waste

Technology	Method	Analyte
GC/MS	8270C,D / SOM01.2	Nitrobenzene
GC/MS	8270C,D	Nitroquinoline-1-oxide
GC/MS	8270C,D	n-Nitrosodiethylamine
GC/MS	8270C,D / SOM01.2	n-Nitrosodimethylamine
GC/MS	8270C,D	n-Nitroso-di-n-butylamine
GC/MS	8270C,D / SOM01.2	n-Nitrosodi-n-propylamine
GC/MS	8270C,D / SOM01.2	n-Nitrosodiphenylamine
GC/MS	8270C,D	n-Nitrosomethylethylamine
GC/MS	8270C,D	n-Nitrosomorpholine
GC/MS	8270C,D	n-Nitrosopiperidine
GC/MS	8270C,D	n-Nitrosopyrrolidine
GC/MS	8270C,D	o o o-Triethyl phosphorothioate
GC/MS	8270C,D	o-Toluidine
GC/MS	8270C,D	Pentachlorobenzene
GC/MS	8270C,D	Pentachloronitrobenzene
GC/MS	8270C,D/ SOM01.2	Pentachlorophenol
GC/MS	8270C,D	Phenacetin
GC/MS	8270C,D / SOM01.2	Phenanthrene
GC/MS	8270C,D / SOM01.2	Phenol
GC/MS	8270C,D	Phorate
GC/MS	8270C,D	Pronamide
GC/MS	8270C,D / SOM01.2	Pyrene
GC/MS	8270C,D	Pyrididne
GC/MS	8270C,D	Safrole
GC/MS	8270C,D	Thionazin
GC/MS	8270C,D / SOM01.2	Indeno(1 2 3-cd)pyrene
GC/MS	8270C,D / SOM01.2	1 2 4-Trichlorobenzene
GC/MS	8270C,D	1 3 5-Trinitrobenzene
GC/MS	8270C,D / SOM01.2	1 2 4 5-Tetrachlorobenzene
GC/MS	8270C,D / SOM01.2	2 4 5-Trochlorophenol
GC/MS	8270C,D / SOM01.2	2 4 6-Trichlorophenol
GC/MS	8270C,D / SOM01.2	2 3 4 6-Tetrachlorophenol
GC/MS	8270C,D / SOM01.2	1 2-Dichlorobenzene
GC/MS	8270C,D	1 2-Diphenylhydrazine
GC/MS	8270C,D / SOM01.2	1 3-Dichlorobenzene
GC/MS	8270C,D	1 3-Dinitrobenzene
GC/MS	8270C,D / SOM01.2	1 4-Dichlorobenzene
GC/MS	8270C,D	1 4-Dioxane
GC/MS	8270C,D	1 4-Naphthoquinone
GC/MS	8270C,D	1 4-Phenylenediamine
GC/MS	8270C,D / SOM01.2	bis(2-Chloroisopropyl) ether (2 2`-Oxybis(1-chloropropane))
GC/MS	8270C,D / SOM01.2	2 4-Dichlorophenol
GC/MS	8270C,D / SOM01.2	2 4-Dimethylphenol
GC/MS	8270C,D / SOM01.2	2 4-Dinitrophenol

Solid and Chemical Waste

Technology	Method	Analyte
GC/MS	8270C,D / SOM01.2	2 4-Dinitrotoluene (2 4-DNT)
GC/MS	8270C,D	2 6-Dichlorophenol
GC/MS	8270C,D / SOM01.2	2 6-Dinitrotoluene (2 6-DNT)
GC/MS	8270C,D / SOM01.2	3 3`-Dichlorobenzidine
GC/MS	8270C,D	3 3'-Dimethylbenzidine
GC/MS	8270C,D / SOM01.2	2-Methyl-4 6-dinitrophenol
GC/MS	8270C,D	7,12-Dimethylphenethylamine
HPLC	8330/8330A/8330B (Analysis Only)	1 3 5-Trinitrobenzene
HPLC	8330/8330A/8330B (Analysis Only)	1 3-Dinitrobenzene
HPLC	8330/8330A/8330B (Analysis Only)	2 4 6-Trinitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	2 4-Dinitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	2 6-Dinitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	2-Amino-4 6 -dinitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	2-Nitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	3-Nitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	4-Amino-2,3-dinitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	4-Nitrotoluene
HPLC	8330/8330A/8330B (Analysis Only)	Hexahydr-1 3 5-trinitro-1 3 5-triazine (RDX)
HPLC	8330/8330A/8330B (Analysis Only)	Nitrobenzene
HPLC	8330/8330A/8330B (Analysis Only)	Nitroglycerin
HPLC	8330/8330A/8330B (Analysis Only)	Octahydro-1 3 5 7-tetrazocine (HMX)
HPLC	8330/8330A/8330B (Analysis Only)	Tetryl
CVAA	7471B/ ILM05.3	Mercury
CVAF	1631E	Low Level Mercury
ICP	6010B,C /ILM05.3	Aluminum
ICP	6010B,C /ILM05.3	Antimony
ICP	6010B,C /ILM05.3	Arsenic
ICP	6010B,C /ILM05.3	Barium
ICP	6010B,C /ILM05.3	Beryllium
ICP	6010B,C	Boron
ICP	6010B,C /ILM05.3	Cadmium

Solid and Chemical Waste

Technology	Method	Analyte
ICP	6010B,C /ILM05.3	Calcium
ICP	6010B,C /ILM05.3	Chromium
ICP	6010B,C /ILM05.3	Cobalt
ICP	6010B,C /ILM05.3	Copper
ICP	6010B,C /ILM05.3	Iron
ICP	6010B,C /ILM05.3	Lead
ICP	6010B,C /ILM05.3	Magnesium
ICP	6010B,C /ILM05.3	Manganese
ICP	6010B,C	Molybdenum
ICP	6010B,C /ILM05.3	Nickel
ICP	6010B,C /ILM05.3	Potassium
ICP	6010B,C /ILM05.3	Selenium
ICP	200.7	Silicon
ICP	6010B,C /ILM05.3	Silver
ICP	6010B,C /ILM05.3	Sodium
ICP	6010B,C	Strontium
ICP	6010B,C /ILM05.3	Thallium
ICP	6010B,C	Tin
ICP	6010B,C	Titanium
ICP	6010B,C /ILM05.3	Vanadium
ICP	6010B,C /ILM05.3	Zinc
ICP/MS	6020/6020A / ILM05.3	Aluminum
ICP/MS	6020/6020A / ILM05.3	Antimony
ICP/MS	6020/6020A / ILM05.3	Arsenic
ICP/MS	6020/6020A / ILM05.3	Barium
ICP/MS	6020/6020A / ILM05.3	Beryllium
ICP/MS	6020/6020A	Boron
ICP/MS	6020/6020A / ILM05.3	Cadmium
ICP/MS	6020/6020A	Calcium
ICP/MS	6020/6020A / ILM05.3	Chromium
ICP/MS	6020/6020A / ILM05.3	Cobalt
ICP/MS	6020/6020A / ILM05.3	Copper
ICP/MS	6020/6020A	Iron
ICP/MS	6020/6020A / ILM05.3	Lead
ICP/MS	6020/6020A / ILM05.3	Magnesium
ICP/MS	6020/6020A	Manganese
ICP/MS	6020/6020A	Molybdenum
ICP/MS	6020/6020A / ILM05.3	Nickel
ICP/MS	6020/6020A	Potassium
ICP/MS	6020/6020A / ILM05.3	Selenium
ICP/MS	6020/6020A / ILM05.3	Silver
ICP/MS	6020/6020A	Sodium
ICP/MS	6020/6020A	Strontium
ICP/MS	6020/6020A / ILM05.3	Thallium
ICP/MS	6020/6020A	Tin

LABORATORY ACCREDITATION BUREAU

Certificate # L2223

Solid and Chemical Waste

Technology	Method	Analyte
ICP/MS	6020/6020A	Titanium
ICP/MS	6020/6020A / ILM05.3	Vanadium
ICP/MS	6020/6020A / ILM05.3	Zinc
IC	9056/9056A	Chloride
IC	9056/9056A	Fluoride
IC	9056/9056A	Nitrate as N
IC	9056/9056A	Nitrite as N
IC	9056/9056A	Sulfate
Gravimetric	9070A / 9071B	Oil and Grease
Physical	1010A	Ignitability
Physical	9045D	pH
Titration	Chap 7.3.4	Reactive Sulfide
TOC	Lloyd Kahn	Total organic carbon
TOC	9060A / 5310B	Total organic carbon
Turbidimetric	9038 / ASTM 516-02	Sulfate
UV/VIS	350.1 / 4500NH3 H	Ammonia as N
UV/VIS	9251 / 4500Cl E	Chloride
UV/VIS	Chap. 7.3.4	Reactive Cyanide
UV/VIS	376.3	AVS-SEM
UV/VIS	3500Fe D	Ferrous Iron
Cleanup Methods	3630C	Silica Gel
UV/VIS	7196	Chromium VI
UV/VIS	7196A	Chromium VI
UV/VIS	9012B / ILM05.3	Total cyanide
Preparation	**Method**	**Type**
Preparation	1311	Toxicity Characteristic Leaching Procedure
Preparation	1312	Synthetic Precipitation Leaching Procedure
Cleanup Methods	3660B	Sulfur
Cleanup Methods	3620C	Florsil
Cleanup Methods	3630C	Silica Gel
Cleanup Methods	3640A	GPC
Organic Preparation	3540C	Soxhlet Extraction
Organic Preparation	3545A	Pressurized Fluid Extraction
Organic Preparation	3550C	Sonication
Inorganics Preparation	3050B	Hotblock
Inorganics Preparation	3060A	Alkaline Digestion
Volatile Organics Preparation	5035/5035A	Closed System Purge and Trap

Notes:

1) This laboratory offers commercial testing service.

Approved By: Date: November 4, 2009

R. Douglas Leonard
Chief Technical Officer

Issued: 11/04/09

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-101
Revision History
Cover Page
Page 1

TITLE: **EQUIPMENT MAINTENANCE**

Prepared By: Julie Ricardi Date: 8-96

Approved By:

Group Supervisor: _____ Date: _____

Operations Manager: Joel C. Burton Date: 12/15/00

QA Officer: Deborah J. Nadeau Date: 12·13·00

General Manager: JCBurton for D. McGrath Date: 12/15/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Added requirements for recording maintenance in appropriate log. Added Archon maintenance. Modified Lachat maintenance. Attached equipment list.	DN	12·13·00	12·13·00
02	Removed references to General Manager. Removed furnace (GFAA) maintenance from Figure 1 and added ICP-MS. Updated equipment list.	DN	10·22·02	10·22·02
03	Changed Supervisor to department manager. Added maintenance for the ASE and general lab maintenance. Updated equipment list.	HRC	06.03.04	06.03.04
04	Added new equipment list.	LAD	01·28·05	01·28·05
05	Added Horizon SPE Extractor & HPLC maintenance to Table 1. Added TOC analyzer, IC & miscellaneous instrument maintenance to Table 2. Updated equipment list.	DN	03·06	03·06

TITLE: **EQUIPMENT MAINTENANCE**

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Updated ICP maintenance to include maintenance for new ICP (7500). Updated equipment list.	DR	04.07	04.07
07	Updated equipment list.	DR	07.08	07.08

TITLE: EQUIPMENT MAINTENANCE

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **CA-101-07**, titled **EQUIPMENT MAINTENANCE**.

Recipient: _____Date:_____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **CA-101-07**, titled **EQUIPMENT MAINTENANCE**.

Recipient: _____Date:_____

TITLE: EQUIPMENT MAINTENANCE

1.0 SCOPE AND APPLICATION

The consistent generation of high quality analytical results requires properly operating analytical equipment. Accordingly, Katahdin Analytical Services, Inc. has an equipment maintenance program, which prescribes the necessary maintenance measures required and also schedules the frequency of conduct and documentation of those measures.

The purpose of this SOP is to describe the routine maintenance procedures employed for analytical equipment utilized by Katahdin Analytical personnel. It is applicable to all equipment utilized by laboratory personnel for analytical work. The procedures outlined in this SOP provide general guidance for equipment maintenance and documentation. The appropriate equipment manufacturer's manual(s) and specific analytical SOPs must be available and shall be consulted for specific maintenance and cleaning procedures.

1.1 Definitions

1.2 Responsibilities

It is the responsibility of each technical employee to carry out and document the required routine equipment maintenance at the required frequency for the instrumentation that they use.

It is the responsibility of each Department Manager to ensure that maintenance performed in-house is conducted properly by experienced personnel familiar with the equipment. On a routine basis, the department manager or qualified designee reviews equipment maintenance activities and recordkeeping requirements. Deficiencies are brought to the attention of the analyst who is responsible for an unacceptable procedure or entry, and any corrections are made by the appropriate analyst.

The Quality Assurance Officer is responsible for conducting periodic audits of the equipment maintenance program and documentation in each analytical area. Maintenance logs and contracted vendor maintenance records are reviewed for completeness and frequency of maintenance. Deficiencies are immediately brought to the attention of the Supervisor who is responsible for informing his/her staff and initiating the necessary corrective or improved actions in his/her analytical area.

2.0 SUMMARY OF METHOD

Not applicable.

3.0 INTERFERENCES

Refer to specific instrument manufacturer's operating manuals.

4.0 APPARATUS AND MATERIALS

Not applicable.

5.0 REAGENTS

Not applicable.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Not applicable.

7.0 PROCEDURES

7.1 ROUTINE PREVENTIVE MAINTENANCE

Preventive maintenance (PM) may be conducted by qualified Katahdin personnel or may be performed under contract by authorized service technicians. Between service visits, instruments having PM service contracts are cleaned and maintained according to manufacturer's specifications by experienced Katahdin personnel. Instruments not under service contracts are cleaned and maintained by experienced laboratory personnel according to manufacturer's specifications and based on professional experience. The frequency of maintenance performed depends on the equipment. Preventive maintenance in each analytical section at Katahdin Analytical is scheduled, conducted and documented on a regular basis by the analytical section staff. Many maintenance needs (e.g., accidental breakage, part failure) are not satisfied by scheduled maintenance and are performed as needed. This would apply to routine daily instrument maintenance as well as non-scheduled instrument maintenance (e.g., replacement of GC injection port liners, replacement of volatiles Tekmar traps, etc.). Maintenance procedures indicated in Tables 1 and 2 are documented in appropriate logbooks maintained in each analytical area. Included in the documentation are general remarks describing the maintenance operations performed, the date maintenance was performed, and the signature or initials of the individuals who completed the maintenance. If a specific instrument problem prompted the need for maintenance, the problem and its resolution are documented along with the description of maintenance performed. Specific requirements for maintenance logs are described in Section 7.3.

7.2 PREVENTIVE MAINTENANCE SCHEDULES

Tables 1 and 2 summarize the routine preventive maintenance procedures and schedules followed within Katahdin Analytical for major laboratory instrumentation. These items are required to be documented in the applicable instrument or

maintenance log. Refer to Section 7.3 for specific requirements for maintenance log documentation.

7.3 MAINTENANCE LOG (or equivalent) REQUIREMENTS

Maintenance must be recorded in either the instrument run log or in a separate maintenance log. For equipment that does not have a run log or does not necessitate a separate maintenance log (i.e. little maintenance required), any maintenance may be recorded in the appropriate logbook (i.e. balance maintenance recorded in the balance calibration log). Attachment A provides a list of analytical equipment for which maintenance activities are recorded. Balances are not listed, although maintenance activities are recorded for these.

7.3.1 Maintenance logs (or equivalent) shall include the type of equipment and the analytical group/analysis for which it is designated, the manufacturer's model number and serial number, as well as any laboratory-assigned identifier (e.g., GC08, MS2, etc.). Each lab-assigned identifier must be unique and each instrument must have only one lab-assigned identifier. As new instrumentation is received, the date acquired, condition when received (new, used, reconditioned), and date put into use should be recorded in the new instrument's maintenance log.

7.3.2 Each page of the maintenance logbook (or equivalent) must clearly indicate the type of equipment and laboratory-assigned identifier in the header information. Maintenance entries shall include the date and signature or initials of the individual performing the maintenance as well as a brief explanation of the maintenance performed. If maintenance was conducted as the result of a suspected or known instrument problem, a summary of the problem and its final resolution must be included. The maintenance record must clearly indicate whether the problem was resolved and the date when return to control was noted.

7.3.3 On a routine basis the Department Manager or qualified designee shall review equipment maintenance activities and adherence to recordkeeping requirements. Deficiencies are brought to the attention of the analyst who was responsible for the unacceptable procedure or entry, and any corrections are made by the appropriate analyst. Documentation of the maintenance log review can be made by initialing and dating each page of the log or by indicating "reviewed by", initials and date on the front cover of the maintenance log (or equivalent).

7.3.4 On a periodic basis the laboratory QA Officer shall conduct audits of the equipment maintenance program and documentation in the analytical areas. Maintenance logs and contracted vendor maintenance records are reviewed for completeness and adherence to this SOP. Deficiencies are immediately brought to the attention of the Department Manager who is responsible for informing his/her staff and initiating the necessary corrective actions in his/her analytical area.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Not applicable.

9.0 METHOD PERFORMANCE

Not applicable.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Applicable equipment manufacturer's operating manuals.

LIST OF TABLES & ATTACHMENTS

TABLE 1 – ROUTINE MAINTENANCE SCHEDULE FOR ORGANICS INSTRUMENTATION
TABLE 2 - ROUTINE MAINTENANCE SCHEDULE FOR INORGANICS INSTRUMENTATION
TABLE 3 - ROUTINE MAINTENANCE SCHEDULE FOR GENERAL LABORATORY EQUIPMENT
ATTACHMENT A – ANALYTICAL EQUIPMENT

TABLE 1

ROUTINE MAINTENANCE SCHEDULE FOR ORGANICS INSTRUMENTATION

GC/MS

Regularly performed maintenance includes, but is not limited to, the following for GC/MS instrumentation:

- Check to ensure the pressure on the primary regulator never runs below 100 psi
- Check to ensure the gas supply is sufficient for the day's activity, and delivery pressures are set as described in the SOP.
- Change septa weekly or as needed.
- Replace/cut GC column as needed.
- Replace GC injector glass liner weekly or as needed.
- Replace glass jet splitter as needed.
- Replace pump oil as needed.
- Change gas line dryers as needed.
- Replace electron multiplier as needed.

GC

Regularly performed maintenance includes, but is not limited to, the following for GC instrumentation:

- Check to ensure the pressure on the primary regulator never runs below 100 psi.
- Check to ensure the gas supply is sufficient for the day's activity, and delivery pressures are set as described in the SOP.
- Change septa weekly or as needed.
- Replace/cut GC column as needed.
- Replace GC injector glass liner as needed.
- Change O_2/moisture traps as needed
- Clean/replace GC detector as needed.

TABLE 1, cont'd

ROUTINE MAINTENANCE SCHEDULE FOR ORGANICS INSTRUMENTATION

Purge and Trap Sample Concentrator

Regularly performed maintenance includes, but is not limited to, the following:

- Check to ensure the gas supply is sufficient for the day's activity and delivery pressures are set as described in the SOP.
- Replace trap as needed.
- Decontaminate the system after running high concentration samples or as required by blank analysis.
- Check system for leaks when problem suspected.

Archons

Regularly performed maintenance includes, but is not limited to, the following:

- Visually inspect sampler for corrosion or significant water seepage past plunger.
- Check system pressure (denotes leaks).
- Check for sufficient standard materials in standard vials.

Accelerated Solvent Extractor (ASE)

Regularly performed maintenance includes, but is not limited to, the following:

- Check for leaks at the pump solvent reservoir, valves and other components.
- Inspect needle alignment of source needle.
- Check alignment of autoseal arms.
- Change peek seals and o-rings on cell caps after about 50 extractions per cell.
- Inspect cell edges for nicks and gouges on cell body.
- Inspect stainless steel frits and sonicate in solvent if needed.

TABLE 1, cont'd

ROUTINE MAINTENANCE SCHEDULE FOR ORGANICS INSTRUMENTATION

Horizon SPE Automated Extractor System

Regularly performed maintenance includes, but is not limited to, the following:

- Check and clean sensors with a KIMWipe.
- Change sensors as needed.
- Purge system with solvent before use and after use.
- Clean system with hot water by running method 15 after samples are analyzed and before purging system.

HPLC

Regularly performed maintenance includes, but is not limited to, the following for HPLC instrumentation:

- Check and sonicate pump valves as needed.
- Backflush column as needed.
- Replace analytical column or guard column as needed.
- Sonicate and replace solvent with every use.
- Replace the UV lamp as needed.
- Check and replace seal-pak as needed

TABLE 2

ROUTINE MAINTENANCE SCHEDULE FOR INORGANICS INSTRUMENTATION

ICP/MS

Regularly performed maintenance includes, but is not limited to, the following for ICP instrumentation:

- Clean torch assembly and spray chamber when discolored.
- Clean nebulizer as needed.
- Check to ensure that the argon supply is sufficient for the day's activity, and that delivery pressures are set as described in the SOP. Change argon tanks as necessary.
- Replace peristaltic pump tubing when it becomes stretched or develops flat spots.
- Check coolant water level weekly; replenish as necessary.
- Check rinse solution level daily; replenish as necessary.
- Check waste container level daily; empty as necessary.
- Check cleanliness of instrument air filters weekly; clean or replace as necessary.
- Check instrument computer date and time at the start of each day; correct as necessary.
- Check condition of vacuum pump oil weekly, replace as necessary.
- Clean sampling and skimmer cones every 2-4 weeks.
- Clean extraction lenses monthly, or as needed.
- Clean Einzel lenses every 3-6 months.

ICP

Regularly performed maintenance includes, but is not limited to, the following for ICP instrumentation:

- Clean torch assembly and spray chamber when discolored.
- Clean nebulizer as needed.

TABLE 2, cont'd

ROUTINE MAINTENANCE SCHEDULE FOR INORGANICS INSTRUMENTATION

ICP, cont'd

- Check to ensure that the argon supply is sufficient for the day's activity, and that delivery pressures are set as described in the SOP. Change argon tanks as necessary.
- Replace peristaltic pump tubing when it becomes stretched or develops flat spots.
- Check coolant water level weekly; replenish as necessary.
- Check rinse solution level daily; replenish as necessary.
- Check waste container level daily; empty as necessary.
- Check instrument computer date and time at the start of each day; correct as necessary.

Mercury Analyzer

Regularly performed maintenance includes, but is not limited to, the following:

- Replace drying tube as necessary
- Replace peristaltic pump tubing when it becomes stretched or develops flat spots.
- Replace mercury lamp as necessary.
- Clean optical cell quarterly or as needed.
- Clean liquid/gas separator when it becomes cloudy. Replace as needed.
- Check waste container level before each use; empty as necessary.
- Check exhaust system integrity before each use; correct as necessary.
- Check instrument computer date and time at the start of each day; correct as necessary.

TABLE 2, cont'd

ROUTINE MAINTENANCE SCHEDULE FOR INORGANICS INSTRUMENTATION

Lachat Autoanalyzer

Regularly performed maintenance includes, but is not limited to, the following:

- Check pump tubing; replace as needed.
- Clean interference filter with Kimwipe.
- Check reagent levels and expiration dates; refill or replace as needed.
- Rinse manifolds with water after analysis.
- Check manifold board surfaces; clean as needed by running under tap water.
- Check supplies and reagents; order as needed
- Check for leaks
- Check autosampler and autosampler trays;clean and/or lubricate as needed.
- Check fittings and o-rings on boards; replace as needed.
- Check peristaltic pump rollers; clean and lubricate as needed.
- Inspect manifold tubing for kinks and/or stains; replace as needed.
- Inspect valve flares; clean or replace as needed.
- Inspect reagent and waste lines; replace as needed.
- Check flow cell; clean as needed with Kimwipe.

Konelab AutoAnalyzer

Regularly performed maintenance includes, but is not limited to, the following:

- Rinse and refill distilled water container weekly.

TABLE 2, cont'd

ROUTINE MAINTENANCE SCHEDULE FOR INORGANICS INSTRUMENTATION

Konelab AutoAnalyzer, cont'd

- Check cleanness of segments weekly.
- Wash reagent tubes monthly.
- Change lamp as needed.
- Change diluent and wash tubes as needed.
- Change mixing paddles as needed.
- Change syringes as needed.
- Change dispensing needles as needed.
- Change drain and waste tubes as needed.
- Check used cuvettes and waste daily.

TOC Combustion Analyzer

Regularly performed maintenance includes, but is not limited to, the following:

- Check level of dilution water, drain vessel water, humidifier water, autosampler rinse water, and phosphoric acid vessel and fill as needed.
- Replace oxygen cylinder as needed.

IC

Regularly performed maintenance includes, but is not limited to, the following for IC instrumentation:

- Check regenerate pump tubing and replace as needed.
- Clean or regenerate column as needed.

TABLE 2, cont'd

ROUTINE MAINTENANCE SCHEDULE FOR INORGANICS INSTRUMENTATION

IC, cont'd

- Replace analytical column or guard column as needed.
- Change suppressor as needed.

Miscellaneous Instrumentation

Regularly performed maintenance includes, but is not limited to, the following:

- Replace spectrophotometer lamps as needed.
- Inspect DO probe membrane for tears and check for air bubbles under the membrane.
- Replace the DO probe membrane cap and electrolyte solution as needed.
- Clean pH electrode as needed.
- Change the autotitrator filling solution as needed.
- Clean, check, calibrate to manufacturers' specifications all pH, DO, conductivity, and turbidity meters annually (minimum); clean, check, calibrate to manufacturers' specifications all spectrophotometers biannually.

TABLE 3

ROUTINE MAINTENANCE SCHEDULE FOR GENERAL LABORATORY EQUIPMENT

General Laboratory Areas

- Clean and calibrate balances biannually (minimum).
- Check balance calibration each day of use.
- Clean balance pan prior to each use.
- Calibrate automatic pipettes with each use.
- Calibrate thermometers yearly against an NIST traceable thermometer; calibrate heavily used digital thermometers quarterly.
- Record refrigerator, freezer, and oven temperatures each weekday.
- General housekeeping: keep counter tops, hoods, and floors clean.
- Check airflow in hoods once a week.

TITLE: EQUIPMENT MAINTENANCE

ATTACHMENT A

ANALYTICAL EQUIPMENT LIST

ANALYTICAL INSTRUMENTATION

KEY INSTRUMENTATION	DATE IN SERVICE	USED FOR ANALYSIS	Instrument Identification
Hewlett Packard 5973 GC/MS with EPC, Archon Autosampler with foam sensor option and EST ENCON Evolution Purge and Trap Concentrator	**2008**	5030/8260, 5035/8260, 624, Current versions of CLP SOWs (OLM, OLC, & SOM) (VOA), & 524.2	D
Hewlett Packard 5972 GC/MS with EPC, Centurion Autosampler with foam sensor option and EST ENCON Purge and Trap Concentrator	**2006**	5030/8260, 5035/8260, 624, Current versions of CLP SOWs (OLM & SOM) (VOA)	T
Hewlett Packard 5972 GC/MS with EPC; Tekmar LSC-3100 Purge and Trap concentrator; Archon autosampler capable of low soils per Method 5035.	**1998**	5030/8260, 5035/8260, 624, Current versions of CLP SOWs (OLM, OLC, & SOM) (VOA), & 524.2	S
Hewlett Packard 5972 GC/MS with EPC; Tekmar LSC - 3000 purge and trap concentrator; Archon autosampler capable of low soils per Method 5035.	**1996**	5030/8260, 5035/8260, 624, Current versions of CLP SOWs (OLM, OLC, & SOM) (VOA), & 524.2	Z
Hewlett Packard 5972 GC/MS with EPC; Encon Purge and Trap concentrator; Archon autosampler capable of low soils per method 5035.	**1995**	5030/8260, 5035/8260, 624, Current versions of CLP SOWs (OLM, OLC, & SOM) (VOA), & 524.2	M
Hewlett Packard 5972 GC/MS with EPC; Tekmar LSC-3000 Purge and Trap concentrator; Archon autosampler capable of low soils per Method 5035.	**1993**	5030/8260, 5035/8260, 624, Current versions of CLP SOWs (OLM, OLC, & SOM) (VOA), & 524.2	F
Hewlett Packard 5973 GC/MS with EPC and Model 6890 autosampler.	**2008**	8270, 625 & Current versions of CLP SOWS (OLM, OLC, & SOM) (SVOA)	G
Hewlett Packard 5973 GC/MS with EPC and Model 6890 autosampler.	**2006**	8270, 625 & Current versions of CLP SOWS (OLM, OLC, & SOM) (SVOA)	R

TITLE: EQUIPMENT MAINTENANCE

ATTACHMENT A

ANALYTICAL EQUIPMENT LIST, cont'd

Katahdin ANALYTICAL SERVICES

ACCREDITED IN ACCORDANCE WITH nelac

ANALYTICAL INSTRUMENTATION

KEY INSTRUMENTATION	DATE IN SERVICE	USED FOR ANALYSIS	Instrument Identification
Hewlett Packard 5973 GC/MS with EPC and Model 6890 autosampler.	**1999/ 2001**	8270, 625 & Current versions of CLP SOWS (OLM, OLC, & SOM) (SVOA)	U
Hewlett Packard Model 5890 gas chromatograph with a flameionization detector; Agilent Technologies G1888 Network Headspace Analyzer.	**1991/ 2005**	Methane, Ethane and Ethene	GC05
Hewlett Packard Model 5890 gas chromatograph with EPC and dual electron capture detectors(ECD); Hewlett Packard Model 7673 autosampler.	**1993**	504, 556, 608, 8011, 8081, 8082, 8151	GC08
Hewlett Packard Model 5890 gas chromatograph with EPC and dual electron capture detectors(ECD); Hewlett Packard Model 7673 autosampler.	**1993**	8081, 8082, 608	GC06
Hewlett Packard Model 5890 gas chromatograph with dual electron capture detectors (ECD); Hewlett Packard Model 7673A autosampler.	**1988**	504, 8011, 8151	GC03
Hewlett Packard Model 5890 gas chromatograph with EPC and dual flame ionization detectors; Hewlett Packard Model 7673 autosampler.	**1993/ 1996**	8015 MOD., MAEPH, DRO	GC10
Hewlett Packard Model 5890 gas chromatograph with flame ionization detector; Tekmar ALS 2016 autosampler; Tekmar LSC 2000 purge and trap.	**1991**	8015 MOD., GRO	GC04
Hewlett Packard Model 5890 gas chromatograph with flame ionization detector and photo ionization detector; Tekmar ALS 2016 autosampler; Tekmar LSC 3000 purge and trap.	**1988**	8015 MOD., MAVPH, GRO	GC09

ATTACHMENT A

ANALYTICAL EQUIPMENT LIST, cont'd

ANALYTICAL INSTRUMENTATION

KEY INSTRUMENTATION	DATE IN SERVICE	USED FOR ANALYSIS	Instrument Identification
Hewlett Packard Model 5890 gas chromatograph with flame ionization and nitrogen-phosphorous detectors; Hewlett Packard Model 7673 autosampler.	**1992/ 1996**	ALCOHOLS, DMF, GLYCOLS	GC11
Hewlett Packard Model 5890 gas chromatograph with EPC and dual flame ionization detectors; Hewlett Packard Model 7673 autosampler.	**1987**	8015 MOD., MAEPH, DRO	GC12
Agilent Model 6890 gas chromatograph with dual EPC injection ports and micro electron capture detectors; Agilent Model 7683 autosampler	**2000**	8081, 8082 (including congeners), 608, current versions of CLP SOWs (OLM, OLC, & SOM) (P/P)	GC07
Hewlett Packard series 1100 HPLC with Quaternary pump, Diode Array detector and autosampler	**2008**	8330, 8332	HPLC03
ABC Autoprep 1000 GPC with UVD-1 ultraviolet detector and chart recorder.	**1994**	Current versions of CLP SOWS (OLM, OLC, $ SOM) (GPC-3640)	GPC01
Horizon SPE-DEX 4790 Automated Extractor System equipped with 4 extractors.	**2001/ 2005**	3535 method developement	Horizon #1
Horizon SPE-DEX 3000 Automated Extractor System equipped with 3 extractors.	**2006**	1664, 9070, 9071	Horizon #2
Thermo ICAP 6500 ICP Emission Spectrometer with autosampler.	**2006**	6010, 200.7, ILM05.4	I
Agilent 7500a ICP-MS with autosampler.	**2007**	6020, 200.8, ILM05.4	J
CETAC M-6100 Automated Mercury Analyzer with Autosampler	**2004**	7470/7471, 245.1, 245.5, ILM05.4	H

TITLE: EQUIPMENT MAINTENANCE

ATTACHMENT A

ANALYTICAL EQUIPMENT LIST, cont'd

ANALYTICAL INSTRUMENTATION

KEY INSTRUMENTATION		DATE IN SERVICE	USED FOR ANALYSIS	Instrument Identification
Tekran Series 2600 automated mercury analyzer with gold amalgam preconcentration and atomic fluorescence detector; Model 2620 autosampler.		**2000**	Ultra-trace level mercury (1631)	G
CPI ModBlock™ Metals Digestion Unit – Two 48 Place Units.		**2000**	Metals Aqueous Digestions	Digestion Unit #1 Digestion Unit #2
LACHAT Quickchem 8000 Continuum Series – Automated Ion Analyzer and autosampler.		**2000**	Various	WC1
Shimadzu TOC-V Combustion Analyzer, PC Controlled, High Sensitivity, Auto-Aqueous TOC Autosampler w/ 40 mL vials; Model SSM-5000A Solid Sample Module.		**2002**	TOC	WC2
Waters 717 Plus Autosampler, Waters 431 conductivity detector, Spectra System P4000 pump.	Dionex IonPak AS4A column; Dionex AMMS III chemical suppression system	**1991/ 2004**	Ion chromatography – Various Anions	See "Key Instrumentation " column for identification
	Dionex AS16 column; Dionex ultra ASRS electronic suppression system.		Ion Chromatography - Perchlorate	See "Key Instrumentation " column for identification
Scanning Fluorescence Detector		**1997**	Out of Service	NA
10 position Lab Crest Cyanide Midi-Distillation system.		**1998**	Cyanide	WC3
Spectronic 401 spectrophotometer.		**2000**	Various	WC4
HACH Turbidimeter, Model 2100A.		**1993**	Turbidity	WC6

ATTACHMENT A

ANALYTICAL EQUIPMENT LIST, cont'd

ANALYTICAL INSTRUMENTATION

KEY INSTRUMENTATION	DATE IN SERVICE	USED FOR ANALYSIS	Instrument Identification
Accumet pH/Conductivity Meter, Model 20.	**1998**	Various	WC8
Mettler DL25 Autotitrator & Mettler ST20A sample changer.	**1993**	Alkalinity	WC9
YSI Dissolved Oxygen Meter.	**1990**	Dissolved Oxygen & Biochemical Oxygen Demand	WC10
Konelab 20 Multi-Wavelength Photometric Analyzer.	**2003**	Various	Konelab #1
Dionex Accelerated Solvent Extractor ASE 200.	**2003**	3545	ASE #1
Waters HPLC system with 600E multisolvent delivery system, 717 plus autosampler, Spectra System UV200 detector	**2003/2004**	Food Testing	HPLC01
Thermo Spectronic Genesys 10uv spectrophotometer	**2005**	Food Testing	Food #1
DryVap Concentrator System 5000	**2005**	GC and GC/MS Extractables	DryVap #1

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-604
Revision History
Cover Page
Page 1

TITLE: ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP ANALYSIS OF TOTAL OR DISSOLVED METALS

Prepared By: George Brewer Date: 11/97

Approved By:

Group Supervisor: George Brewer Date: 01/19/01

Operations Manager: John C. Burton Date: 1/22/01

QA Officer: Deborah J. Nadeau Date: 1·22·01

General Manager: Duncan F. Keegan Date: 1/22/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 3010A	Format changes, added pollution prevention, block digester; revised database references; revised and added tables.	DN	1·22·01	1/22/01
02 3010A	Added wording allowing use of digestates for ICP-MS analysis. Added use of block digester as primary heating source & adjusted volumes. Revised standard solution names & concs. in Figures 3&4.	DN	8·29·02	8·29·02
03	Added Uranium to spiking solutions for LCS & MS/D. Removed the Internal Custody Record for Metals Digestates figure and reference.	LAD	04/06	04/06
04	Minor changes to Section 7 to reflect current practices. Updated Figure 1 - Sample Prep Logbook. Updated Figure 2 and 3 - Spike amounts.	LAD	05/09	05/09

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ____ of document **SOP CA-604-04**, titled **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ____ of document **SOP CA-604-04**, titled **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**.

Recipient: _____ Date: _____

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedure utilized by Katahdin Analytical Services, Inc. personnel to solubilize metals in aqueous samples, wastes that contain suspended solids, and mobility-procedure extracts prior to analysis by inductively coupled plasma atomic emission spectroscopy (ICP) and inductively coupled plasma mass spectrometry (ICP-MS). This SOP applies to samples prepared by EPA Method 3010, with the method modifications mentioned in Table 2.

1.1 Definitions - none.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the acid digestion of aqueous samples by EPA Method 3010. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in the acid digestion of aqueous samples using EPA Method 3010 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their work in the appropriate lab notebook. Any deviations from the method or irregularities with the samples should also be recorded in the lab notebook and reported to the Supervisor or designated qualified data reviewer responsible for these data.

It is the responsibility of the Supervisor to ensure that technical personnel perform acid digestions in accordance with this SOP and to confirm that their work is properly documented through periodic review of the associated logbooks.

1.3 Safety

The acids used in this procedure are highly corrosive and reactive, and spiking standards contain toxic metals. The toxicity and reactivity of client samples are usually unknown, so samples should always be assumed to present a contact hazard. To reduce or eliminate exposure to potentially harmful chemicals, lab coats, gloves, and safety glasses or goggles must be worn whenever handling samples or reagents. Additional safety apparel, including face shields, rubber aprons, dust masks, and rubber shoe protectors, is available in the metals prep lab and should be worn whenever circumstances warrant.

Acids should be added to samples slowly and carefully while watching for reactions. This should be done under a hood, in case harmful fumes are evolved.

Hood sashes should be lowered as far as possible whenever beakers are being heated in the hood. Use caution when handling hot beakers.

Each qualified analyst or technician must be familiar with Katahdin Analytical Health and Safety Manual including the Katahdin Chemical Hygiene Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of a respirator and all safety equipment. Each analyst shall receive a safety orientation from their supervisor, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Excess spiking solutions must be emptied into the corrosive waste carboy located in the metals prep lab for subsequent appropriate disposal in accordance with the Chemical Hygiene Plan and Safety Manual.

Sample digestates should be stored for a minimum of 60 days after digestion to allow for analysis, and reanalysis if necessary. Digestates older than 60 days may be emptied into the corrosive waste carboy in the metals prep lab for subsequent appropriate disposal in accordance with the Chemical Hygiene Plan and Safety Manual.

Any other wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Chemical Hygiene Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision.

2.0 SUMMARY OF METHOD

The aqueous sample is refluxed with nitric acid in a covered digestion vessel. Additional nitric acid is added until the color of the digestate has stabilized. After the digestate has been evaporated to a low volume, it is refluxed with hydrochloric acid and diluted to the appropriate final volume with reagent water.

Samples may be concentrated (i.e. final digestate volume less than initial sample volume) during digestion if lower detection limits are required. Volumes of reagents and spiking standards must be added in proportion to the final volume of the digestate. Because concentration of samples during digestion increases the concentrations of dissolved solids

and may exacerbate analytical interferences, concentration factors greater than 5 are not recommended.

3.0 INTERFERENCES

Interferences are discussed in the applicable analytical SOPs.

4.0 APPARATUS AND MATERIALS

4.1. 250 mL and 400 mL pre-cleaned Griffin beakers (cleaned according to the current revision of SOP CA-100, "Labware Cleaning") for digestion using a hot plate. If digestion will be performed using a block digester, 70ml graduated, polyethylene block digester tubes (with attached snap caps) will be used instead of glass beakers.

4.2 Ribbed watch glasses. If digestion is performed using a hot plate, 75 mm diameter and 100 mm diameter glass watch glasses (pre-cleaned as above) are used. If digestion is performed using a block digester, 40mm diameter disposable polyethylene watch glasses are used.

4.3 Adjustable volume automatic pipets covering the range from 10 uL to 1000 uL and disposable pipet tips; calibrated Finn pipets or Eppendorf pipets are acceptable.

4.4 Disposable graduated polystyrene specimen containers with pouring lips, 200 mL capacity.

4.5 Hot plate, block digester, or other heating source - adjustable and capable of maintaining a temperature of 90-95°C. Hot plates must be numbered for easy identification.

4.6 Device for measuring hot plate temperature. This may consist of a heat-resistant 100ml beaker containing reagent water in which a thermometer is immersed. When using a block digester, a digestion tube containing reagent water in which a thermometer is immersed may be used. The temperature of one hot plate is measured each day, on a rotating basis. The hot plate identification number and the measured temperature are recorded on the sample preparation logbook sheet.

4.7 Plastic funnels, pre-cleaned as in Section 4.1.

4.8 Filter funnel holders, capable of suspending plastic funnels above disposable specimen containers.

4.9 Polyethylene wash bottles for dispensing reagent water and 5% HNO_3.

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

4.10 Filter paper, Whatman No. 41 or equivalent. Filters are acid-washed immediately prior to use as follows. Place a pre-cleaned funnel in the funnel holder and put a disposable plastic specimen container under the funnel to collect the rinsates. Place a folded filter in the funnel and rinse three times with approximate 10 mL volumes of 5% HNO_3, making sure the entire surface of the filter is wetted each time and allowing each rinse to drain completely before continuing. Then rinse three times with approximate 25 mL volumes of reagent water. Discard the rinsates into the appropriate waste container. The acid-washed filter is now ready for use.

4.11 Polyethylene sample containers with screw caps or graduated polyethylene sample containers with attached snap lids, 125 mL capacity. These are not necessary when using the block digester since the final digestates are stored in the digestion tubes.

4.12 Repipetters (adjustable repeating pipetters with reservoirs) for dispensing concentrated nitric acid and 1:1 HCl.

5.0 REAGENTS

5.1 Concentrated nitric acid, HNO_3 – trace metals grade.

5.2 Concentrated hydrochloric acid, HCl – trace metals grade.

5.3 Reagent water - water that meets the performance specifications of ASTM Type II water (ASTM D1193).

5.4 Hydrochloric acid, 1:1. Add a volume of concentrated hydrochloric acid to an equivalent volume of reagent water and swirl gently to mix.

5.5 Nitric acid, 5% v/v. Add 25 mL concentrated HNO_3 to 475 mL reagent water in a 500 mL wash bottle. Cap, point the dispensing tip into a sink, and shake gently to mix.

5.6 Multi-element spiking solutions (as listed in Figure 3).

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples to be analyzed for dissolved metals should be filtered through a 0.45 um membrane filter and preserved as soon as possible after collection. Samples to be analyzed for total metals should be preserved, unfiltered, as soon as possible after collection. Aqueous samples are preserved by acidification with nitric acid to a pH of <2.

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

7.0 PROCEDURES

7.1 Prior to performing the digestion, make a list of the samples that are to be digested. Enter digestion information (Katahdin Sample Numbers, QC Batch ID, preparation date, analyst initials, etc.) into the ACCESS computer spreadsheet. Print out a copy of the spreadsheet. With a permamament marker, make sample labels and attach to the polyethylene sample containers that will contain the digestates.

7.2 If using glass beakers as the digestion vessels, submerge previously cleaned beakers three times into a 10% nitric acid bath, then rinse three times with reagent water. The polyethylene digestion tubes used in conjunction with the block digester do not require acid rinsing or precleaning. Label the digestion vessels with sample numbers.

7.3 If digestion is performed using a block digester, the sample aliquot may be measured in the digestion vessel using the graduations on the digestion tubes. Measure 50 ml of well-mixed sample into a 70 ml block digestion tube. A larger sample aliquot may be used (up to 250 mL) if concentration of the sample during digestion is desired. Sample volumes larger than 50 mL may be digested in 250 mL beakers. Measure aliquot of well-mixed sample into a graduated specimen cup and transfer into a properly cleaned 250 mL beaker. Sample volumes of more than 50ml may not be digested using the 70ml block digester tubes. The volumes of reagents and spiking solutions used must be adjusted in proportion to the final digestate volume. The reagent and spiking solution volumes listed below are based on a final volume of 50 mL.

7.4 Add spike solutions to matrix spike samples and laboratory control samples (refer to Figure 3 for spiking instructions).

7.5 Use a repipetter to add 1.5 mL of concentrated HNO_3 (per 50 mL final volume) to the sample. Cover with a ribbed watch glass and place on heatsource. Heat cautiously, without boiling the sample, and evaporate to a low volume (10 - 15 mL).

<u>NOTE</u>: Do not allow any portion of the bottom of the digestion vessel to go dry during any part of the digestion. If a sample is allowed to go to dryness, low recoveries may result. Should this occur, discard the digestate and re-prepare the sample.

7.6 Cool the sample and add another 1.5 mL aliquot (per 50 mL final volume) of concentrated HNO_3. Cover and resume heating, increasing the temperature until a gentle reflux action occurs.

7.7 Continue heating, adding additional acid as necessary, until the digestate is light in color or does not change in appearance with continued refluxing.

7.8 Evaporate digestate to a low volume (10 - 15 mL).

7.9 Cool the sample and use a repipetter to add 5 mL (per 50 mL final volume) of 1:1 HCl. Cover the sample and resume heating, refluxing for an additional 15 minutes to dissolve any precipitate or residue resulting from evaporation.

7.10 Allow the sample to cool.

7.11 If the digestate contains visible particulate material, it must be filtered. Use a pre-cleaned funnel and acid-rinsed filter paper to filter the digestate into a clean graduated plastic specimen container or block digester digestion tube. Using a wash bottle, rinse the digestion vessel with reagent water and add the rinsates to the filter apparatus. After all of the liquid in the filter has drained into the specimen container or digestion tube, thoroughly rinse the filter three times with small (5-10 mL) volumes of reagent water, allowing the liquid to drain completely after each rinse.

If the digestion was performed using hot plates and the digestate does not contain particulate material, simply decant the digestate into a clean graduated specimen container (or graduated sample container with attached snap lid), rinse the beaker with reagent water, and add the rinsates to the container.

If the digestion was performed using a block digester and the digestate contains no visible particulate material, the digestate may be brought to final volume and stored in the digestion tube without decanting or rinsing.

7.12 Using the graduations on the specimen container, snap-lid container or digestion tube, dilute to the required final volume with reagent water. If a specimen container has been used, transfer the contents to the corresponding labeled polyethylene sample bottle, cap the bottle, and discard the empty specimen container. If a snap-lid container or digestion tube has been used, close and secure the snap-lid. Shake the container gently to mix. The digestate is now ready for analysis.

7.13 Review the ACCESS computer spreadsheet for accuracy. If any information is incorrect, make the necessary changes to the computer spreadsheet and print out a corrected copy. Do not discard the original copy of the spreadsheet. Record (hand write) the sample bottle ID, reagent lot numbers, spiking information, initial and final volumes, hot plate ID and hot plate temperature in the appropriate spaces on the spreadsheet. Record any method deviations, irregularities with the samples, or other pertinent observations at the bottom of the page, and sign and date the spreadsheet. Bind all copies of the spreadsheet in the sample preparation log. An example sample preparation logbook page (ACCESS spreadsheet) is included as Figure 1.

7.14 Place each batch of digestates in a box labeled with the QC Batch ID, and put the box of digestates in the metals digestates storage area.

7.15 A condensation of the procedure described above is included in this SOP as Table 3. A controlled copy of this table may be posted in the metals preparation laboratory for reference by the analyst.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

8.1 At least one preparation blank for waters (PBW) is processed concurrently with each digestion batch of 20 or fewer samples, and is used to assess contamination resulting from the digestion procedure. The PBW consists of an aliquot of reagent water that is digested using the same reagents as those used to digest associated samples. The initial and final volumes of the PBW must be identical to those of the associated samples (i.e., if the associated samples were concentrated during digestion, the PBW must also be concentrated). Refer to the appropriate analytical SOP for PBW acceptance criteria and corrective actions.

8.2 At least one laboratory control sample for waters (LCSW) is processed concurrently with each digestion batch of 20 or fewer samples. The LCSW consists of an aliquot of reagent water that is spiked to contain all analytes of interest at known concentrations, and is digested using the same reagents as those used to digest associated samples. The initial and final volumes of the LCSW must be identical to those of the associated samples (i.e., if the associated samples were concentrated during digestion, the LCSW must also be concentrated). Directions for spiking the LCSW are contained in Figures 3 and 4. The measured analyte recoveries for the LCSW are used to assess digestion method performance. Refer to the appropriate analytical SOP for LCSW recovery acceptance criteria and corrective actions.

8.3 Matrix spiked samples are processed concurrently with each digestion batch at a minimum frequency of one per digestion batch. A matrix spike sample consists of an aliquot of a sample that is spiked with known amounts of all analytes of interest. Matrix spike recoveries are used to assess the effects of sample matrix on digestion and analysis performance. Directions for spiking matrix spike samples are contained in Figures 3 and 4. Refer to the appropriate analytical SOP for matrix spike recovery acceptance criteria and corrective actions.

8.4 Matrix spiked duplicate samples are processed concurrently with each digestion batch at a minimum frequency of one per digestion batch. Matrix spiked duplicate samples are used to assess the precision of the digestion and analysis methods. Refer to the appropriate analytical SOP for matrix spike duplicate precision acceptance criteria and corrective actions.

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

NOTE: Clients may choose specific samples for matrix spike and matrix spike duplicate analysis; otherwise, the choice is left to the person performing the digestion. The sample volumes available may restrict the choice of samples used for matrix spike and duplicate digestion. Field blank samples should not be chosen for matrix spike and matrix spike duplicate analysis.

8.5 The quality control measures and frequencies described above are minimum requirements. They are summarized for reference in Table 1. Individual clients and analytical programs may impose additional QC requirements.

9.0 METHOD PERFORMANCE

Refer to the applicable analytical SOPs for method performance information.

10.0 APPLICABLE DOCUMENTS/REFERENCES

"Test Methods for the Evaluation of Solid Waste," United States Environmental Protection Agency, SW-846, Third Edition, Final Update III, 12/96, Method 3010A.

LIST OF TABLES AND FIGURES

Table 1 QC Requirements
Table 2 Summary of Method Modifications
Table 3 Procedure Condensation
Figure 1 Example Page From Metals Sample Preparation Logbook
Figure 2 Preparation of Matrix Spikes, LCSs, and Spiking Solutions: Method 3010
Figure 3 Element Concentrations in Matrix Spikes, LCSs, and Spiking Solutions: Method 3010

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

TABLE 1

QC REQUIREMENTS

Analytical Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
3010	Preparation Blank for Waters (PBW)	One per prep batch of 20 or fewer samples	Refer to analytical method	Refer to analytical method
	Laboratory Control Sample for Waters (LCSW)	One per prep batch of 20 or fewer samples	Refer to analytical method	Refer to analytical method
	Matrix Spike Sample	One per prep batch	Refer to analytical method	Refer to analytical method
	Matrix Spike Duplicate Sample	One per prep batch	Refer to analytical method	Refer to analytical method
	Demonstration of analyst proficiency; accuracy and precision	One time demonstration by each analyst performing the method	Must pass all applicable QC for method	Repeat analysis until able to perform passing QC; document successful performance in personal training file

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-604-04	EPA METHOD 3010, current revision
Apparatus/Materials	1) Disposable plastic specimen cup used to measure sample volume. 2) Digestion performed in 250 mL, 400 mL Griffin beaker, or 70ml digestion tube to facilitate evaporation. 3) Ribbed watch glass used throughout digestion to reduce contamination.	1) Graduated cylinder used to measure sample volume. 2) Digestion performed in 150 mL Griffin beaker. 3) Ribbed and non-ribbed watch glasses alternated in digestion.
Procedures	1) Digestate may be analyzed for antimony and silver. 2) Sample aliquots larger or smaller than 100 mL may be used. 3) Sample evaporated to 10 - 15 mL.	1) Digestate may not be analyzed for antimony and silver. 2) Requires sample aliquot of 100 mL. 3) Sample evaporated to 5 mL.

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

TABLE 3

PROCEDURE CONDENSATION: EPA METHOD 3010

1. If performing digestion on a hot plate, rinse glass beakers and ribbed watch glasses 3 times in acid bath. Then rinse beakers and watch glasses 3 times with reagent water. If performing digestion with block digester, polyethylene digestion tubes do not require precleaning.
2. Label digestion vessels with sample numbers.
3. Mix sample well, measure 50 mL (or smaller or larger aliquot) into a polyethylene digestion tube. If using glass beakers, measure aliquot into graduated specimen container, and transfer to appropriate digestion vessel.
4. Add spike solutions to matrix spike samples and LCSW (refer to Figure 3 of this SOP).
5. Add 1.5 mL (per 50 mL final volume) concentrated HNO_3 to sample.
6. Cover with a ribbed watch glass.
7. Place on heating device (hotplate or block digester) and evaporate to 10 - 15 mL.
8. Cool sample and add another 1.5 mL (per 50 mL final volume) concentrated HNO_3.
9. Resume heating until gentle reflux action occurs.
10. Continue heating, adding additional HNO_3 as necessary until digestion is complete.
11. Evaporate to 10 - 15 mL.
12. Cool sample and add 5 mL (per 50 mL final volume) 1:1 HCl. Resume heating and reflux gently for 15 minutes.
13. Cool sample and filter (if necessary) or decant into a graduated polyetheyne digestion tube. Rinse beaker with reagent water and filter or decant rinsate into specimen container.
14. Dilute to appropriate final volume with reagent water.
15. Cap sample container and shake gently to mix.

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

FIGURE 1

EXAMPLE PAGE FROM METALS SAMPLE PREPARATION LOGBOOK

Katahdin Analytical Services, Inc. — Metals Preparation Benchsheet

Reagent Information:
JT Baker HNO3: C20022 JT Baker HCL: C07046 Ashland H2O2: NA Method: 3010

Standards/Spiking Information:
I.V. CLPP-SPK-1 (ID/Vol): MS1362 / 0.05 mL NA : NA
CLPP-SPK-INT1 (ID/Vol): MW9975 / 0.5 mL Hot Plate No.: A
CLPP-SPK-INT2 (ID/Vol): MW9930 / 0.5 mL Temp.: 95 °C
CL-Spike (ID/Vol): MS1332 / 0.05 mL
Spiking Witnessed by: NA

REVIEWED
EM 06-27-06
KATAHDIN ANALYTICAL METALS SECTION

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Initial Color	Initial Clarity	Final Color	Final Clarity	Artifacts	Bottle
LC2WWF27ICW1	WF27ICW1	0.05	L	0.05	L	AQ	IC	DJJ	06/27/2006	N/A	N/A	N/A	N/A	N/A	N/A
LCSWWF27ICW1	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006	N/A	N/A	N/A	N/A		
PBWWF27ICW1	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006	N/A	N/A	N/A	N/A		
WW3165-001	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-002	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-003	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-004	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-005	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-006	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-007	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-008	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-009	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-010	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-011	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-012	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-013	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-014	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-015	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-016	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-017	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-018	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-019	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						
WW3165-020	WF27ICW1		L		L	AQ	IC	DJJ	06/27/2006						

DJJ 6/27/06

Digestion performed by: DJJ On: 6/27/06 Page: WF061 Revision: 00

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

FIGURE 2

PREPARATION OF MATRIX SPIKES, LABORATORY CONTROL SAMPLES, AND SPIKING SOLUTIONS FOR DIGESTION OF AQUEOUS SAMPLES BY METHOD 3010

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 50 mL Final Volume (mL)
Laboratory Control Sample (LCSW) and Matrix Spike	CLPP-SPK-1	Inorganic Ventures	0.050
	CLPP-SPK-INT1	Lab Prepared (see below)	0.50
	CLPP-SPK-INT2	Lab Prepared (see below)	0.50
	1000 mg/L Uranium Standard	Inorganic Ventures	0.005

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
CLPP-SPK-INT1	1000 mg/L Se	High Purity Standards	5.0
	1000 mg/L As	High Purity Standards	5.0
	1000 mg/L Pb	High Purity Standards	5.0
	1000 mg/L Cd	High Purity Standards	2.5
	1000 mg/L Sb	High Purity Standards	5.0
	10000 mg/L K	High Purity Standards	10.0
	10000 mg/L Na	High Purity Standards	7.5
	10000 mg/L Mg	High Purity Standards	5.0
	10000 mg/L Ca	High Purity Standards	2.5
CLPP-SPK-INT2	2007ICS-1	Inorganic Ventures	10.0
	1000 mg/L Sr	High Purity Standards	5.0
	1000 mg/L Sn	High Purity Standards	5.0
	10000 mg/L Si	High Purity Standards	5.0

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

FIGURE 3

ELEMENT CONCENTRATIONS IN MATRIX SPIKES, LABORATORY CONTROL SAMPLES, AND THEIR COMPONENT SPIKING SOLUTIONS FOR DIGESTION OF AQUEOUS SAMPLES BY METHOD 3010

Element	CONCENTRATION IN SOLUTION, mg/L							
	Matrix Spike	LCSW	CLPP-SPK-1	CLPP-SPK-4	CLPP-SPK-INT1	CLPP-SPK-INT2	2007 ICS-1	1000 mg/L U
Aluminum	2.000	2.000	2000					
Antimony	0.500	0.500		100	50			
Arsenic	0.500	0.500		4	50			
Barium	2.000	2.000	2000					
Beryllium	0.050	0.050	50					
Boron	0.500	0.500		50		50	500	
Cadmium	0.250	0.250		5	25			
Calcium	2.500	2.500			250			
Chromium	0.200	0.200	200					
Cobalt	0.500	0.500	500					
Copper	0.250	0.250	250					
Iron	1.000	1.000	1000					
Lead	0.500	0.500		2	50			
Magnesium	5.000	5.000			500			
Manganese	0.500	0.500	500					
Molybdenum	0.300	0.300		30		30	300	
Nickel	0.500	0.500	500					
Potassium	10.000	10.000			1000			
Selenium	0.500	0.500		5	50			
Silicon	5.230	5.230				523	230	
Silver	0.050	0.050	50					
Sodium	7.500	7.500			750			
Strontium	0.500	0.500		50		50		
Thallium	0.500	0.500		5	50			
Tin	0.500	0.500		50		50		
Titanium	1.000	1.000		100		100	1000	
Uranium	0.100	0.100						1000
Vanadium	0.500	0.500	500					
Zinc	0.500	0.500	500					

TITLE: TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010

Prepared By: George Brewer Date: 7/98

Approved By:

Group Supervisor: George Brewer Date: 01/23/01

Operations Manager: John C. Burton Date: 1/23/01

QA Officer: Deborah J. Nadeau Date: 1.23.01

General Manager: Duncan F. Hughes Date: 1/25/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 6010B	Format changes, added pollution prevention, expanded procedure and QC sections. Added tables.	DN	1.23.01	1/23/01
02 6010B	Calibration begins with analysis of S0 (cal. blank) followed by S1 (Mixed Cal. Std.) Changes to section 7.5 and Table 8 to reflect this. Made changes to element concs. in Tables 3, 4, 5, 6 to reflect current practices.	DN	10.21.02	10.21.02
03 6010B	Added MN-IEC to standards run. Changed frequency of LRS. Changed concentration of HNO_3 in calibration blank. CRI changed from three separate solutions to one. Changed CRI vendor.	MRC	04.15.04	04.15.04
04	updated ICV, CCV, ICB, PQL Chkstd. PBW, PBS, MS & MSD acceptance criteria updated Table 1	LAD	05/06	05/06
05	Updated Tables 3, 4, 5, 6 and 7 with current standard concentrations and prep. Updated Table 1 with current practices including NAUY audit findings. Updated sections 2, 7.2, 7.6 and Table 1 with new ICP information. Updated Table 8 with current sequence requirements.	LAD	07/07	07/07

TITLE: TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Added hardness definition and calculation (APP. 1)	LAD	09/07	09/07
07	Updated Summary to reflect new ICP functions. Removed ICP set-up updated tables to reflect changes in standard concentrations and preparation	LAD	11/08	11/08
08	Updates to Sections 8 and 10, Tables 1 and 2 to reflect changes from 6010B to 6010C. Added LLQC information and criteria to Sect. 8 and Table 1. Added criteria to analyze PQL standard at the beginning and END of each run.	LAD	02/09	02/09

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-608-08**, titled **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-608-08**, titled **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010.**

Recipient: _____ Date: _____

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

1.0 SCOPE AND APPLICATION

Inductively coupled plasma atomic-emission spectroscopy (ICP-AES) determines trace elements, including metals, in solution. The purpose of this SOP is to describe the procedures used by Katahdin Analytical Services, Inc. personnel to analyze aqueous and solid samples for trace metals by USEPA Method 6010 (Test Methods for Evaluating Solid Waste, Physical/ Chemical Methods, USEPA SW846).

Sample types that may be analyzed using these methods include drinking waters, ground waters, aqueous samples, TCLP, SPLP and EP Toxicity extracts, industrial and organic wastes, soils, sludges, sediments, and other solid wastes. The following elements may be analyzed under this SOP: Al, Sb, As, Ba, Be, B, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Mo, Ni, K, Se, Si, Ag, Na, Sn, Sr, Tl, Ti, V, and Zn.

All samples, except filtered ground water samples, analyzed under USEPA Method 6010 require digestion prior to analysis. USEPA Methods 3005, 3010, and 3050 describe appropriate digestion procedures for samples to be analyzed by ICP-AES under EPA Method 6010. Refer to current revisions of Katahdin SOPs CA-604 and CA-605, current revisions, for sample digestion procedures.

1.1 Definitions

Analytical Spike - An aliquot of a sample to which a known amount of analyte has been added before analysis and after digestion, if digestion is required.

CCB - Continuing Calibration Blank - An analyte-free solution consisting of acidified reagent water used to verify calibration accuracy periodically during analysis.

CCV - Continuing Calibration Verification - A midrange standard used to verify calibration accuracy periodically during analysis.

CRI - Contract Required detection limit sample for ICP - A low concentration standard used to verify calibration accuracy near the low end of the calibration range.

Duplicate - A second aliquot of a sample that is prepared and analyzed in the same way as the original sample in order to determine the precision of the method.

ICB - Initial Calibration Blank - An analyte-free solution consisting of acidified reagent water used to verify calibration accuracy.

ICP-AES - Inductively Coupled Plasma Atomic Emission Spectroscopy.

ICS - Interference Check Sample - Two standards (ICSA and ICSAB) used to verify the effectiveness of interelement correction and background correction. Solution ICSA contains only interferents (Al, Ca, Fe, and Mg) at high concentrations (200 to

500 mg/L); solution ICSAB contains interferents at the same concentrations as well as analytes at low (20 mg/L or less) concentrations.

ICV - Initial Calibration Verification - A standard made from a source independent from the calibration standards and with analyte concentrations different from those in the CCV; used to verify the accuracy of the instrument calibration.

IDL - Instrument Detection Limit - The lowest concentration of an analyte that can be determined with 99% confidence.

LCS - Laboratory Control Sample - A standard or solid reference material that has been brought through the sample preparation process.

LRS - Linear Range Standard - A high-concentration standard used to determine the upper reporting limit of the ICP calibration.

PB - Preparation Blank - Reagent water that has been brought through the sample preparation process.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the IDL.

Matrix Spike - An aliquot of a sample to which a known amount of analyte has been added before digestion.

Serial Dilution - The dilution of a sample by a factor of five. When corrected by the dilution factor, the measured analyte concentrations of the diluted sample should agree with those of the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents.

Hardness – The sum of the calcium and magnesium concentrations, both expressed as calcium carbonate, in mg/L.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in ICP analysis by EPA Method 6010. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in ICP analysis by Method 6010 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the group supervisor or designated qualified data reviewer responsible for this data.

It is the responsibility of the Group Supervisor to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

Samples, sample digestates, standards, and other reagents used in ICP analysis may contain high concentrations of acids and toxic metals. Safety glasses should be worn when changing or adjusting argon tanks.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Wastes from ICP analysis should be disposed of in a manner appropriate to the hazards they present. Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Analytical Environmental Health and Safety Manual I and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

2.0 SUMMARY OF METHOD

This method describes multielemental determinations by ICP-AES using simultaneous optical systems and radial and axial viewing of the plasma. The basis of the method is the measurement of atomic emission from sample atoms entrained in an argon plasma by optical spectroscopy. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where thermal excitation of entrained atoms and ions occurs. Characteristic atomic-line and ionic-line emission spectra are produced by a radio-frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating and the intensities of the emitted lines are monitored by a solid state charge injection device (CID) camera system. Photocurrents from the CID camera system are measured by a computer system. Element concentrations of unknown samples are quantitated by comparison of sample emission intensities to emission intensities of standards of known concentration. A background correction technique is used to compensate for variable background contribution to the determination of trace elements. Background is measured adjacent to the analyte lines on samples during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, has been determined by the complexity of the spectrum adjacent to the analytical line. The position used must be relatively free of spectral interference and must reflect the same change in background intensity as occurs at the analyte wavelength. Physical interferences are corrected through the use of an internal standard (yttrium) that is automatically added to all samples and standards prior to nebulization. The possibility of additional interferences (noted in section 3) must be recognized and appropriate corrections applied.

3.0 INTERFERENCES

Several types of interference effects may contribute to inaccuracies in the determination of trace elements. They can be summarized as spectral interferences, physical interferences, and chemical interferences.

Spectral interferences can be categorized as 1) overlap of a spectral line from another element; 2) unresolved overlap of molecular band spectra; 3) background contribution from continuous or recombination phenomena; and 4) background from stray light from the line emission of high concentration elements. The first of these effects is compensated by utilizing the computer correction of raw data, requiring the monitoring and measurement of the interfering element (interelement correction). The second effect is controlled by choosing analytical wavelengths that are free from overlapping molecular emission spectra. The third and fourth effects are usually compensated by a background correction adjacent to the analyte line. Uncorrected spectral interferences may be detected through examination of serial dilution and matrix spike data.

Physical interferences are generally considered to be effects associated with sample nebulization and transport processes. Such properties as changes in viscosity and surface tension can cause significant inaccuracies, especially in samples that may contain high dissolved solids and/or acid concentrations. Matrix matching of standards and samples

and the use of a peristaltic pump may lessen these interferences. If these types of interferences are operative, they must be reduced by dilution of the sample and/or utilization of standard addition techniques. Another problem that can occur from high dissolved solids is salt buildup at the tip of the nebulizer. This affects aerosol flow rate causing instrumental drift. Regular cleaning of nebulizer tips and dilution of samples with high dissolved solids contents are used to control this problem. Physical interferences are also corrected by this laboratory through the use of an internal standard. Uncorrected physical interferences may be detected through examination of serial dilution and matrix spike data. Instrument drift caused by the salting up of nebulizer tips may also be detected by looking for oriented drift in calibration verification standards analyzed regularly throughout the run.

Chemical interferences are characterized by molecular compound formation, ionization effects, and solute vaporization effects. Normally these effects are not pronounced with the ICP technique; however, if observed they can be minimized by careful selection of operating conditions (i.e., incident power, observation position, etc.), by matrix matching, and by standard addition procedures. These types of interferences can be highly dependent on matrix type and the specific analyte element. Uncorrected chemical interferences may be detected through examination of serial dilution data.

4.0 APPARATUS AND MATERIALS

4.1 Computer-controlled inductively-coupled plasma atomic emission spectrometer (plasma viewed radially or axially) equipped for internal standardization, and capable of performing automatic background correction and interelement correction. For more information refer to the current revision of Katahdin SOP CA-632, "Operation and Maintenance of the Thermo ICAP 6500 ICP Spectrophotometer".

4.2 Computer-controlled autosampler.

4.3 Argon gas supply – high purity.

4.4 Volumetric glassware of suitable precision and accuracy.

4.5 Automatic pipets of suitable precision and accuracy. Calibrated Eppendorf Reference pipets and Finn digital pipets are appropriate.

Refer to the appropriate instrument-specific SOP for additional required equipment.

5.0 REAGENTS

5.1 Hydrochloric acid, concentrated (HCl) – spectroscopic grade.

5.2 Nitric acid, concentrated (HNO_3) – spectroscopic grade.

5.3 Reagent water, trace metals free.

5.4 Calibration blank – reagent water containing HCl (5% v/v) and HNO_3 (5% v/v). Calibration blank solution is prepared in large volumes (up to 20 liters) and stored in a carboy. Calibration blank solution is used in establishing the analytical curve, and in all initial and continuing calibration blank determinations. This solution is also used to flush the system between standards and samples. Intermediate and working standards are prepared by diluting stock standards and intermediate standards with calibration blank solution so that all standards and blanks are acid matrix-matched to sample digestates.

5.5 Single element and multielement stock standard solutions – purchased standards prepared from high purity salts or metals, and supplied by the vendors with certificates of purity and analysis. Refer to Tables 3 and 4 for a listing of stock standards required, and to Table 7 for element concentrations in stock standards.

5.6 Intermediate standard solutions – laboratory-prepared multielement standards that are used in the subsequent preparation of working standards. Refer to Table 4 for a listing of intermediate standards required and for preparation instructions. Refer to Table 6 for element concentrations in intermediate standards.

5.7 Working standard solutions – laboratory-prepared multielement standards that are used to calibrate the instrument and to perform all necessary QC checks. Refer to Table 3 for a listing of working standards and for preparation instructions. Refer to Table 5 for element concentrations in working standards.

5.8 5 mg/L yttrium internal standard solution – add 0.5 mL 10000 mg/L yttrium stock standard to a 1000 mL volumetric flask half filled with calibration blank solution. Bring to volume with calibration blank solution.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples to be analyzed for trace metals by ICP should be collected and preserved as described in the following table.

Matrix	Container [1]	Collection Volume/ Weight	Preservation/ Treatment	Holding Time
Aqueous (total)	P, G	250 mL	HNO_3 to pH < 2	6 months
Aqueous (dissolved)	P, G	250 mL	Filter, HNO_3 to pH < 2	6 months
Solid	P, G	10 g	Cool, 4°C	6 months

[1] P = polyethylene or , G = glass

7.0 PROCEDURES

7.1 Begin by following the startup and calibration instructions provided in the current revision of Katahdin SOP CA-632, "Operation and Maintenance of the Thermo ICAP 6500 ICP Spectrophotometer"

7.2 Analysis must proceed in the sequence described in Table 8 to ensure that all necessary quality control samples are analyzed at the appropriate frequencies. A minimum of two replicate integrations is required for all standards and samples. Analysis always begins with the analysis of a calibration blank solution (S0) followed by analysis of a multielement calibration standard (S1 in Table 3) to calibrate the instrument. The system is flushed with calibration blank for two minutes between each sample and standard, and each sample and standard is aspirated for one minute prior to the beginning of emission measurements.

7.3 Analysis continues with analysis of the initial calibration verification standard (ICV) and the initial calibration blank (ICB) to verify the accuracy of the calibration. Refer to Section 8 and Table 1 for additional information.

7.4 A continuing calibration verification standard (CCV) and a continuing calibration blank (CCB) must be analyzed at the beginning of the run, after every ten samples, and at the end of the run to verify the continued accuracy of the calibration. Refer to Section 8 and Table 1 for additional information.

7.5 Interference check standard solutions (ICSA and ICSAB) must be analyzed at the beginning, end, and at periodic intervals (4-6 hours, 30-40 analytical samples) throughout the sample run to verify the accuracy of the IEC factors. Refer to Section 8 and Table 1 for additional information.

7.6 A practical quantitation limit standard (PQL) must be analyzed at the beginning of each run to determine the accuracy of the calibration at the reporting limit. Refer to Section 8 and Table 1 for additional information.

7.7 All sample analytical results for a particular element that are bracketed (preceded or followed) by failing results in a QC sample (ICV, ICB, CCV, CCB, ICSA, or ICSAB) for that element must not be reported. The sample must be reanalyzed for the element in question.

7.8 All samples that exceed the linear dynamic range must be diluted and reanalyzed. This includes samples with interfering elements that exceed the calibration ranges, because accurate quantitation of interfering elements is necessary for reliable interelement correction. For example, if a sample has been submitted to the

laboratory for lead analysis, and the measured aluminum concentration of that sample exceeds the calibration range for aluminum, it must be diluted sufficiently to bring aluminum within the linear dynamic range and the lead result must be reported from that dilution analysis.

7.9 If dilutions of digested samples are performed, the measured element concentrations must be multiplied by the dilution factor prior to reporting. This is accomplished automatically by entering the dilution factor in the autosampler table prior to initiation of analysis.

7.10 All analyses are performed using yttrium as an internal standard to compensate for enhancement or depression of the analytical signal due to matrix effects. Yttrium solution is pumped at a constant rate through one channel of the peristaltic pump. Samples and standards are pumped through a second channel of the pump. The tubing carrying the internal standard is connected to the tubing carrying samples and standards downstream from the pump, and mixing of the two streams is accomplished in a mixing coil downstream from the connection, prior to nebulization. For each sample or standard, the computer that controls the spectrometer divides the detected emission signal for each element by the detected yttrium emission signal prior to quantitation, thus normalizing all emission signals to that of yttrium.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 6010 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information

will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

INITIAL DEMONSTRATION OF PERFORMANCE

8.1 Instrument detection limits (IDL) are determined quarterly for each analyte analyzed on each instrument. This determination requires seven replicate analyses of a reagent water spiked at 3-5 times the anticipated detection limit for each analyte, performed on three non-consecutive days. The standard deviation of the 21 analyses is multiplied by three to obtain the IDL. For more information on performing IDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.2 Method detection limits (MDL) are determined annually for each analyte analyzed on each instrument. This determination requires at least seven replicate digestions and analyses of a reagent water spiked at 3-5 times the anticipated MDL for each analyte. MDLs differ from IDLs in that the seven replicates are digested prior to analysis, and they may be analyzed on a single day. The standard deviation of the 7 (or more) replicate analyses is multiplied by the Student's t-value to obtain the MDL. For more information on performing MDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.3 A Lower Limit of Quantitation Check (LLQC) sample must be prepared and analyzed annually or on an as-needed basis to confirm the laboratory's Practical Quantitation Limits (PQLs). The LLQC sample is equivalent to the PQL standard (Section 8.10) but is carried through the entire sample preparation and analysis process. Element recoveries for the LLQC sample must fall within 70% to 130% of the expected concentrations to confirm the previously established PQLs.

8.4 The upper limit of the linear dynamic range (LDR) must be established for each wavelength utilized. It must be determined from a linear calibration prepared in the normal manner using the established analytical operating procedure for the instrument. The LDR should be determined by analyzing succeedingly higher standard concentrations of the analyte until the observed analyte concentration differs by no more than 10% from the stated concentration of the standard. Determined LDRs must be documented and kept on file. The LDR which may be used for the analyses of samples should be judged by the analyst from the resulting data. Determined sample analyte concentrations that are greater than the determined upper LDR limit must be diluted and reanalyzed. The LDRs should be verified **every six months** or whenever, in the judgment of the analyst, a change in analytical performance caused by either a change in instrument hardware or operating conditions would dictate they be redetermined.

8.5 The alkali and alkaline earth metals may have non-linear response curves due to ionization and self-absorption effects. These curves may be used for quantitation of samples if the effective range is checked and if the second order curve fit has a correlation coefficient of 0.998 or better. Third order fits are not acceptable. Non-linear response curves must be revalidated and recalculated every six months.

ANALYTICAL RUN QC SAMPLES

8.6 An Initial Calibration Verification (ICV) solution is analyzed after the initial calibration to check calibration accuracy. The ICV solution is prepared by combining compatible elements from a standard source different than that of the calibration standard and at concentrations within the linear working range of the instrument. The results of the ICV must fall within 90% to 110% of the expected values. If the ICV fails, result for the failing elements may not be reported from the run unless the ICV recovery is greater than 110%and the sample result is less than the PQL.

8.7 Continuing Calibration Verification (CCV) solutions are analyzed after the initial calibration, after every ten samples, and at the end of the analytical run. The CCV solution is prepared using the same standards used for calibration at concentrations near the mid-point of the calibration curve. Results of the CCVs must fall within 90% to 110% of the expected values. If a CCV fails, results for the failing elements may not be reported from the run unless the CCV recovery is greater than 110% and the sample result is less than the PQL. Also, for failing elements, all samples analyzed after the last passing CCV must be reanalyzed.

8.8 Calibration blank solution is analyzed after each ICV and CCV. A calibration blank that is analyzed after the ICV is called an Initial Calibration Blank (ICB). A calibration blank that is analyzed after a CCV is called a Continuing Calibration Blank (CCB). The absolute values of results of ICBs and CCBs must be less than the Practical Quantitation Level (PQL) for each element. If an ICB or a CCB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has been analyzed, with the following exception. If the result for a CCB a ICB is greater than the PQL, sample results that are less than the PQL or greater than or equal to ten times the measured CCB concentration may be reported. Also, for failing elements, all samples analyzed after the last passing CCB must be reanalyzed, with the exception noted above.

8.9 Interference check solutions ICSA and ICSAB (refer to Section 1.1) are analyzed at the beginning of each run to verify interelement correction factors and background correction. ICSA contains interferent elements (Al, Ca, Fe, and Mg) only, at concentrations of 200 mg/L to 500 mg/L. Results for interfering elements in the ICSA must fall within 80% to 120% of the expected values. Results for unspiked elements in ICSA must fall within ± PQL if the PQL is greater than 0.01 mg/L, within ± 2xPQL if the PQL is less than or equal to 0.01 mg/L. ICSAB contains interferent elements at concentrations of 200 mg/L to 500 mg/L, and analytes at

concentrations of 20 mg/L or less. Results for all elements (interferents and analytes) in ICSAB must fall within 80% to 120% of the expected values. If the ICSA or ICSAB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICSA or ICSAB has been analyzed.

8.10 A Practical Quantitation Limit (PQL) Check Standard or low level continuing calibration verification (LLCCV) is analyzed at the beginning (after the ICV and ICB samples) and at the end of each run. Element concentrations in this solution are at the laboratories practical quantitation limit. Element recoveries for the PQL check Standard must fall between 70-130% of the expected values. If the PQL Check Standard fails, the results for the failing elements may not be reported from the run, unless the PQL Check Standard recovery is greater than 130% and the samples results are less than the PQL.

PREPARATION BATCH QC SAMPLES

8.11 Each digestion batch of twenty or fewer samples will contain a preparation blank and a laboratory control sample. Each batch will also contain one or more of the following QC samples: laboratory control sample duplicate, sample duplicate, matrix spike sample or matrix spike sample duplicate.

8.12 A preparation blank (PBW or PBS), consisting of reagent water carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. The results of preparation blanks must be less than the Practical Quantitation Level (PQL) for each element. If a preparation blank fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested, with the following exception. If the result for a preparation blank is greater than the PQL, associated sample results that are less than the PQL or greater than or equal to ten times the measured preparation blank concentration may be reported.

8.13 A laboratory control sample (LCS), consisting of spiked reagent water or a solid reference material carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. Results for laboratory control samples must fall within 80% to 120% of the expected value, unless vendor-supplied limits (for solid reference materials) or laboratory-generated statistical limits are available. If a laboratory control sample fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested with the following exception. If the LCS fails high, sample results less than the PQL may be reported.

SAMPLE MATRIX QC SAMPLES

8.14 Matrix spiked duplicate samples are prepared at a minimum frequency of one per digestion batch. The recovery for each element in a spiked sample or spiked

duplicate sample must fall within 75% to 125% of the actual value if the result for the unspiked sample is less than four times the amount of spike added. If one or both spike recoveries fail, the associated sample result must be flagged on the report of analysis.

The relative percent difference between sample duplicate, matrix spiked duplicate or LCS duplicate, is calculated as follows:

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

Where: D_1 = sample result
D_2= duplicate sample result

A control limit of 20% RPD is applied to duplicate analysis if the original sample result is greater than 50X the IDL. If the matrix spike duplicate analysis fails, the associated sample result must be flagged on the report of analysis.

8.15 A serial dilution is analyzed to check for chemical or physical interferences. If the analyte concentration of a sample is sufficiently high (minimally, 50 x IDL), the measured concentration of a serial dilution (1:5 dilution) of the sample should agree within 90% to 110% of the original determination. The percent difference between the original sample and the serial dilution should be calculated as follows:

$$\text{Difference (\%)} = \frac{|L-S|}{S} * 100\%$$

where:
L = Serial dilution result (corrected for dilution)
S = Original sample result

If the serial dilution analysis fails, a matrix interference should be suspected. The associated sample result should be flagged on the report of analysis or the sample should be reanalyzed at dilution to eliminate the interference.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Metals Supervisor and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit and Instrument Detection Limit Studies, for procedures on determining the MDL.

Refer to the current revision of Method 6010 for other method performance parameters and requirements.

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Method 6010C.

List of Tables & Figures

Table 1 QC Requirements
Table 2 Summary of Method Modifications
Table 3 Preparation of Calibration and Quality Control Standards
Table 4 Preparation of Intermediate Standards
Table 5 Element Concentrations in Working Standards
Table 6 Element Concentrations in Intermediate Standards
Table 7 Element Concentrations in Stock Standards
Table 8 Required Analytical Sequence
Attachment 1 Hardness by Calculation

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 1

QC REQUIREMENTS

Method	QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
USEPA 6010	Initial Calibration, minimum 1 point plus a calibration blank.	Daily prior to sample analysis.	Correlation coefficient (r) ≥ 0.998	Recalibrate
	Initial Calibration Verification (ICV), prepared from a second source.	Before beginning a sample run.	Recovery within ± 10% of true value.	1) Do not use results for failing elements unless the ICV > 110% and the sample < the PQL. 2) Investigate and correct problem.
	Initial Calibration Blank (ICB)	Immediately after the ICV.	Absolute value of ICB < PQL.	1) Do not use results if ≥ PQL and < 10x CCB level. 2) Investigate and correct problem.
	Continuing Calibration Verification (CCV)	At beginning of run, after every 10 samples, and at end of run.	Recovery within ± 10% of true value.	1) Do not use results for failing elements unless the ICV > 110% and the sample < the PQL. 2) Investigate and correct problem.
	Continuing Calibration Blank (CCB)	After every 10 samples and at end of the run.	Absolute value of CCB < PQL.	1) Do not use results if ≥ PQL and < 10x CCB level. 2) Investigate and correct problem.
	Practical Quantitation Level Check Standard (PQL) (LLCCV)	At beginning and end of run.	Recovery within ± 30% of true value.	1) Do not use results for failing elements unless the ICV > 110% and the sample < the PQL. 2) Investigate and correct problem.
	Interference Check Solution A (ICSA)	At beginning and end of run.	For Al, Ca, Fe, and Mg, recovery within ± 20% of true value. For analytes not spiked, ± PQL, or, if PQL ≤ 0.01 mg/L, ± 2x PQL.	1) Do not use results for failing elements. 2) Investigate and correct problem.
	Interference Check Solution AB (ICSAB)	At beginning and end of run.	Recovery of each analyte within ± 20% of true value.	1) Do not use results for failing elements. 2) Investigate and correct problem.

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 1, CONTINUED

QC REQUIREMENTS

Method	QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
USEPA 6010, continued	Preparation Blank (PBW/PBS)	One per digestion batch of 20 or fewer samples.	Less than PQL.	1) Investigate source of contamination. 2) Redigest and reanalyze all associated samples if sample concentration ≥ PQL and <10x the blank concentration.
	Laboratory Control Sample (LCSW/LCSS)	One per digestion batch of 20 or fewer samples.	Recovery within ± 20% of true value, unless vendor-supplied or statistical limits have been established.	1) Investigate source of problem. 2) Redigest and reanalyze all associated samples.
	Matrix Spike Sample (S)	One per digestion batch of 20 or fewer samples.	Recovery ± 25% of true value, if sample < 4x spike added.	Flag results.
	Matrix Spike Duplicate Sample (P)	One per digestion batch of 20 or fewer samples.	1) Recovery ± 25% of true value, if sample < 4x spike added. 2) RPD ≤20% for duplicate spikes.	Flag results
	Serial Dilution (L)	One per digestion batch.	If original sample result is at least 50x IDL, 5-fold dilution must agree within ± 10% of the original result.	Flag result or dilute and reanalyzed sample to eliminate interference.
	Instrument Detection Limit (IDL) Study	Quarterly.	IDL < MDL PQL > 2-3 * the IDL	1) Repeat IDL study. 2) Raise PQL.
	Method Detection Limit (MDL) Study	Annually.	M PQL > 2-3 * the MDL	1) Repeat MDL study. 2) Raise PQL.
	Lower Limit of Quantitation Check (LLQC) Sample	Digest and analyze annually or as needed to confirm PQLs	70% - 130% of true value	Reevaluate PQLs
	Linear Range Study	Every six months	Run succeedingly higher stds until recovery not within ± 10%. Use highest passing concentration as upper limit of linear range.	Only accept data to highest passing concentration until next linear range study.

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-608-08	METHOD 6010, current revision
Apparatus/Materials		
Reagents		
Sample preservation/ handling		
Procedures		
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		
QC - Calibration Blanks	Acceptance criteria employed for 6010: ± PQL	Acceptance criteria stated in 6010: less than 10% of PQL

TABLE 3

PREPARATION OF CALIBRATION AND QUALITY CONTROL STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
Calibration Standard (STD1 or S1)	ICP- intermediate Standard	Lab Prepared (see Table 4)	10.0
	QCS 26	High Purity Standards	1.0
Initial Calibration Verification (ICV)	QCP-CICV-3	Inorganic Ventures	0.96
	1000 mg/L Si	Inorganic Ventures	0.98
	1000 mg/L Al	High Purity Standards	0.96
	IV-28	Inorganic Ventures	0.4
	1000 mg/L Sn	Inorganic Ventures	0.04
Interference Check Sample A (ICSA)	CLPP-ICS-A	Inorganic Ventures	10.0
Interference Check Sample AB (ICSAB)	CLPP-ICS-A	Inorganic Ventures	10.0
	CLPP-ICS-B4	Inorganic Ventures	1.0
	ICSAB-INT	Lab Prepared (see Table 4)	5.0
Continuing Calibration Verification (CCV)	ICP intermediate standard	Lab Prepared (see Table 4)	5.0
	QCS 26	High Purity Standards	0.5
Practical Quantitation Limit Sample (PQL)	PQL-INT	Lab Prepared (see Table 4)	1.0

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 4

PREPARATION OF INTERMEDIATE STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
PQL-INT	1000 mg/L B,Li,Sn,Sr, W, U	H-P or IV	1.0 each
	10000 mg/L K, Na	H-P or IV	1.0 each
	1000 mg/L Ni	High Purity Standards	0.4
	1000 mg/L Co	High Purity Standards	0.3
	1000 mg/L Cu,V,Zn	High Purity Standards	0.25 each
	1000 mg/L Si	High Purity Standards	2.0
	1000 mg/L Cr,Ti,Tl,Ag	High Purity Standards	0.15 each
	1000 mg/L Cd,Se, Mo	High Purity Standards	0.1 each
	10000 mg/L Al	High Purity Standards	0.3
	1000 mg/L As,Sb	High Purity Standards	0.08 each
	1000 mg/L Ba,Be,Mn,Pb	High Purity Standards	0.05 each
	10000 mg/L Ca,Mg	High Purity Standards	0.05 each
	10000 mg/L Fe	High Purity Standards	0.1
ICSAB-INT	10000 mg/L K,Na	H-P or IV	4.0 each
	10000 mg/L B, Li, Mo,Sr,Sn,Ti, W, U	High Purity Standards	1.0 each
	1000 mg/L Si	High Purity Standards	4.0

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 5

ELEMENT CONCENTRATIONS IN WORKING STANDARDS

	CONCENTRATION IN SOLUTION, mg/L								
Element	**STD1**	**ICV**	**PQL**	**ICSA**	**ICSAB**	**CCV**	**AL_IEC**	**FE_IEC**	**MN_IEC**
Aluminum	25	10	0.3	500	500	12.5	500		
Antimony	1	0.4	0.008		0.6	0.5			
Arsenic	1	0.4	0.008		0.1	0.5			
Barium	1	0.4	0.005		0.5	0.5			
Beryllium	1	0.4	0.005		0.5	0.5			
Boron	1	0.4	0.1		0.5	0.5			
Cadmium	1	0.4	0.01		1.0	0.5			
Calcium	25	10	0.05	500	500	12.5			
Chromium	1	0.4	0.015		0.5	0.5			
Cobalt	1	0.4	0.03		0.5	0.5			
Copper	1	0.4	0.025		0.5	0.5			
Iron	25	10	0.1	200	200	12.5		200	
Lead	1	0.4	0.005		0.05	0.5			
Lithium	1	0.4	0.1		0.5	0.5			
Magnesium	25	10	0.05	500	500	12.5			
Manganese	1	0.4	0.005		0.5	0.5			10
Molybdenum	1	0.4	0.01		0.5	0.5			
Nickel	1	0.4	0.04		0.5	0.5			
Potassium	25	13.6	1		20	12.5			
Selenium	1	0.4	0.01		0.05	0.5			
Silicon	1	0.4	0.2		2	0.5			
Silver	1	0.4	0.015		0.2	0.5			
Sodium	25	10	1		20	12.5			
Strontium	1	0.4	0.1		0.5	0.5			
Thallium	1	0.4	0.015		0.1	0.5			
Tin	1	0.4	0.1		0.5	0.5			
Titanium	1	0.4	0.015		0.5	0.5			
Tungsten	1	0.4	0.1		0.5	0.5			
Uranium	1	0.4	0.1		0.5	0.5			
Vanadium	1	0.4	0.025		0.5	0.5			
Zinc	1	0.4	0.025		1.0	0.5			

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 6

ELEMENT CONCENTRATIONS IN INTERMEDIATE STANDARDS

	CONCENTRATION IN SOLUTION, mg/L			
Element	**ICP Intermed STD**		**PQL-INT**	**ICSAB-INT**
Aluminum	240		30	
Antimony			0.8	
Arsenic			0.8	
Barium			0.5	
Beryllium			0.5	
Boron			10	10
Cadmium			1.0	
Calcium	240		5.0	
Chromium			1.5	
Cobalt			3.0	
Copper			2.5	
Iron	240		10	
Lead			0.5	
Lithium	10		10	10
Magnesium	240		5.0	
Manganese			0.5	
Molybdenum			1.0	10
Nickel			4.0	
Potassium	150		100	400
Selenium			1.0	
Silicon	250		20	40
Silver			1.5	
Sodium	240		100	400
Strontium	10		10	10
Thallium			1.5	
Tin	10		10	10
Titanium			1.5	10
Tungsten	10		10	10
Uranium	10		10	10
Vanadium			2.5	
Zinc			2.5	

TITLE: **TRACE METALS ANALYSIS BY ICP-AES USING USEPA METHOD 6010**

TABLE 7

ELEMENT CONCENTRATIONS IN STOCK STANDARDS

Element	CONCENTRATION IN SOLUTION, mg/L					
	IV-28	QCS-26	2007 ICS-1	CLPP-ICS-A	CLPP-ICS-B4	QCP-CICV-3
Aluminum	100	100		5000		
Antimony	100	100			60	
Arsenic	100	100			10	500
Barium	100	100			50	
Beryllium	100	100			50	
Boron	100	100	500			
Cadmium	100	100			100	250
Calcium	100	100		5000		
Chromium	100	100			50	
Cobalt	100	100			50	
Copper	100	100			50	
Iron	100	100		2000		
Lead	100	100			5	500
Lithium	100					
Magnesium	100	100		5000		
Manganese	100	100			50	
Molybdenum	100	100	300			
Nickel	100	100			100	
Potassium	1000	1000				
Selenium	100	100			5	500
Silicon	50	50	230			
Silver	100	100			20	
Sodium	100	100				
Strontium	100					
Thallium	100	100			10	500
Tin						
Titanium	100	100	1000			
Vanadium	100	100			50	
Zinc	100	100			100	

TABLE 8

REQUIRED ANALYTICAL SEQUENCE

Sequence Number	Standard/Sample	Purpose
1	Blank (Calibration Blank)	Initial calibration
2	S1 (Calibration Standard)	Initial calibration
3	ICV (Initial Calibration Verification)	Check calibration accuracy
4	ICB (Initial Calibration Blank)	Check calibration accuracy
5	PQL (Practical Quantitation Level Sample)	Check calibration accuracy near PQL, repeat before final CCV, CCB
6	ICSA (Interference Check Solution A)	Verify accuracy of IEC factors, repeat before final CCV, CCB
7	ICSAB (Interference Check Solution AB)	Verify accuracy of IEC factors, repeat before final CCV, CCB
8	CCV (Continuing Calibration Verification)	Check calibration stability
9	CCB (Continuing Calibration Blank)	Check calibration stability
10-19	Analyze up to 10 samples	
20	CCV (Continuing Calibration Verification)	Check calibration stability
25	CCB (Continuing Calibration Blank)	Check calibration stability
...	Continue analyzing sequences of up to 10 samples, followed by a CCV and a CCB	

ATTACHMENT 1

HARDNESS BY CALCULATION

As referenced in "Standard Methods for the Examination if Water and Wastewater," Methods 2340 A & B, Hardness Introduction and Hardness by Calculation, American Public Health Association, 18th Edition, Revised 1992, total hardness is the sum of the calcium and magnesium concentrations, both expressed as calcium carbonate, in milligrams per liter.

Once the calcium and magnesium concentrations have been determined by EPA methods 6010, 6020, 200.7 or 200.8, the total hardness of an aqueous sample may be calculated as follows:

$$\text{Total Hardness, mg equivalent } CaCO_3/L = 2.497\ (Ca, mg/L) + 4.118\ (Mg, mg/L)$$

The calcium hardness of an aqueous sample may also be calculated as follows:

$$\text{Calcium Hardness, mg equivalent } CaCO_3/L = 2.497\ (Ca, mg/L)$$

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-615
Revision History
Cover Page
Page 1

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

Prepared By: George Brewer Date: 01/01

Approved By:

Group Supervisor: George Brewer Date: 01/29/01

Operations Manager: Joel C. Benton Date: 1/29/01

QA Officer: Deborah J. Nadeau Date: 1·29·01

General Manager: Dennis F. Hufham Date: 1/29/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
00 7470A	NA	DH	1·29·01	1/29/01
01	Revised Sect. 4, 5 and 7 to reflect current practice. Revised Sect. 8 to reflect current QC Limits. Revised sect. 10 to reflect current Applicable Documents and references. Removed figure 2. Update table 1 to reflect current QC Limits. Minor changes throughout	LAD	02-16-05	02-16-05
02	Updated Fig. 1 - new prep Logbook page	LAD	04/08	04/08
03	Updated Figure 1 - Example of a mercury Preparation Logbook page.	LAD	03/09	03/09

TITLE: **DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-615-03,** titled **DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-615-03,** titled **DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470.**

Recipient: _____ Date: _____

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedure used by Katahdin Analytical Services, Inc. personnel for the digestion and analysis aqueous samples for mercury using cold vapor atomic absorption spectrophotometry.

This method is applicable to the determination of mercury in groundwaters, aqueous wastes, and mobility-procedure extracts under USEPA Method 7470 (Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods, SW-846, 2nd edition, 1982 (revised 1984), 3rd edition, 1986, and Updates I, II, IIA, and III 1996, Office of Solid Waste and Emergency Response, U.S. EPA.

1.1 Definitions

CCB - Continuing Calibration Blank - An analyte-free solution consisting of acidified laboratory grade reagent water used to verify calibration accuracy periodically during analysis.

CCV - Continuing Calibration Verification - A midrange standard used to verify calibration accuracy periodically during analysis.

Duplicate - A second aliquot of a sample that is prepared and analyzed in the same way as the original sample in order to determine the precision of the method.

ICB - Initial Calibration Blank - An analyte-free solution consisting of acidified laboratory grade reagent water used to verify calibration accuracy.

ICV - Initial Calibration Verification - A standard made from a source independent from the calibration standards and with analyte concentrations different from those in the CCV; used to verify the accuracy of the instrument calibration.

IDL - Instrument Detection Limit - The lowest concentration of an analyte that can be determined with 95% confidence by the instrument.

LCS - Laboratory Control Sample - A standard or solid reference material that has been brought through the sample preparation process.

PB - Preparation Blank - Laboratory grade reagent water that has been brought through the sample preparation process.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the IDL.

Matrix Spike - An aliquot of a sample to which a known amount of analyte has been added before digestion.

MDL - Method Detection Limit - The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of mercury by USEPA Method 7470. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in analysis of mercury by USEPA Method 7470 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to ensure that members of their group follow this SOP, that their work is properly documented, and to indicate periodic review of the associated logbooks.

1.3 Safety

Many of the samples and reagents used in cold vapor atomic absorption are toxic or corrosive. Rubber gloves, safety glasses, lab coats, and other protective clothing should be worn whenever these materials are handled. Because of the toxic nature of mercury vapor, care must be taken to avoid its inhalation. The instrument exhaust fan must be in operation whenever the mercury analyzer is in use (the fan should never be shut off).

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with the Katahdin Analytical Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and follow appropriate procedures such as wearing safety glasses and gloves when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location and use of all safety equipment.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

Samples, sample digestates, standards, and other reagents used in cold vapor atomic absorption may contain high concentrations of acids, mercury, and other toxic metals. They should be disposed of in a manner appropriate to the types of hazards they present. All digested mercury samples and standards and excess reagents and standards should be disposed of in the satellite waste container for corrosive wastes (labeled "Waste Stream A") that is located in the Metals Prep lab. Further information regarding waste classification and disposal may be obtained by consulting the laboratory's Hazardous Waste Management Plan and Safety Manual and the Department Manager.

2.0 SUMMARY OF METHOD

The cold vapor atomic absorption technique is based on the absorption of radiation at 253.7 nm by mercury vapor. It relies on the volatility of elemental mercury at room temperature. During preparation, organic mercurials are oxidized and elemental mercury is ionized to Hg^{3+}. During instrumental analysis, mercuric ions are reduced to elemental mercury by the addition of stannous chloride. Elemental mercury is then aerated from solution and passes through a cell positioned in the path of a mercury spectrophotometer, where absorbance (peak height) is measured as a function of mercury concentration and recorded by the associated computer. The mercury vapor is then swept out of the instrument into an exhaust hood, where it is evacuated from the laboratory.

3.0 INTERFERENCES

In addition to inorganic forms of mercury, organic mercurials may be present in environmental samples. These organo-mercury compounds will not respond to the cold vapor atomic absorption technique unless they are first broken down and converted to mercuric ions. The presence of undigested organo-mercurials in samples will result in a low

bias for analytical results. Certain volatile organic materials will also non-specifically absorb radiation at the 253.7 nm analytical wavelength. The presence of such compounds may result in a high bias for analytical results. For these reasons, complete digestion using potassium permanganate and potassium persulfate is required for all environmental samples. Complete digestion is indicated by the persistence of the purple permanganate color (indicating the presence of excess permanganate) following digestion.

Sea waters, brines, and industrial effluents high in chlorides may require additional permanganate to maintain a persistent purple color following digestion. During the oxidation step, chlorides are converted to free chlorine which will absorb radiation at the 253.7 nm analytical wavelength. Any free chlorine thus generated will be present in the headspace of the digestion vessel following digestion. Because samples are poured into autosampler tubes prior to analysis by the mercury analyzer, any free chlorine present in the headspace of the digestion vessels is not sampled by the instrument and the analysis is free of chlorine interference.

4.0 APPARATUS AND MATERIALS

4.1 40 mL VOA vials, for use as digestion vessels.

4.2 250 mL Pyrex media bottles with plastic screw caps, for use in digesting calibration standards.

4.3 Water bath capable of maintaining a constant temperature of 95° C.

4.4 Adjustable volume automatic pipettes - 2 to 20 uL, 10 to 100 uL, 100 to 1000 uL. Calibrated Eppendorf Reference pipets and Finn digital pipets are appropriate.

4.5 Repipetters (adjustable repeating pipetters with reservoirs) for dispensing concentrated nitric acid, concentrated sulfuric acid, and other reagents

4.6 Spirit-filled thermometer, NIST-traceable, covering the range from 20° to 110° C, for monitoring the temperature of the water bath. Mercury-filled thermometers are not acceptable for use in the metals laboratory, due to the possibility of breakage and consequent contamination.

4.7 Disposable graduated polystyrene sample cups, 200 mL capacity

4.8 CETAC M-6100 automated mercury analyzer and associated peripherals and parts

4.9 Disposable graduated dose cups, 30 mL capacity

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

Refer to Katahdin SOP CA-629, current revision, "Operation and Maintenance of the CETAC M-6100 Automated Mercury Analyzer" for additional required materials.

5.0 REAGENTS

5.1 Laboratory grade reagent water – mercury-free water meeting the specifications of ASTM Type II water

5.2 Concentrated sulfuric acid, trace metals grade

5.3 Concentrated nitric acid, trace metals grade

5.4 Concentrated hydrochloric acid, trace metal grade

5.5 Potassium permanganate solution, 5% w/v: Dissolve 50 g of potassium permanganate in 1 L laboratory grade reagent water. The source reagent should be labeled as suitable for use in mercury determination.

5.6 Potassium persulfate solution, 5% w/v: Dissolve 50g of potassium permanganate in 1L laboratory grade reagent water. The source reagent should be labeled as suitable for use in mercury determination.

5.7 Sodium chloride – hydroxylamine hydrochloride solution: Dissolve 120 g sodium chloride and 120 g hydroxylamine hydrochloride in laboratory grade reagent water and dilute to a final volume of 1 L.

5.8 Stannous chloride solution: Add 70 mL concentrated hydrochloric acid to 500 mL of laboratory grade reagent water. Add 100 g stannous chloride and bring to a final volume of 1 L. Mix to dissolve. Reagent should be labeled as suitable for use in mercury determination.

5.9 Intermediate Mercury Standard A: Appropriately dilute a mercury stock standard to obtain a solution containing 1000 ug of mercury per liter in 2% nitric acid. This intermediate standard is used to prepare calibration standards, matrix spikes, CCVs, and laboratory control samples (refer to Section 8). The identity of the stock standard currently used to prepare this intermediate may be obtained by consulting the Standards Preparation Logbook maintained in the Section. Intermediate Mercury Standard A must be prepared fresh monthly ,and disposed of appropriately after use. (Note: the concentrations of all stock standards must be certified by the vendors as traceable to NIST reference materials).

5.10 Intermediate Mercury Standard B: Appropriately dilute a mercury stock standard to obtain a solution containing 1000 ug of mercury per liter in 2% nitric acid. The

source of the stock standard used to prepare Intermediate Mercury Standard B must be distinct from that used to prepare Intermediate Mercury Standard A (i.e. obtained from a separate vendor). Intermediate Mercury Standard B is used to prepare the ICV (refer to Section 8). The identity of the stock standard currently used to prepare this intermediate standard may be obtained by consulting the Standards Preparation Logbook maintained in the Section. Intermediate Mercury Standard B must be prepared fresh monthly, and disposed of appropriately after use.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Aqueous samples to be analyzed for mercury should be collected and preserved as described in the following table.

Matrix	Container[1]	Collection Volume/ Weight	Preservation/ Treatment	Holding Time
Aqueous (total)	P, G	250 mL	HNO_3 to pH < 2	28 days
Aqueous (dissolved)	P, G	250 mL	HNO_3 to pH < 2	28 days

[1] P = polyethylene or G = glass

7.0 PROCEDURES

BOTTLE PREPARATION

7.1 Mercury digestions are performed in two different types of vessels. Calibration standards, the Initial Calibration Verification (ICV) standard, and the Initial/Continuing Calibration Blank (ICB/CCB) are prepared in 250 mL Pyrex media bottles. Large bottles are used to provide sufficient volumes of these standards to allow for multiple reanalyses when required. Field samples, Method Blanks, and Laboratory Control Samples are digested in 40 mL VOA vials. These smaller vials provide enough digestate to allow one or two reanalyses when required, but reduce the amounts of samples consumed and waste generated.

VOA vials are reused if the samples they have contained have no measurable mercury above the PQL. After the previous contents of the vials have been discarded, these vials are segregated according to whether the measured mercury concentrations of the previous contents were above the PQL (contaminated vials) or below the PQL (uncontaminated vials). Labels are removed from the vials by

wiping with a paper towel saturated with toluene. Uncontaminated vials are rinsed with laboratory grade reagent water. Contaminated vials are discarded.

The Pyrex media bottles in which standards are prepared are emptied, rinsed, and reused. Each of these bottles is permanently marked with the concentration of the standard it contains.

PREPARATION OF STANDARDS, QC SAMPLES, AND BLANKS

7.2 Prior to performing the digestion, make a list of the samples that are to be digested. Enter digestion information (Katahdin Sample Numbers, QC Batch ID, preparation date, analyst initials, etc.) into the ACCESS Metals database and print out a copy of the sample prep bench sheet. All necessary details of sample preparation (standards preparation information, digestion times, initial and final volumes, pertinent observations, etc.) must be recorded on this spreadsheet, which will be bound in the Mercury Preparation Logbook. Refer to Figure 1 for an example page from the Mercury Preparation Logbook.

7.3 Using a silver paint marker, label clean VOA vials with the appropriate sample numbers and standard identifications for each sample and standard to be digested.

7.4 Use a bottle-top dispenser to add 100 mL of laboratory grade reagent water to 6 standards digestion bottles (250 mL media bottles). Using calibrated adjustable pipettes, prepare calibration standards by adding 0 uL, 20 uL, 50 uL, 100 uL, 500 uL, and 1000 uL of Intermediate Mercury Standard A to separate appropriately-labeled media bottles containing 100 mL of laboratory grade reagent water. The mercury concentrations of these calibration standards are, respectively, 0 ug/L (calibration blank), 0.2 ug/L, 0.5 ug/L, 1.0 ug/L, 5.0 ug/L, and 10.0 ug/L. The 0.2 ug/L and 0.5 ug/L standards are analyzed after calibration as the PQL standard and the CCV (refer to Section 8.0), respectively, as well as being used in the creation of the calibration curve.

7.5 Add 100 mL of laboratory grade reagent water to the media bottle labeled "ICV". Using a calibrated adjustable pipette, prepare the Initial Calibration Verification standard (refer to Section 8) by adding 600 uL of Intermediate Mercury Standard B to the water in this bottle, and record the bottle number in the Mercury Preparation Logbook. The mercury concentration of the ICV is 6.0 ug/L.

7.6 Prepare an appropriate number of preparation blanks (PBW) by adding 25 mL of laboratory grade reagent water to labeled vials.

7.7 Prepare an appropriate number of laboratory control samples (LCSW) by adding 125 uL of Intermediate Mercury Standard A to labeled digestion vials containing 25

mL of laboratory grade reagent water. The mercury concentration of each LCSW is 5.0 ug/L.

7.8 Matrix spikes are prepared by adding 25 uL of Intermediate Mercury Std A to 25 mL aliquots of samples. The concentration of mercury added to each matrix spike is 1.0 ug/L.

7.9 All QC samples and blanks are digested in the same manner as client samples. Refer to Sample Preparation and Digestion, sections 7.10 through 7.13 of this SOP. The volumes of reagents added to the standards prepared in the media bottles are four times those listed in sections 7.10 through 7.13.

SAMPLE PREPARATION AND DIGESTION

7.10 Using a graduated disposable dosecup, transfer 25 mL of sample, or an aliquot diluted to 25 mL, to a digestion vial. Add 1.25 mL of concentrated sulfuric acid and 0.625 mL of concentrated nitric acid, swirling to mix after each addition. Add 3.75 mL of potassium permanganate solution, swirl to mix, and allow to stand for at least 15 minutes. Samples that contain large amounts of organic substances may require additional 3.75 mL aliquots of potassium permanganate solution. This is indicated by the failure of the purple permanganate color to persist for the entire 15 minute waiting period. Add additional 3.75 mL aliquots to samples as necessary until the purple color persists for 15 minutes. If any of the samples require these additional aliquots of potassium permanganate solution, record the additional volume used for each sample on the mercury preparation benchsheet.

7.11 Add 2 mL of potassium persulfate solution to each sample. Cap the vials and place them in a preheated water bath. Monitor the temperature of the bath with a spirit thermometer throughout the digestion. The temperature of the water bath will fall below 95° C upon addition of the digestion vials. After the temperature of the bath has risen back to 95° C, continue heating the samples at 95° C for two hours.

7.12 Remove bottles from the water bath and allow to cool to room temperature. If the purple permanganate color has failed to persist after digestion in any of the samples, add additional 3.75 mL aliquots of potassium permanganate solution as required to the samples, and record these additions in the mercury preparation benchsheet. Heat the samples that required additional permanganate in the water bath at 95° C for an additional two hours. Remove the bottles from the water bath and allow to cool to room temperature. If the purple color fails to persist after the second heating step, consult the Department Manager for advice on how to proceed.

7.13 Add 1.5 mL of sodium chloride – hydroxylamine hydrochloride solution to each digestion vial and swirl to mix. This will reduce the excess permanganate, and the

sample will change from purple to colorless. Wait at least 30 seconds before proceeding with analysis.

INSTRUMENTAL ANALYSIS

7.14 Digested mercury samples are analyzed using the CETAC M-6100 Automated Mercury Analyzer. Analysis is automated and is controlled by the QuickTrace Mercury Analyzer software running on a dedicated PC. Detailed instructions for setting up the instrument and analyzing samples are given Katahdin SOP CA-629, "Operation and Maintenance of the CETAC M-6100 Automated Mercury Analyzer".

METHOD OF STANDARD ADDITIONS

7.15 The standard addition technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample constituent that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift. The method of standard additions shall be used for analysis of all EP extracts, on all analyses submitted as part of a delisting petition, and whenever a new sample matrix is being analyzed.

7.15.1 The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample solution, each of volume V_x, are taken. To the first (labeled A) is added a known volume V_S of a standard analyte solution of concentration C_S. To the second aliquot (labeled B) is added the same volume V_S of the solvent. The analytical signals of A and B are measured and corrected for nonanalyte signals. The unknown sample concentration C_x is calculated:

$$C_X = \frac{S_B V_S C_S}{(S_A - S_B) V_X}$$

where S_A and S_B are the analytical signals (corrected for the blank) of solutions A and B, respectively. V_s and C_s should be chosen so that S_A is roughly twice S_B on the average, avoiding excess dilution of the sample. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure.

7.15.2 Improved results can be obtained by employing a series of standard additions. To equal volumes of the sample are added a series of standard solutions containing different known quantities of the analyte, and all solutions are diluted to the same final volume. For example, addition 1 should be prepared so that the resulting concentration is approximately 50 percent of the expected absorbance from the endogenous analyte in the

sample. Additions 2 and 3 should be prepared so that the concentrations are approximately 100 and 150 percent of the expected endogenous sample absorbance. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated to zero absorbance, the point of interception of the abscissa is the endogenous concentration of the analyte in the sample. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate. An example of a plot so obtained is shown in Figure 3. A linear regression program may be used to obtain the intercept concentration.

7.15.3 For the results of this MSA technique to be valid, the following limitations must be taken into consideration:

- The apparent concentrations from the calibration curve must be linear over the concentration range of concern. For the best results, the slope of the MSA plot should be nearly the same as the slope of the standard curve. If the slope is significantly different (greater than 20%), caution should be exercised.

- The effect of the interference should not vary as the ratio of analyte concentration to sample matrix changes, and the standard addition should respond in a similar manner as the analyte.

- The determination must be free of spectral interference and corrected for nonspecific background interference.

DATA REDUCTION AND REPORTING

7.16 Results are obtained in concentration units (ug/L) from the instrument. Electronic instrument data files are imported into the Metals ACCESS database for data reduction. Sample preparation information (initial sample volumes and final digestate volumes) are entered directly into the Metals ACCESS database to allow calculation of final results for reporting. Results are calculated as follows:

$$\text{Mercury concentration (ug/L)} = \frac{MC \times DF \times IV}{FV}$$

Where:
MC = Measured mercury concentration (ug/L)
DF = Dilution factor at instrument
IV = Initial sample volume (mL)
FV = Final digestate volume (mL)

7.17 Results that exceed the calibration range of the instrument may not be reported - the sample must be appropriately diluted and reanalyzed. Results for diluted samples should be multiplied by the dilution factor prior to reporting. If additional aliquots of potassium permanganate were added during digestion, the resulting dilution must be corrected for before reporting.

7.18 Results are reported down to the laboratory's practical quantitation level (PQL), unless otherwise requested. Results below the PQL should be reported to the PQL and flagged with a "U" qualifier.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 7470 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Preparation instructions and the resulting mercury concentrations for calibration standards, QC standards, and matrix spikes are detailed in Sections 7.4 through 7.8 of this SOP. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The Department Manager, Operations Manager, General Manager and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

INITIAL DEMONSTRATION OF PERFORMANCE

8.1 Instrument detection limits (IDL) are determined quarterly for each analyte analyzed on each instrument by each method. This determination requires seven replicate

analyses of a laboratory grade reagent water spiked at 3-5 times the anticipated detection limit for each analyte, performed on three non-consecutive days. The standard deviation of the 21 analyses is multiplied by three to obtain the IDL. For more information on performing IDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.2 Method detection limits (MDL) are determined annually for each analyte analyzed on each instrument. This determination requires at least seven replicate digestions and analyses of laboratory grade reagent water spiked at 3-5 times the anticipated MDL for each analyte. MDLs differ from IDLs in that the replicates are digested prior to analysis, and they may be analyzed on a single day. The standard deviation of the 7 (or more) replicate analyses is multiplied by the Student's t-value to obtain the MDL. For more information on performing MDL determinations, refer to the current revision of Katahdin SOP QA-806.

ANALYTICAL RUN QC

8.3 Instrument calibration - The instrument must be calibrated each time it is set up, and calibration standards must be digested each day that samples are digested. Calibration includes analysis of a calibration blank and five calibration standards with graduated concentrations in the appropriate range. The concentration of one of the calibration standards must be at the Practical Quantitation Level (PQL). The intermediate standards used for preparing the calibration standards are prepared at least once per month in 2% nitric acid. Because mercury may be adsorbed onto the walls of glass and plastic containers, the calibration standards must be prepared fresh daily. The correlation coefficient for the calibration curve must be at least 0.995. If the calibration curve does not pass this test, analysis must be halted, the problem corrected, and the instrument recalibrated.

8.4 An Initial Calibration Verification (ICV) solution is analyzed after the initial calibration to check calibration accuracy. The ICV solution is prepared from a standard source different than that of the calibration standard and at a concentration within the working range of the instrument. The result of the ICV must fall within 90% to 110% of the expected value. If the ICV fails, results may not be reported from the run until the problem is corrected and a passing ICV has been analyzed.

8.5 The Continuing Calibration Verification (CCV) solution is analyzed after the initial calibration, after every ten samples, and at the end of the analytical run. The CCV solution is prepared using the same standard used for calibration at a concentration near the mid-point of the calibration curve. Results of the CCVs must fall within 80% to 120% of the expected value. If a CCV fails, associated sample results may not be reported from the run until the problem is corrected and a passing CCV has been analyzed. Also, all samples analyzed after the last passing CCV must be reanalyzed.

8.6 A calibration blank is analyzed after each ICV and CCV. A calibration blank that is analyzed after the ICV is called an Initial Calibration Blank (ICB). A calibration blank that is analyzed after a CCV is called a Continuing Calibration Blank (CCB). The absolute values of results of ICBs and CCBs must be less than the Practical Quantitation Level (PQL) for each element. If an ICB or a CCB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has been analyzed. Also, all samples analyzed after the last passing CCB must be reanalyzed.

8.7 A standard with a mercury concentration that is at the Practical Quantitation Limit (PQL) is analyzed at the beginning of the run to determine calibration accuracy at the reporting limit. Result of the PQL standard should fall within 50% to 150% of the expected values. No corrective action has been established at this time.

PREPARATION BATCH QC SAMPLES

8.8 A preparation blank (PBW), consisting of laboratory grade reagent water carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. The results of preparation blanks must be less than the Practical Quantitation Level (PQL). If a preparation blank fails, results may not be reported from the digestion batch, and all associated samples must be redigested, with the following exception. Associated sample results that are below the PQL may be reported without notation.

8.9 A laboratory control sample (LCSW), consisting of spiked reagent carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. Results for laboratory control samples must fall within 80% to 120% of the expected value, unless laboratory-generated statistical limits are available. If a laboratory control sample fails, results may not be reported from the digestion batch, and all associated samples must be redigested.

SAMPLE MATRIX QC SAMPLES

8.10 Matrix spiked duplicate samples are prepared at a minimum frequency of one per digestion batch. Matrix spike recoveries for these samples are calculated as follows:

$$\text{Recovery (\%)} = \frac{(P - S)}{A} \times 100\%$$

where:

P = Spiked sample value
S = Original sample value
A = Spike amount

The recovery for each element in a spiked sample or spiked duplicate sample must fall within 75% to 125% of the actual value if the result for the unspiked sample is less than four times the amount of spike added. If one or both spike recoveries fail, a matrix interference should be suspected and the associated sample result should be flagged on the report of analysis.

The relative percent difference between matrix spiked duplicate sample results is calculated as follows:

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

where:

D_1 = Spike sample result
D_2= Spike duplicate sample result

A control limit of 20% RPD is applied to matrix spike duplicate analysis. If the matrix spike duplicate analysis fails, the associated sample result should be flagged on the report of analysis.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit and Instrument Detection Limit Studies, for procedures on determining the MDL.

Refer to the current revision of USEPA Method 245.1 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

(Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods, SW-846, 2nd edition, 1982 (revised 1984), 3rd edition, 1986, and Updates I, II, IIA, and III 1996, Office of Solid Waste and Emergency Response, U.S. EPA, Method 7470

QuickTrace M-6100 Mercury Analyzer – Operator Manual, Version 1.0.1, CETAC Technologies, 2003.

TITLE: **DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470**

QuickTrace Mercury Analyzer – Software Manual, Version 1.0, Cetac Technologies, June 2002

List of Tables and Figures

Table 1 QC Requirements
Table 2 Method Modifications
Figure 1 Example Mercury Preparation Logbook Page
Figure 2 Standard Additions Plot

TITLE: **DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470**

TABLE 1

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Mercury/ USEPA 7470	Initial Calibration, 5 points plus a calibration blank.	Daily prior to sample analysis.	Correlation coefficient ≥ 0.995.	Correct problem and repeat calibration.
	Initial Calibration Verification (ICV), prepared from a second source.	Before beginning a sample run.	Recovery within ± 10% of true value.	Correct problem and repeat calibration.
	Initial Calibration Blank (ICB)	Before beginning a sample run.	Less than PQL.	Correct problem and repeat calibration.
	Practical Quantitation Level Standard (PQL)	Before beginning a sample run.	Recovery within ± 50% of true value.	No corrective action required at this time.
	Continuing Calibration Verification (CCV)	At beginning or run, after every 10 samples, and at end of the run	Recovery within ± 20% of true value	Repeat calibration and reanalyze all samples analyzed since the last successful CCV.
	Continuing Calibration Blank (CCB)	At beginning or run, after every 10 samples, and at end of the run	Less than PQL.	Repeat calibration and reanalyze all samples analyzed since the last successful CCB.
	Preparation Blank (PBW)	One per digestion batch of 20 or fewer samples.	Less than PQL.	1) Investigate source of contamination. 2) Redigest and reanalyze all associated samples if sample concentration ≥ PQL and < 10x the blank concentration.
	Laboratory Control Sample (LCSW)	One per digestion batch of 20 or fewer samples.	Recovery within ± 20% of true value.	Redigest all affected samples.
	Matrix Spike Sample (S)	One per digestion batch of 20 or fewer samples.	Recovery ±25% of true value, if sample > 4x spike value.	Flag results.
	Matrix Spike Duplicate Sample (P)	One per digestion batch of 20 or fewer samples.	1) Recovery ± 25% of true value, if sample < 4x spike added. 2) RPD ≤20% for duplicate spikes.	Flag results
	Instrument Detection Limit (IDL) Study	Quarterly.	IDL < PQL	1) Repeat IDL study. 2) Raise PQL.
	Method Detection Limit (MDL) Study	Annually.	Ideally, PQL = at least 3 X the MDL.	1) Repeat MDL study. 2) Raise PQL.

TITLE: **DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470**

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-615-02	USEPA METHOD 7470
Reagents	Stannous chloride dissolved in hydrochloric acid to prevent clogging of mercury analyzer, per instrument manufacturer's recommendation.	Stannous chloride dissolved/suspended in sulfuric acid.
Procedures	1)Sampling and gas stream switching performed automatically by mercury analyzer. 2)Working Mercury standard prepared monthly in 2% nitric; calibration standards prepared fresh daily.	1)Sampling and gas stream switching performed manually by analyst. 2)Working Mercury standard prepared fresh daily and acidity maintained at 0.15% nitric.
QC – Calibration Verification	1) Known reference sample (ICV) analyzed daily. 2) Calibration verified after every 10 samples with CCV.	1) Known reference sample analyzed quarterly. 2) Calibration verified after every 20 samples.
QC - Calibration Blanks	Acceptance criteria employed for 245.1: ± PQL	Acceptance criteria stated in 245.1: ± MDL

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

FIGURE 1

EXAMPLE PAGE FROM MERCURY PREPARATION LOGBOOK

Katahdin Analytical Services, Inc.

Metals Preparation Benchsheet

Reagent Information:
JT Baker HNO3: G41034 JT Baker HCL: N/A JT Baker H2SO4: G20002 Method: 7470
JT Baker KMNO4: MR781 JT Baker K2S2O8: MR780 JT Baker NH2OH-HCl: MR778

Standards/Spiking Information:
1ppm A: ~~[illegible]~~ MW11565
1ppm B: MW11566 Dwm 2-25-09
LCSW = 125uL of 1ppm A to 25mL
Spike(S/P) = 25uL of 1ppm A to 25mL

ICV = 600uL of 1ppm B to 100 mL
S0.2 = 20uL of 1ppm A to 100 mL
S0.5 = 50uL of 1ppm A to 100 mL

S1.0 = 100uL of 1ppm A to 100 mL
S5.0 = 500uL of 1ppm A to 100 mL
S10.0 = 1000uL of 1ppm A to 100 mL

Balance ID: N/A
Water Bath ID: B
Digestion Start Time (@ 96 °C): 13:30
Thermometer ID: ACC-5
Digestion End Time(@ 95 °C): 15:30

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Initial Color	Initial Clarity	Final Color	Final Clarity	Artifacts	Bottle
LCSWZB24HGW0	ZB24HGW0	0.025	L	0.025	L	AQ	HG	DWM	02/24/2009	N/A	N/A	N/A	N/A		
PBWZB24HGW0	ZB24HGW0		L		L	AQ	HG	DWM	02/24/2009	N/A	N/A	N/A	N/A		
SC0797-001T	ZB24HGW0		L		L	AQ	HG	DWM	02/24/2009						
SC0805-007T	ZB24HGW0		L		L	AQ	HG	DWM	02/24/2009						
SC0834-013T	ZB24HGW0		L		L	AQ	HG	DWM	02/24/2009						
SC0835-002 Dwm 2-25-09	ZB24HGW0		L		L	AQ	HG	DWM	02/24/2009						E
SC0836-001	ZB24HGW0		L		L	AQ	HG	DWM	02/24/2009						V
SC0846-013	ZB24HGW0		L		L	AQ	HG	DWM	02/24/2009						E
SC0858-001T	ZB24HGW0		L		L	AQ	HG	DWM	02/24/2009						
SC0858-002T	ZB24HGW0		L		L	AQ	HG	DWM	02/24/2009						
SC0868-013	ZB24HGW0		L		L	AQ	HG	DWM	02/24/2009						E
SC0868-015	ZB24HGW0		L		L	AQ	HG	DWM	02/24/2009						E
SC0834-013TA			L		L										
013TS			L		L										

Dwm 2-25-09

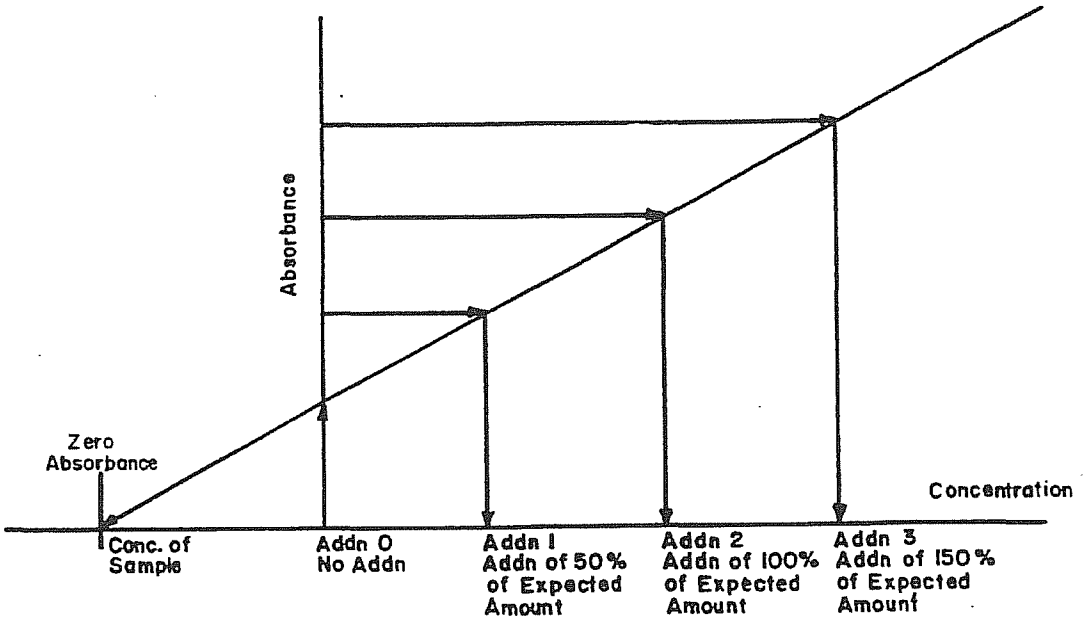
Digestion performed by: Dwm On: 2-24-09

Page: ZB071 Revision: 00

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

FIGURE 2

STANDARD ADDITIONS PLOT



KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-627
Revision History
Cover Page
Page 1

TITLE: TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020

Prepared By: George Brewer Date: 03/01

Approved By:

Group Supervisor: George Brewer Date: 04/02/01

Operations Manager: Joel C. Burton Date: 3/29/01

QA Officer: Deborah J. Nadeau Date: 03·27·01

General Manager: Dennis P. Hufnagle Date: 04/03/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Changed acid solution conc. changed Run ID Naming convention added data reduction and reporting procedures updated standards tables (4-8) updated Table 10 in include ISIS configuration	LAD	02-16-05	02-16-05
02	Sect. 4.2 - changed tubing size sect. 5 - changed acid conc.'s sect. 7 - major changes to reflect current practises including reporting data in the metals database. sect. 8: major changes updating acceptance criteria. Updated tables 1, 3 → 8, 10 & 11	LAD	04/06	04/06
03	Updated Tables 4, 5 and 6 with current standards. Updated Table 1 with serial dilution, Post Digestion Matrix spike, MSA, ICS-A, ICS-AB and IDL minimum frequency or criteria. Updated Sect. 8 regarding client specific requirements.	LAD	07/07	07/07
04	Section 7.18-changed instrument identifier to reflect new instrument; section 8-changed acceptance criteria and ICSAB analyte list; Table 1-updated acceptance criteria and corrective action to QC. Table 3-added all analytes to list-removed "for information only" list.	LAD	04/08	04/08
05	Updates to reflect changes from 6020 to 6020A. Added Hardness by calculation attachment. Added LLOQ requirement and criteria to Sect 8 and Table 1. Added criteria to analyze PQL Std. at beginning and END of run.	LAD	02/09	02/09

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-627-05**, titled **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**
.

Recipient: _____Date:_____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-627-05**, titled **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

Recipient: _____Date:_____

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

1.0 SCOPE AND APPLICATION

Inductively coupled plasma-mass spectrometry (ICP-MS) is applicable to the determination of sub-ppb (ug/L) concentrations of a large number of elements in water samples and in waste extracts or digests. When dissolved constituents are required, samples must be filtered and acid-preserved prior to analysis. No digestion is required prior to analysis for dissolved elements in water samples. Acid digestion prior to filtration and analysis is required for groundwater, aqueous samples, industrial wastes, soils, sludges, sediments, and other solid wastes for which total (acid-leachable) elements are required.

ICP-MS has been applied to the determination of over 60 elements in various matrices. Analytes for which EPA has demonstrated the acceptability Method 6020 in a multi-laboratory study on solid wastes are listed as "analytes" in Table 3. Instrument detection limits, sensitivities, and linear ranges will vary with the matrices, and operating conditions.

If Method 6020 is used to determine any analyte not listed in Table 3, it is the responsibility of the analyst to demonstrate the accuracy and precision of the method in the waste to be analyzed. The analyst is always required to monitor potential sources of interferences and take appropriate action to ensure data of known quality.

An appropriate internal standard is required for each analyte determined by ICP-MS. Recommended internal standards are ^{6}Li, ^{45}Sc, ^{89}Y, ^{103}Rh, ^{115}In, ^{159}Tb, ^{165}Ho, and ^{209}Bi. The lithium internal standard should have an enriched abundance of ^{6}Li, so that interference from lithium native to the sample is minimized. Other elements may need to be used as internal standards when samples contain significant amounts of the recommended internal standards.

1.1 Definitions:

CCB - Continuing Calibration Blank - An analyte-free solution consisting of acidified reagent water used to verify calibration accuracy periodically during analysis.

CCV - Continuing Calibration Verification - A midrange standard used to verify calibration accuracy periodically during analysis.

Duplicate - A second aliquot of a sample that is prepared and analyzed in the same way as the original sample in order to determine the precision of the method.

ICB - Initial Calibration Blank - An analyte-free solution consisting of acidified reagent water used to verify calibration accuracy.

ICP-MS - Inductively Coupled Plasma Mass Spectrometry.

ICS - Interference Check Samples - Two standards (ICS-A and ICS-AB) used to verify the effectiveness of interference correction equations. Solution ICS-A contains only interferents (Al, Ca, Fe, Mg, Na, K, P, S, Mo, Ti, C, Cl) at high concentrations; solution ICS-AB contains interferents at the same concentrations as well as analytes at low (20 ug/L) concentrations.

ICV - Initial Calibration Verification - A standard made from a source independent from the calibration standards and with analyte concentrations different from those in the CCV; used to verify the accuracy of the instrument calibration.

IDL - Instrument Detection Limit - The lowest concentration of an analyte that can be determined with 95% confidence.

LCS - Laboratory Control Sample - A standard or solid reference material that has been brought through the sample preparation process.

PB - Preparation Blank - Reagent water that has been brought through the sample preparation process.

Post-Digestion Spike - An aliquot of a sample to which a known amount of analyte has been added before analysis and after digestion, if digestion is required.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the IDL.

Matrix Spike - An aliquot of a sample to which a known amount of analyte has been added before digestion.

Serial Dilution - The dilution of a sample by a factor of five. When corrected by the dilution factor, the measured analyte concentrations of the diluted sample should agree with those of the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in ICP-MS analysis by USEPA Method 6020 who are knowledgeable in the recognition and in the correction of spectral, chemical, and physical interferences in ICP-MS. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in ICP-MS analysis by USEPA Method 6020 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented, and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

Samples, sample digestates, standards, and other reagents used in ICP analysis may contain high concentrations of acids and toxic metals. Spilled samples and reagents should be cleaned up from instrument and laboratory surfaces immediately.

Liquid argon represents a potential cryogenic and suffocation hazard and safe handling procedures should be employed at all times when handling liquid argon tanks and fittings. Safety glasses and cryogenic-resistant gloves should be worn when changing or adjusting argon tanks.

The Agilent 7500 ICP-MS spectrometer is safety-interlocked to prevent user exposure to harmful electrical voltages, radio frequency emissions, ultraviolet radiation, high temperatures, and other hazards. At no time should the operator attempt to disable these interlocks or operate the instrument if any safety interlock is suspected to be disabled

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention and waste minimization techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Samples, sample digestates, standards, and other reagents used in ICP-MS spectrometry may contain high concentrations of acids and toxic metals. They should be disposed of in a manner appropriate to the types of hazards they present. All digested samples and excess reagents and standards should be disposed of in the satellite waste container for corrosive wastes (labeled “Waste Stream A”) that is located in the Metals Instrument lab. Further information regarding waste

classification and disposal may be obtained by consulting Katahdin Analytical Environmental Health and Safety Manual and the Department Manager.

2.0 SUMMARY OF METHOD

2.1 Prior to analysis, samples that require total ("acid-leachable") values must be digested using appropriate sample preparation methods (such as USEPA Methods 3005 - 3051).

2.2 USEPA Method 6020 describes the multi-elemental determination of analytes by ICP-MS. The method measures ions produced by a radio-frequency inductively coupled argon plasma. Analyte species originating in a liquid are nebulized and the resulting aerosol transported by argon gas into the plasma torch. The ions produced are entrained in the plasma gas and introduced, by means of a vacuum interface, into a mass spectrometer. The ions produced in the plasma are sorted according to their mass-to-charge ratios and quantified with a channel electron multiplier. Interferences must be assessed and valid corrections applied or the data flagged to indicate problems. Interference correction must include compensation for background ions contributed by the plasma gas, reagents, and constituents of the sample matrix.

3.0 INTERFERENCES

Isobaric elemental interferences in ICP-MS are caused by isotopes of different elements forming atomic ions with the same nominal mass-to-charge ratio (m/z). The Agilent 7500 ChemStation data system is used to correct for these interferences. This involves determining the signal for another isotope of the interfering element and subtracting the appropriate signal from the analyte isotope signal. Isobaric molecular and doubly-charged ion interferences in ICP-MS are caused by ions consisting of more than one atom or charge, respectively. Most isobaric interferences which could affect ICP-MS determinations have been identified. Examples include $ArCl^+$ ions on the As signal and MoO^+ ions on the cadmium isotopes. While the approach used to correct for molecular isobaric interferences is demonstrated below using the natural isotopic abundances from the literature, the most precise coefficients for an instrument must be determined from the ratio of the net isotope signals <u>observed</u> for a standard solution at a concentration providing suitable (<1 percent) counting statistics. Because the ^{35}Cl natural abundance of 75.77 percent is 3.13 times the ^{37}Cl abundance of 24.23 percent, the chloride correction for arsenic can be calculated (approximately) as follows (where the $^{38}Ar^{37}Cl^+$ contribution at m/z 75 is a negligible 0.06 percent of the $^{40}Ar\ ^{35}Cl^+$ signal):

Corrected ^{75}As signal (using natural isotopic abundances for coefficient approximations) =

(m/z 75 signal) - (3.13) (m/z 77 signal) + (2.73) (m/z 82 signal),

where the final term adjusts for any selenium contribution at 77 m/z.

NOTE: Arsenic values can be biased high by this type of equation when the net signal at m/z 82 is caused by ions other than $^{82}Se^+$, (e.g., $^{81}BrH^+$ from bromine wastes or ^{82}Kr from krypton contamination in the Ar).
Similarly:

Corrected ^{114}Cd signal (using natural isotopic abundances for coefficient approximations) =(m/z 114 signal) - (0.027)(m/z 118 signal) - (1.63)(m/z 108 signal),

where last 2 terms adjust for any tin or MoO^+ contributions at m/z 114.

NOTE: Cadmium values will be biased low by this type of equation when $^{92}ZrO^+$ ions contribute at m/z 108. Also, use of m/z 111 for Cd is even subject to direct ($^{92}ZrOH^+$) ions and indirect ($^{90}ZrO^+$) additive interferences when Zr is present.

NOTE: As for the arsenic equation above, the coefficients in the Cd equation are only illustrative. The most appropriate coefficients for an instrument can be determined from the ratio of the net isotope signals observed for a standard solution at a concentration providing suitable (<l percent) counting precision.

The interference correction equations that are used by this laboratory in performing USEPA Method 6020 are listed in Table 3. The accuracy of these types of equations is based upon the constancy of the observed isotopic ratios for the interfering species. Corrections that presume a constant fraction of a molecular ion relative to the "parent" ion have not been found to be reliable, e.g., oxide levels can vary. If a correction for an oxide ion is based upon the ratio of parent-to-oxide ion intensities, the correction must be adjusted for the degree of oxide formation by the use of an appropriate oxide internal standard previously demonstrated to form a similar level of oxide as the interferent. This type of correction has been reported for oxide-ion corrections using ThO^+/Th^+ for the determination of rare earth elements. The use of aerosol desolvation and/or mixed plasmas have been shown to greatly reduce molecular interferences (the Agilent 7500 ICP-MS spectrometer employs spray chamber cooling to effect aerosol desolvation). These techniques can be used provided that method detection limit, accuracy, and precision requirements for analysis of the samples can be met.

3.1 Physical interferences are associated with the sample nebulization and transport processes as well as with ion-transmission efficiencies. Nebulization and transport processes can be affected if a matrix component causes a change in surface tension or viscosity. Changes in matrix composition can cause significant signal suppression or enhancement. Dissolved solids can deposit on the nebulizer tip of a pneumatic nebulizer and on the interface skimmers (reducing the orifice size and the instrument performance). Total solid levels below 0.2% (2,000 mg/L) are recommended to minimize solid deposition. An internal standard can be used to correct for physical interferences, if it is carefully matched to the analyte so that the two elements are similarly affected by matrix changes. The internal standard used

should differ from the analyte of interest by no more than 50 amu. See table 13 for a list of internal standards used. When the intensity level of an internal standard is less than 70 percent or greater than 120 percent of the intensity of the first standard used during calibration, the sample must be reanalyzed after a fivefold (1+4) or greater dilution has been performed.

3.2 Memory interferences can occur when there are large concentration differences between samples or standards that are analyzed sequentially. Sample deposition on the sampler and skimmer cones, spray chamber design, and the type of nebulizer affect the extent of the memory interferences that are observed. The rinse period between samples must be long enough to eliminate significant memory interference.

4.0 APPARATUS AND MATERIALS

4.1 Agilent 7500 ICP-MS system, consisting of the Agilent 7500 ICP-mass spectrometer and its controlling computer data station. The spectrometer is capable of providing resolution better than or equal to unit resolution at 10% peak height. The Agilent 7500 mass range of 2-260 amu exceeds the method requirement of 2- 240 amu. The Agilent 7500 ChemStation software allows automatic corrections for isobaric interferences and correction for internal standard responses as required by the method. All critical argon flows including nebulizer argon are under mass flow controller control and a peristaltic pump is used for sample introduction. Peripheral equipment include a Cetac ASX-500 random-access autosampler, and Agilent ISIS dilution and prep station, and an Agilent LaserJet printer.

4.2 Peristaltic pump tubing – 3-stop Tygon white-white (1.02 mm ID), orange-blue (0.25 mm ID), and 2-stop red-red (1.14 mm ID). 3-stop Pharmed blue-yellow (1.52 mm ID).

4.3 15 ml 17x100 mm polypropylene or polystyrene disposable test tubes for samples and 50 ml polypropylene centrifuge tubes for standards.

4.4 Automatic adjustable-volume pipetters of suitable precision and accuracy. Calibrated Eppendorf Reference pipets and Finn digital pipets are appropriate.

4.5 Trace metal grade pipette tips.

4.6 Volumetric glassware or plasticware of suitable precision and accuracy.

4.7 Talc free vinyl gloves.

4.8 Argon gas supply (high purity grade gas or liquid, 99.99%).

4.9 For the determination of trace levels of elements, contamination and loss are of prime consideration. Potential contamination sources include improperly cleaned laboratory apparatus and general contamination within the laboratory environment from dust etc. A clean laboratory work area, designed for trace element sample handling must be used. Standards, samples and blanks should be exposed to the laboratory environment as little as possible. The use of preparation blanks and spikes should be used to verify the absence of sources of contamination and loss. If necessary, polypropylene sample tubes should be rinsed and stored in dilute acid prior to use.

NOTE: Chromic acid must not be used for cleaning glassware for trace metals analysis.

5.0 REAGENTS

5.1 Acids used in the preparation of standards and for sample processing must be of high purity. Redistilled acids are recommended because of the high sensitivity of ICP-MS. Mallincrodt/Baker "Instra-Analyzed" trace-metals grade acids are appropriate. It is important to match the acid concentration in standards and samples. Concentrations of antimony and silver between 50-500 ug/L require 1% (v/v) HCl for stability; for concentrations above 500 ug/L additional HCl will be needed. For this reason, it is recommended that antimony and silver concentrations in samples and standards be maintained below 500 ppb wherever possible. Acids are received in poly-coated glass bottles, and are stored under the hood in the Metals sample preparation laboratory at room temperature until use. All acids are considered to have a shelf life of three years from date of receipt unless otherwise indicated by the vendor. Refer to the current revision of Katahdin SOP CA-105, Reagent and Solvent Handling, for further details.

5.2 Reagent water, trace metals free, equivalent to ASTM Type 1 (ASTM D 1193), >18 Megohm/centimeter resistivity. Polyethylene carboys are filled with reagent water in the glassware prep room and are transported to the laboratory for use. Reagent water is replaced at least weekly.

5.3 Single element and multielement stock standard solutions – purchased standards prepared from high purity salts or metals, and supplied by the vendors with certificates of purity and analysis. Refer to Tables 4 and 5 for a listing of stock standards required, and to Table 8 for element concentrations in stock standards. Purchased stock standards are received in polyethylene containers and are stored in their original containers at room temperature in the Metals Standards Preparation Laboratory. All purchased stock standards are given an expiration date as indicated by the manufacturer. Refer to the current revision of Katahdin SOP CA-106, Standard Preparation, Documentation and Traceability, for further details.

5.4 Intermediate standard solutions – laboratory-prepared multielement standards that are used in the subsequent preparation of working standards. Refer to Table 5 for a listing of intermediate standards required and for preparation instructions. Refer to Table 7 for element concentrations in intermediate standards. Intermediate standards are stored at room temperature in acid-washed polyethylene containers in the Metals Standards Preparation Laboratory. Intermediate standards are assigned an expiration date of three months from the date of preparation, or the earliest expiration date of a component standard, whichever comes first. Refer to the current revision of Katahdin SOP CA-106, Standard Preparation, Documentation and Traceability, for further details.

5.5 Working standard solutions – laboratory-prepared multielement standards that are used to calibrate the instrument, to provide internal standardization through on-line addition, and to perform all necessary QC checks. Refer to Table 4 for a listing of working standards and for preparation instructions. Refer to Table 6 for element concentrations in working standards. Working standards are stored at room temperature in acid-washed polyethylene containers in the Metals Standards Preparation Laboratory. All working standards except the ICSA and ICSAB solutions are assigned an expiration date of three months from the date of preparation, or the earliest expiration date of a component standard, whichever comes first. The ICSA and ICSAB solutions are assigned an expiration date of one week from the date of preparation, or the earliest expiration date of a component standard, whichever comes first. Refer to the current revision of Katahdin SOP CA-106, Standard Preparation, Documentation and Traceability, for further details.

5.6 Blanks: Three types of blanks are required for the analysis. The calibration blank is used in establishing the calibration curve. The preparation blank is used to monitor for possible contamination resulting from the sample preparation procedure. The rinse blank is used to flush the system between all samples and standards.

5.6.1 The calibration blank consists of the same concentrations of the same acid(s) used to prepare the final dilution of the analyte calibration solutions (currently 1% HNO_3 and 0.5% HCl, v/v, in laboratory reagent grade water). Use of HCl for antimony and silver is cited in Section 5.1.

5.6.2 The preparation blank must be carried through the complete preparation procedure and contain the same volumes of reagents as the associated digested sample solutions.

5.6.3 The rinse blank consists of 4% HNO_3 and 0.5% HCl,v/v, in reagent water. Prepare a sufficient quantity to flush the system between standards and samples.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples to be analyzed for trace metals by ICP-MS should be collected and preserved as described in the following table.

Matrix	Container[1]	Collection Volume/ Weight	Preservation/ Treatment	Holding Time
Aqueous (total)	P, G	250 mL	HNO_3 to pH < 2	6 months
Aqueous (dissolved)	P, G	250 mL	Filter, HNO_3 to pH < 2	6 months
Solid	P, G	10 g	Cool, 4°C	6 months

[1] P = polyethylene or, G = glass

7.0 PROCEDURES

7.1 Instrument control and data acquisition are completely automated through the use of the Agilent Chemstation software. The main Chemstation screen is accessed by double-clicking the "ICP-MS Top" icon on the Windows desktop. Autosampler tables are edited by double clicking the "Edit Sample Log Table" icon on the Windows desktop. In the following discussion, software menu items that are to be selected are printed in boldface. The instrument operating conditions, acquisition parameters, acquisition masses, and internal standards for analysis USEPA Method 6020 are detailed in Table 10.

7.2 Turn on the argon supply at the tank and set the pressure to >700 kPa.

7.3 Turn on the water chiller/recirculator.

7.4 Verify that the exhaust hood is in operation.

7.5 Ensure that the internal standard solution bottle is adequately full. Consumption is approximately 2.5 mL/hour.

7.6 Verify that the rinse station reservoir has an adequate supply of reagent water.

7.7 Verify that the drain reservoir has adequate room to accept the day's drain waste. Empty the reservoir as necessary into an appropriate waste container (Waste Stream A) located in the Hazardous Waste Storage Area.

7.8 Inspect the peristaltic pump tubes for signs of flattening and wear, and replace them as necessary. Clamp the peristaltic pump tubes into the peristaltic pump.

7.9 Open the Chemstation software by double-clicking the "ICP-MS Top" icon. Initiate the plasma by selecting **Instrument>>Instrument Control>>Plasma>>Plasma On**

and allow the instrument to aspirate calibration blank solution for at least 15 minutes. During this warm-up, select **Tune>>Sensitivity>>Start** to start the instrument scanning the mass range. Verify that the flow of sample and internal standard solutions through the uptake lines and into the nebulizer is free from pulsations by introducing an air bubble into each line and observing its progress. Adjust the pump clamp tension on each line to obtain a constant, pulse-free flow.

7.10 After the 15 minute warm-up, check the responses of masses 82 and 83 to insure minimal krypton intereference with selenium. Mass 83 response should be < 2000 counts per second. Then aspirate the Instrument Tune Solution (10 ppb Li, Y, Ce, Tl) and check the responses and RSDs at masses 7, 89, and 205.

7.11 Generate a tune report by selecting **Tune>>File>>Generate Report**. Evaluate the tune report against the tune specifications listed in Table 11. If the tune passes all specifications, proceed to step 7.14.

7.12 If the tune report indicates unacceptable instrument performance for any parameter, initiate an autotune by selecting **Tune>>Autotune>>Run**. The Chemstation software will attempt to tune the instrument to meet the tune specifications, and will generate a new tune report after autotuning. Evaluate the new tune report against the tune specifications listed in Table 11.

7.13 Repeat step 7.12 until all tune specifications have been met. File the final tune report.

7.14 Aspirate a 100 ppb solution of all analytes and run a P/A autotune by selecting **Tune>>Tune>>P/A Factor>>Run**. This will calibrate the pulse and analog modes of the detector. File the P/A report with the Tune report.

7.15 Load the instrument analytical method and calibrations table for USEPA Method 6020 into memory by selecting **Methods>>Load>>K6020.M>>K6020.C**.

7.16 Edit the sequence template "K6020.S" to create an analytical sequence table listing all of the samples to be analyzed. To do this, double click the "Edit Sample Log Table" icon on the Windows desktop. Select **Open>>K6020.S**. Click the drop-down menu at the top left of the screen, and select the **Sample** block. Fill in the sample table with sample IDs, vial numbers, analytical method (K6020.M for all samples), dilution factors, and failure actions. When the sample table is complete, click the drop-down menu at the top left of the screen, and select **Whole List** to view the entire analytical sequence, including all calibration standards and QC samples. Select **Print** to print this table. Save the sample log table under a new name by selecting **Save** and then typing the name. Close the "Edit Sample Log Table" window.

7.17 Load the autosampler racks according to the analytical sequence printout.

7.18 Select **Sequence>>Load and Run Sequence**, and select the appropriate autosampler sequence table from the displayed list. Enter the analyst's initials in the Operator box. Change data file name to appropriate designation. The protocol for naming data files is as follows: the 1st character is a letter that identifies the instrument ("J" for the Agilent 75000 ICP-MS), the 2nd character is a letter that identifies the year of the run ("X" for 2007, "Y" for 2008, etc.), the 3rd character is a letter that identifies the month of the run ("A" for January, "B" for February, etc.), the 4th and 5th characters are numbers that identify the date of the run ("01" for the first day of the month, etc.), and the 6th character is a letter that sequentially identifies the run ("A" for the first run of the day on that instrument, "B" for the second run, etc.). For example, the run identified as "JYA16A" is the first run of the day that was performed on January 16, 2008, using the Agilent 7500 ICP-MS. Select **Run**. The instrument will analyze all samples in the order listed in the table. Analysis must proceed in the sequence described in Table 9 to ensure that all necessary quality control samples are analyzed at the appropriate frequencies. A minimum of three replicate scans is required for all standards and samples. Analysis always begins with the analysis of a calibration blank followed by at least three multielement calibration standards to calibrate the instrument. The system is flushed with rinse blank between each sample and standard, and each sample and standard is aspirated for at least one minute prior to the beginning of mass scanning.

7.19 Analysis continues with analysis of the initial calibration verification standard (ICV) and the initial calibration blank (ICB) to verify the accuracy of the calibration. Refer to Section 8 and Table 1 for additional information.

7.20 A practical quantitation limit standard (PQL) is analyzed at the beginning of the run to verify calibration accuracy at the reporting limit. Refer to Section 8 and Table 1 for additional information.

7.21 A continuing calibration verification standard (CCV) and a continuing calibration blank (CCB) must be analyzed at the beginning of the run, after every ten samples, and at the end of the run to verify the continued accuracy of the calibration. Refer to Section 8 and Table 1 for additional information.

7.22 Interference check standard solutions ICS-A and ICS-AB must be analyzed at the beginning of each run and every 12 hours to verify the adequacy of interference corrections. Refer to Section 8 and Table 1 for additional information.

7.23 All sample analytical results for a particular element that are bracketed (preceded or followed) by failing results in a calibration verification sample (ICV, ICB, CCV, or CCB) for that element must not be reported, except as noted in Sections 8.5, 8.6, and 8.9. The sample must be reanalyzed for the element in question.

7.24 All samples that exceed the linear dynamic range must be diluted and reanalyzed.

7.25 If dilutions of digested samples are performed, the measured element concentrations must be multiplied by the dilution factor prior to reporting. This is

accomplished automatically by entering the dilution factor in the sample log table prior to initiation of analysis.

7.26 If an element has more than one monitored isotope, examination of the concentration calculated for each isotope, or the isotope ratios, will provide useful information for the analyst in detecting a possible spectral interference. Consideration should therefore be given to both primary and secondary isotopes in the evaluation of the element concentration. In some cases, secondary isotopes may be less sensitive or more prone to interferences than the primary recommended isotopes, therefore differences between the results do not necessarily indicate a problem with data calculated for the primary isotopes. In the case of Pb, quantitation is based on the sum of isotopes 206, 207 and 208 to compensate for any variation in naturally occurring isotope ratios. This is accomplished through the use of the interference correction equation for lead.

7.27 Calculations, aqueous samples: Final element concentrations for aqueous samples are reported in units of micrograms per liter (ug/L). The reported concentrations are calculated from measured digestate concentrations as follows:

$$\text{Concentration (ug/L)} = \frac{MC \times DF \times FV}{IV}$$

where:

MC = Measured digestate concentration (ug/L)
DF = Instrument dilution factor
FV = Final digestate volume (L)
IV = Digested sample volume (L)

7.28 Calculations, solid samples: Final element concentrations for solid samples are reported in units of milligrams per kilogram (mg/kg) on a dry weight basis. The reported concentrations are calculated from measured digestate concentrations as follows:

$$\text{Concentration (mg/kg dry weight)} = \frac{MC \times DF \times FV \times 100}{W \times S}$$

where:

MC = Measured digestate concentration (ug/L)
DF = Instrument dilution factor
FV = Final digestate volume (L)
W = Weight of digested wet sample (g)
S = Percent solids

DATA REDUCTION AND REPORTING

7.29 When the analytical run is completed, the analyst should print a run summary and create a data import file.

7.30 Follow these steps to print the run summary: Open the FileView program using the "FIVIEW" icon on the Windows Desktop. Select the data file of interest and move

the required samples into the "Process List". Make sure the "Corrected Data" box is checked. Click the "Process" button. The data will be displayed in a spreadsheet format.

7.31 Select "Configure" from the top menu and "Sublists" from the displayed options. Select "Load Sublist" and then select "K6020" from this list of options. Make sure the "Enable Sublist" box is checked. Click the "OK" button. This will display only the analyte masses in the spreadsheet.

7.32 Select "Quant Info" from the top menu and "Quant Results" from the displayed options. This will display the data in concentration units. Click on the cell in the top left corner of the spreadsheet to highlight all data. Select "Tools" from the top menu and "Import Data into Spreadsheet Application" from the displayed options. Click the "Save" button. The data is transferred to a Microsoft Excel spreadsheet. Minimize this window and return to the FileView spreadsheet.

7.33 Select "Configure" from the top menu and "Sublists" from the displayed options. Select "Load Sublist" and then select "INTSTDS" from this list of options. Make sure the "Enable Sublist" box is checked. Click the "OK" button. This will display only the internal standard masses in the spreadsheet.

7.34 Select "Count Info" from the top menu and "Counts/sec" from this list of options. This will display the data in counts per second units. Click on the cell in the top left corner of the spreadsheet to highlight all data. Select "Tools" from the top menu and "Import Data into Spreadsheet Application" from the displayed options. Click the "Save" button. The data is transferred to a Microsoft Excel spreadsheet. Copy the internal standard names and results and paste into the analyte results Excel spreadsheet.

7.35 In a separate cell of the Excel spreadsheet, input a formula to calculate the percent recovery of the internal standard relative to that of the calibration blank for each internal standard measurement of each sample.

7.36 Input a formula into the sample results cells to correct the data for dilution.

7.37 Format, print and save (as FileName.XLS, e.g. JYA28A.XLS) this run summary.

7.38 Follow these steps to create the data import file: Open the FileViewNT program using the "FIVIEWNT" icon on the Windows Desktop. Select the data file of interest and move the required samples into the "Process List". Make sure the "Corrected Data" box is checked. Click the "Process" button. The data will be displayed in a spreadsheet format.

7.39 Select "Configure" from the top menu and "Sublists" from the displayed options. Select "Load Sublist" and then select "K6020" from this list of options. Make sure the "Enable Sublist" box is checked. Click the "OK" button. This will display only the analyte masses in the spreadsheet.

7.40 Select "Quant Info" from the top menu and "Quant Results" from the displayed options. This will display the data in concentration units.

7.41 Select the "Transpose" from the menu.

7.42 Select "Tools" from the top menu and "Copy Selected Data to CSV File" from this list of options. Set the name the file as "FileName.CSV", e.g., "JYA28A.CSV". Click the "Save" button.

To import data into the Metals Database:

7.43 Select the "ICPMS Import" icon from the Windows Desktop, the ICPMS Import window will appear. Enter the datafile name without extension, (e.g., "JYA28A") and click the "Import" button.

7.44 When the "Import finished" message appears, close the ICPMS Import window and select the "KIMS_METALS" icon from the Windows Desktop. The Metals Database Main Menu will appear.

7.45 Select the "Reporting Menu" button. From the Reporting Menu screen select the Batch QC Menu button and then the "Calculate Batch QC" button.

7.46 From the resulting list of QC results, deselect any items that fail run QC or do not need ICP-MS analysis. Click on the "Accept Selected Batch QC" button.

7.47 From the Metals Main Menu, select the "Additional Data Handling" button. The Data Menu will appear. Select the "Report Added Compounds" button.

7.48 From the resulting list of sample results, deselect any items that fail run QC or do not need ICP-MS analysis. Click on the "Accept Data" button.

7.49 Once all associated data from an analysis run have been processed, go to the Generate Coverage portion of the Export Menu and print the Run Log and Logbook Page for the analysis run.

7.50 Combine these reports with the raw data printout and the Run Info Sheet and scan into a PDF format. Save in the "ICPMS DATA" section of the "METPDF" directory on the IMAGESERVER.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 6020 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery

ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgements. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

INITIAL DEMONSTRATION OF PERFORMANCE

8.1 Instrument detection limits (IDL) are determined quarterly for each analyte analyzed on each instrument. This determination requires seven replicate analyses of a reagent blank, performed on three non-consecutive days. The standard deviation of the 21 analyses is multiplied by three to obtain the IDL. For more information on performing IDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.2 Method detection limits (MDL) are determined annually for each analyte analyzed by each method on each instrument. This determination requires at least seven replicate digestions and analyses of reagent water spiked at 3-5 times the anticipated MDL for each analyte. MDLs differ from IDLs in that the seven replicates are digested prior to analysis, and they may be analyzed on a single day. The standard deviation of the 7 (or more) replicate analyses is multiplied by the Student's t-value to obtain the MDL. For more information on performing MDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.3 A Lower Limit of Quantitation Check (LLQC) sample must be prepared and analyzed annually or on an as-needed basis to confirm the laboratory's Practical Quantitation Limits (PQLs). The LLQC sample is equivalent to the PQL standard (Section 8.9) but is carried through the entire sample preparation and analysis process. Element recoveries for the LLQC sample must fall within 70% to 130% of the expected concentrations to confirm the previously established PQLs.

ANALYTICAL RUN QC SAMPLES

8.4 Initial instrument calibration: The instrument is calibrated by running a calibration blank and at least three multielement calibration standards. For each element, calibration is performed fitting a single order equation to the calibration data, as follows:

$$Y=aX + [Blank]$$

where:

Y = Concentration (ug/L)
X = Measured signal intensity (counts per second)
a = Slope of the calibration line
[Blank] = Measured signal intensity of the calibration blank

Fitting the calibration equation through the measured intensity of the calibration blank, rather than through the y-intercept of the line, provides the best calibration accuracy at the low end of the calibration range. When this equation is used, however, the Agilent software does not calculate a calibration coefficient. For this reason, calibration accuracy at the high end of the calibration range is checked by reanalyzing the highest calibration standard as a sample immediately after instrument calibration. Recoveries for all elements must be within 90% to 110% of the true value in the high calibration standard. If the high calibration standard fails, result for the failing elements may not be reported from the run until the problem is corrected and a passing high calibration standard has been analyzed. Calibration accuracy in the middle of the calibration range is verified by analysis of the CCV solution (see Section 8.6 below). Calibration accuracy at the reporting limit is verified by analysis of the PQL Check Standard (see Section 8.7 below).

8.5 The intensities of all internal standards must be monitored for every analysis. When the intensity of any internal standard fails to fall between 70 and 120 percent of the intensity of that internal standard in the initial calibration standard, the following procedure is followed. The sample must be diluted fivefold (1+4) and reanalyzed with the addition of appropriate amounts of internal standards. This procedure must be repeated until the internal standard intensities fall within the prescribed window. The intensity levels of the internal standards for the calibration blanks (ICB and CCBs) and calibration verification standards (ICV and CCVs) must agree within ± 20 percent of the intensity level of the internal standard of the original calibration solution. If they do not agree, terminate the analysis, correct the problem, recalibrate, verify the new calibration, and reanalyze the affected samples.

8.6 An Initial Calibration Verification (ICV) solution is analyzed after the initial calibration to check calibration accuracy. The ICV solution is prepared by combining compatible elements from standard sources different than those of the calibration standards and at concentrations within the linear working range of the instrument. The results of the ICV must fall within 90% to 110% of the expected values. If the ICV fails, result for the failing elements may not be reported from the run, unless the ICV recovery is greater than 110% and the sample result is less than the PQL.

8.7 Continuing Calibration Verification (CCV) solutions are analyzed after the initial calibration, after every ten samples, and at the end of the analytical run. The CCV solution is prepared using the same standards used for calibration at concentrations

near the mid-point of the calibration curve. Results of the CCVs must fall within 90% to 110% of the expected values. If a CCV fails, results for the failing elements may not be reported from the run, unless the CCV recovery is greater than 110% and the sample result is less than the PQL.

8.8 A Practical Quantitation Limit (PQL) Check Standard or low level conintuing calibration verification (LLCCV) is analyzed at the beginning of each run (after the ICV and ICB samples) and at the end of each run. Element concentrations in this solution are one-fifth the laboratory's practical quantitation limit (assuming a 5-fold dilution of all digestates during analysis). Element recoveries for the PQL Check Standard must fall within 70% to 130% of the expected values (unless the samples analyzed are for the Department of Defense (80% to 120% recovery limits) or other client-specific limits are imposed). If the PQL Check Standard fails, results for the failing elements may not be reported from the run, unless the PQL Check Standard recovery is greater than the high limit and the sample result is less than the PQL.

8.9 A calibration blank solution is analyzed after each ICV and CCV. A calibration blank that is analyzed after the ICV is called an Initial Calibration Blank (ICB). A calibration blank that is analyzed after a CCV is called a Continuing Calibration Blank (CCB). The absolute values of results of ICBs and CCBs must be less than the applicable reporting limit (or PQL) for each element. The reporting limit should be determined on a project specific basis, taking into account the data quality objectives for the project. This information must be communicated through a project QAPP and through the Katahdin project manager. When no project specific reporting limit is specified, the laboratory PQL shall be used. Some project specific limits may require reporting down to the MDL or IDL and taking corrective action based on these levels. Results that fall between the PQL and the IDL or MDL must always be flagged as "estimated" with a "J".

8.10 If an ICB or a CCB fails the acceptance criteria of less than the reporting limit, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has been analyzed, with the following exception. If the result for an ICB or CCB is greater than the PQL (or reporting limit), sample results that are less than the PQL (or reporting limit) or that are greater than or equal to ten times the measured ICB or CCB concentration may be reported. Also, for failing elements, all samples analyzed after the last passing CCB must be reanalyzed, with the exception noted above.

8.11 To obtain analyte data of known quality, it is necessary to measure more than the analytes of interest in order to apply corrections or to determine whether interference corrections are necessary. If the concentrations of interference sources (such as C, Cl, Mo, Zr, W) are such that, at the correction factor, the analyte is less than the limit of quantitation and the concentration of interferents are insignificant, then the data may go uncorrected. Note that monitoring the interference sources does not necessarily require monitoring the interferent itself, but that a molecular species may be monitored to indicate the presence of the interferent. When correction equations are used, all QC criteria must also be met.

Extensive QC for interference corrections are required at all times. The monitored masses must include those elements whose hydrogen, oxygen, hydroxyl, chlorine, nitrogen, carbon and sulfur molecular ions could impact the analytes of interest. Interference check solutions ICS-A and ICS-AB are analyzed at the beginning of each run and at least every 12 hours during the run to verify the effectiveness of interference corrections. Solution ICS-A contains high concentrations of interferents (Al, Ca, Fe, Mg, Na, P, K, S, C, Cl, Mo, and Ti) only. These should recover between 80% and 120% of the true value. The measured concentrations of other elements in this solution should be very low, indicating that interfering mass correction equations are adequate. Solution ICS-AB contains interferents at the same high concentrations, and all other analytes at 20 ug/L. Measured recoveries for all analytes should be within 80% to 120% of the true values.

PREPARATION BATCH QC SAMPLES

Each digestion batch of twenty or fewer samples will contain a preparation blank and a laboratory control sample. Each batch will also contain one or more of the following QC samples: laboratory control sample duplicate, sample duplicate, matrix spiked sample, or matrix spiked sample duplicate.

8.12 A preparation blank (PBW or PBS), consisting of reagent water carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. The results of preparation blanks must be less than the Practical Quantitation Level (PQL) (or project specific reporting limit, if applicable) for each element. If a preparation blank fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested, with the following exception. If the result for a preparation blank is greater than the PQL or reporting limit, associated sample results that are less than the PQL or reporting limit or that are greater than or equal to ten times the measured preparation blank concentration may be reported.

8.13 A laboratory control sample (LCSW, LCSO, or LCSS), consisting of spiked reagent water or a solid reference material carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. Results for laboratory control samples must fall within 80% to 120% of the expected value, unless vendor-supplied limits (for solid reference materials) or laboratory-generated statistical limits are available. If a laboratory control sample fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested, with the following exception. If the recovery of a laboratory control sample is greater than 120%, associated sample results that are less than the PQL or reporting limit may be reported.

SAMPLE MATRIX QC SAMPLES

8.14 The relative percent difference (RPD) between matrix duplicate, matrix spike duplicate, or laboratory control duplicate sample results is calculated as follows:

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

where:

D_1 = First sample or LCS result
D_2= Second (duplicate) sample or LCS result

A control limit of 20% RPD is applied to duplicate analysis, if the result is greater than 100 times the instrument detection limit. If the matrix duplicate analysis fails, the associated sample result must be flagged on the report of analysis.

8.15 The recovery for each element in a spiked sample must fall within 75% to 125% of the actual value if the result for the unspiked sample is less than four times the amount of spike added. If a recovery fails, the associated sample result must be flagged on the report of analysis. The spike recovery should be calculated as follows:

$$\text{Recovery (\%)} = \frac{S-U}{SA} * 100\%$$

where:

S = Measured concentration of spiked aliquot
U = Measured concentration of unspiked aliquot
SA = Amount of spike added

8.16 A serial dilution is analyzed to check for chemical or physical interferences. If the analyte concentration of a sample is sufficiently high (minimally, 50 x IDL), the measured concentration of a five-fold dilution (1:5 dilution) of the sample should agree within 90% to 110% of the original determination. The percent difference between the original sample and the serial dilution should be calculated as follows:

$$\text{Difference (\%)} = \frac{|L-S|}{S} * 100\%$$

where:

L = Serial dilution result (corrected for dilution)
S = Original sample result

If the serial dilution analysis fails, a matrix interference should be suspected. The action taken is dependent upon project requirements. The associated sample result may be flagged on the report of analysis, the sample may be reanalyzed at dilution to eliminate the interference, or a post-digestion spike may be performed (see section 8.16).

8.17 An analyte spike that is added to an aliquot of a prepared sample, or its dilution, should be recovered within 80% to 120% of the known value if the result for the unspiked aliquot is less than four times the amount of spike added. If the post-digestion spike is not recovered within these limits, the sample should be diluted

and reanalyzed to compensate for the matrix interference or the method standard additions may be employed.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined annually per type of instrument and filed with the Metals Department Manager and with the QAO.

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit and Instrument Detection Limit Studies, for procedures on determining the MDL. Refer to the current revisions of USEPA Method 6020 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846 Update II, September 1994, Method 6020.

Agilent 7500 ICP-MS ChemStation Operator's Manual, Agilent Technologies, Inc., 2000.

List of Tables

Table 1	QC Requirements
Table 2	Summary of Method Modifications
Table 3	Isotopes Monitored and Correction Equations Used
Table 4	Preparation of Calibration and QC Standards
Table 5	Preparation of Intermediate Standards
Table 6	Element Concentrations in Working Standards
Table 7	Element Concentrations in Intermediate Standards
Table 8	Element Concentrations in Stock Standards
Table 9	Required Analytical Sequence
Table 10	Instrument Operating Conditions
Table 11	Instrument Tune Specifications
Table 12	Method Tune Specifications
Table 13	Reported Isotopes and Internal Standards
Attachment 1	Hardness by Calculation

TABLE 1

QC REQUIREMENTS

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
Initial Calibration, minimum 3 points plus a calibration blank.	Daily prior to sample analysis.	Correlation coefficient (r) ≥ 0.998	Recalibrate
Initial Calibration Verification (ICV), prepared from a second source.	Before beginning a sample run.	Recovery within ± 10% of true value.	Do not use results for failing elements, unless ICV >110% and sample result < PQL/reporting limit.
Initial Calibration Blank (ICB)	Immediately after the ICV.	Absolute value of ICB < PQL or project specific reporting limit.	Do not use results if sample ≥ PQL/reporting limit and < 10x ICB level.
PQL Standard or LLCCV	At beginning and end of run	70-130% (80%-120% for D.O.D. or other projects) of true value	Do not use results for failing elements, unless PQL rec.> upper limit and sample result < PQL/reporting limit.
Continuing Calibration Verification (CCV)	At beginning of run, after every 10 samples, and at end of run.	Recovery within ± 10% of true value.	Do not use sample results for failing elements, unless CCV >110% and sample result < PQL/reporting limit.
Continuing Calibration Blank (CCB)	Immediately after every CCV	Absolute value of CCB < PQL or project specific reporting limit.	Do not use sample results if ≥ PQL/reporting limit and < 10x CCB level.
Interference Check Solution A (ICS-A)	Before analyzing samples, and every 12 hours during a run.	Interferents: Recovery within ± 20% of true value. Analytes: No criteria established (Project specific criteria may apply)	Do not use sample results for failing elements.
Interference Check Solution AB (ICS-AB)	Before analyzing samples, and every 12 hours during a run.	Recovery within ± 20% of true value.	Do not use sample results for failing elements, unless ICSAB >120% and sample result < PQL/reporting limit.
Preparation Blank (PBW/PBS)	One per digestion batch of 20 or fewer samples.	Less than PQL (standard practice), or based on the project specific guidelines.	1) Investigate source of contamination. 2) Redigest and reanalyze all associated samples if sample concentration ≥ PQL and <10x the blank conc.

TABLE 1 (continued)

QC REQUIREMENTS

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
Laboratory Control Sample (LCSW/LCSS/LCSO)	At least one per digestion batch of 20 or fewer samples.	Recovery within ± 20% of true value, unless vendor-supplied or statistical limits have been established.	1) Investigate source of problem. 2) Redigest and reanalyze all associated samples, unless LCS >120% and sample result < PQL.
Matrix Spike Sample (S)	See section 8.11	Recovery ± 25% of true value, if sample < 4x spike added.	Flag results and/ or perform post-digestion spike.
Duplicate Sample (D), Matrix Spike Duplicate (P), or LCS Duplicate (LC2W/LC2S/LC2O)	See section 8.11	1) RPD ≤ 20%, if sample > 100x IDL.	Flag results
Post-Digestion Matrix Spike (A)	When serial dilution fails and analyte concentration < 100 x MDL.	Recovery ± 20% of true value, if sample < 4x spike added.	Flag results and/or analyze sample by method of standard additions.
Serial Dilution (L)	1 per digestion batch	If original sample result is at least 50x IDL, 5-fold dilution must agree within ± 10% of the original result.	Flag result or dilute and reanalyze sample to eliminate interference.
Internal Standard (IS) Appropriate IS required for all analytes in all samples. Mass of IS must be <50 amu different from that of analyte.		1) For each sample, IS intensity within 70%-120% of that of initial calib. blank. 2) For ICV, ICB, CCV, and CCB, IS intensity within 80%-120% of that in initial calib. blank.	Do not use results for failing elements.
Instrument Detection Limit (IDL) Study	Quarterly.	IDL < MDL PQL at least 2-3x IDL	1) Repeat IDL study. 2) Raise PQL.
Method Detection Limit (MDL) Study	Annually.	PQL at least 2-3x MDL	1) Repeat MDL study. 2) Raise PQL.
Lower Limit of Quantitation Check (LLQC) Sample	Digest and analyze annually or as needed to confirm PQLs	70% - 130% of true value	Reevaluate PQLs
Method of Standard Additions	When matrix interference is suspected	r ≥ 0.995	Dilute and reanalyze sample to eliminate interference.

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-627-05	METHOD 6020, current revision
Apparatus/Materials		
Reagents		
Sample preservation/ handling		
Procedures		
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		
QC - Calibration Blanks	Acceptance criteria employed for 6020: ± PQL	Acceptance criteria stated in 6020: <10% of PQL

TABLE 3

ISOTOPES MONITORED AND CORRECTION EQUATIONS USED FOR USEPA METHOD 6020

Element Class	Element	Sym-bol	Isotopes Monitored	Correction Equations [See note 1]
Analytes	Aluminum	Al	27	
	Antimony	Sb	121, 123	
	Arsenic	As	75	^{75}As = (75)*1 - (77)*2.95 + (82)*2.548 – (83)*2.571 [See note 2]
	Barium	Ba	135, 137	
	Beryllium	Be	9	
	Boron	B	11	
	Cadmium	Cd	106, 108, 111, 114	^{111}Cd = (111)*1 – (108)*1.073 + (106)*0.764 [See note 3] ^{114}Cd = (114)*1 – (118)*0.0268 [See note 4]
	Calcium	Ca	44	^{44}Ca = (44)*1 – (88)*0.0169 [See note 7]
	Chromium	Cr	52, 53	
	Cobalt	Co	59	
	Copper	Cu	63, 65	
	Iron	Fe	54, 56, 57	^{54}Fe = (54)*1 – (52)*0.0282 [See note 8] ^{57}Fe = (57)*1 – (43)*0.03 [See note 9]
	Lead	Pb	206, 207, 208	^{208}Pb = (208)*1 + (206)*1 + (207)*1 [See note 5]
	Magnesium	Mg	25	
	Manganese	Mn	55	
	Molybdenum	Mo	98	^{98}Mo = (98)*1 – (99)*0.146 [See note 10]
	Nickel	Ni	60, 61	
	Potassium	K	39	
	Selenium	Se	82	^{82}Se = (82)*1 – (83)*1.009 [See note 11]
	Silver	Ag	107, 109	
	Sodium	Na	23	
	Strontium	Sr	88	
	Thallium	Tl	203, 205	
	Thorium	Th	232	
	Tin	Sn	118, 120	
	Tungsten	W	182	
	Uranium	U	238	
	Vanadium	V	51	^{51}V = (51)*1 – (53)*2.95 + (52)*0.333 [See note 12]
	Zinc	Zn	66, 67, 68	
Internal Stan-dards.	Bismuth	Bi	209	
	Germanium	Ge	72	
	Indium	In	115	^{115}In = (115)*1 – (118)*0.016 [See note 6]
	Lithium	Li	6	
	Scandium	Sc	45	
	Terbium	Tb	159	
	Yttrium	Y	89	

TABLE 3 (continued)

ISOTOPES MONITORED AND CORRECTION EQUATIONS USED FOR USEPA METHOD 6020

Notes:

1) Numbers in parentheses, e.g "(51)", indicate measured counts at the indicated mass.
2) Corrects for ArCl interference, taking into account secondary interferences from Se and Kr
3) Corrects for MoO interference, taking into account secondary interference from ^{108}Cd
4) Corrects for Sn interference
5) Corrects for variations in isotopic composition of lead
6) Corrects for Sn interference
7) Corrects for interference from $^{88}Sr^{2+}$
8) Corrects for Cr interference
9) Corrects for Ca interference
10) Corrects for Ru interference
11) Corrects for Kr interference
12) Corrects for ClO, taking into account secondary interference from Cr

TABLE 4

PREPARATION OF CALIBRATION AND QUALITY CONTROL STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
Calibration Standard 1 (S1) (1.0% HNO_3 / 0.5% HCl)	MS-MIX-Z	Lab Prepared	0.010
	Instrument Calibration Sol'n 1	Spex Industries	0.0025
	Instrument Calibration Sol'n 2	Spex Industries	0.005
Calibration Standard 2 (S2) (1.0% HNO_3 / 0.5% HCl)	MS-MIX-Z	Lab Prepared	0.10
	Instrument Calibration Sol'n 1	Spex Industries	0.025
	Instrument Calibration So'ln 2	Spex Industries	0.050
Calibration Standard 3 (S3) / CCV (1.0% HNO_3 / 0.5% HCl)	MS-MIX-Z	Lab Prepared	0.50
	Instrument Calibration Sol'n 1	Spex Industries	0.125
	Instrument Calibration Sol'n 2	Spex Industries	0.25
Calibration Standard 4 (S4) / High Standard (1.0% HNO_3 / 0.5% HCl)	MS-MIX-Z	Lab Prepared	1.0
	Instrument Calibration Sol'n 1	Spex Industries	0.25
	Instrument Calibration Sol'n 2	Spex Industries	0.50
Initial Calibration Verification (ICV) (1.0% HNO_3 / 0.5% HCl)	CL-ICS-1,CL-ICS-4, CL-ICS-5	Spex Industries	0.20 of each
	CL-ICS-3	Spex Industries	2.0
	1000 mg/L Si	Inorganic Ventures	0.040
	1000 mg/L Al	Inorganic Ventures	0.038
	1000 mg/L B, W	Inorganic Ventures	0.002 of each

TABLE 4 (continued)

PREPARATION OF CALIBRATION AND QUALITY CONTROL STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
Practical Quantitation Limit Solution (PQL) (1.0% HNO_3 / 0.5% HCl)	ICP-MS PQL Intermediate 1	Lab Prepared	1.0
	ICP-MS PQL Intermediate 2	Lab Prepared	0.1
Interference Check Solution A (ICS-A) (1.0% HNO_3 / 0.5% HCl)	6020ICS-0A	Inorganic Ventures	10.0
Interference Check Solution AB (ICS-AB) (1.0% HNO_3 / 0.5% HCl)	6020ICS-0A	Inorganic Ventures	10.0
	Instrument Calibration Sol'n 1	Spex Industries	0.1
	ICP-MS ICSAB Intermediate	Lab Prepared	1.0
P/A Tuning Solution (1.0% HNO_3 / 0.5% HCl)	CL-CAL-1	Spex Industries	1.0 of each
Instrument Tuning Solution (1.0% HNO_3 / 0.5% HCl)	ICP-MS-TS-2	High Purity Standards	0.10
Internal Standard Solution (5.0% HNO_3 / 0.5% HCl)	Internal Standard Mix	Spex Industries	10
Method Tuning Solution (1.0% HNO_3 / 0.5% HCl)	ICP-MS Method Tune Intermediate	Lab Prepared	1.0

TABLE 5

PREPARATION OF INTERMEDIATE STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
ICP-MS PQL Intermediate 1 (1.0% HNO_3 / 0.5% HCl)	1000 mg/L As, Se	High Purity Standards	0.01 of each
	1000 mg/L Zn 10,000 mg/L Ca, Fe, Mg	High Purity Standards	0.02 of each
	10,000 mg/L Si	High Purity Standards	0.04
	10,000 mg/L Al	High Purity Standards	0.06
	1000 mg/L B	High Purity Standards	0.08
	10,000 mg/L K, Na	High Purity Standards	0.20 of each
	CL-ICS-4	Spex Industries	2.0
ICP-MS PQL Intermediate 2 (1.0% HNO_3 / 0.5% HCl)	1000 mg/L Sb, Ba, Be, Cd, Co, Cu, Pb, Mn, Ni, Ag, Sr, Sn	High Purity Standards or Inorganic Ventures	0.02 of each
	1000 mg/L Tl	High Purity Standards	0.04
	1000 mg/L Cr	High Purity Standards	0.06
	1000 mg/L Mo	High Purity Standards	0.08
	1000 mg/L V, W	High Purity Standards or Inorganic Ventures	0.1 of each
ICP-MS Method Tune Intermediate (1.0% HNO_3 / 0.5% HCl)	1000 mg/L Be, Co, In, Pb, Li, Tl 10,000 mg/L Mg	High Purity Standards or Inorganic Ventures	1.0 of each
MS-MIX-Z (1.0% HNO_3 / 0.5% HCl)	10,000 mg/L Ca, Fe, Mg, K, Na	High Purity Standards or Inorganic Ventures	5.0 of each
	10,000 mg/L Si	High Purity Standards	1.0
	10,000 mg/L Al	High Purity Standards	0.95
	1000 mg/L B, Sr, Sn, Ti, W	High Purity Standards	0.50 of each
ICP-MS ICSAB Intermediate (1.0% HNO_3 / 0.5% HCl)	10,000 mg/L Si	High Purity	0.50
	1,000 mg/L B, Sn, Sr, W	High Purity or Inorganic Ventures	0.20 each

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 6

ELEMENT CONCENTRATIONS IN WORKING STANDARDS

Element	CONCENTRATION IN SOLUTION, ug/L						
	S1	S2	S3/CCV	S4	ICV	PQL	P/A Tune Soln.
Aluminum	10.0	100.0	500.0	1000.0	400.0	60.0	200
Antimony	0.5	5.0	25.0	50.0	20.0	0.2	200
Arsenic	0.5	5.0	25.0	50.0	20.0	1.0	200
Barium	0.5	5.0	25.0	50.0	20.0	0.2	200
Beryllium	0.5	5.0	25.0	50.0	20.0	0.2	200
Boron	0.5	5.0	25.0	50.0	20.0	8.0	
Cadmium	0.5	5.0	25.0	50.0	20.0	0.2	200
Calcium	100.0	1000.0	5000.0	10000.0	4000.0	20.0	
Chromium	0.5	5.0	25.0	50.0	20.0	0.6	200
Cobalt	0.5	5.0	25.0	50.0	20.0	0.2	200
Copper	0.5	5.0	25.0	50.0	20.0	0.2	200
Iron	100.0	1000.0	5000.0	10000.0	4000.0	20.0	
Lead	0.5	5.0	25.0	50.0	20.0	0.2	200
Magnesium	100.0	1000.0	5000.0	10000.0	4000.0	20.0	
Manganese	0.5	5.0	25.0	50.0	20.0	0.2	200
Molybdenum	0.5	5.0	25.0	50.0	40.0	1.0	200
Nickel	0.5	5.0	25.0	50.0	20.0	0.2	200
Potassium	100.0	1000.0	5000.0	10000.0	4000.0	20.0	
Selenium	0.5	5.0	25.0	50.0	20.0	1.0	200
Silicon	10.0	100.0	500.0	1000.0	400.0	40.0	
Silver	0.5	5.0	25.0	50.0	20.0	0.2	200
Sodium	100.0	1000.0	5000.0	10000.0	4000.0	20.0	
Strontium	0.5	5.0	25.0	50.0	20.0	0.2	
Thallium	0.5	5.0	25.0	50.0	20.0	0.4	200
Thorium	0.5	5.0	25.0	50.0	20.0	0.2	200
Tin	0.5	5.0	25.0	50.0	20.0	0.2	
Tungsten	0.5	5.0	25.0	50.0	20.0	1.0	
Uranium	0.5	5.0	25.0	50.0	20.0	0.2	200
Vanadium	0.5	5.0	25.0	50.0	20.0	1.0	200
Zinc	0.5	5.0	25.0	50.0	20.0	2.0	200

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 6 (continued)

ELEMENT CONCENTRATIONS IN WORKING STANDARDS

Element	CONCENTRATION IN SOLUTION, ug/L			
	ICSA[1]	ICSAB[1]	Internal Std Solution	Instrument Tuning Solution
Aluminum	100000	100000		
Antimony		20		
Arsenic		20		
Barium		20		
Beryllium		20		
Boron		20		
Cadmium		20		
Calcium	100000	100000		
Chromium		20		
Cobalt		20		
Copper		20		
Iron	100000	100000		
Lead		20		
Magnesium	100000	100000		
Manganese		20		
Molybdenum	2000	2000		
Nickel		20		
Potassium	100000	100000		
Selenium		20		
Silver		20		
Sodium	100000	100000		
Strontium		20		
Thallium		20		10.0
Tin		20		
Tungsten		20		
Uranium		20		
Vanadium		20		
Zinc		20		
Bismuth			1000.0	
Germanium			1000.0	
Lithium (^{6}Li)			1000.0	
Scandium			1000.0	
Terbium			1000.0	
Yttrium			1000.0	10.0
Cerium				10.0
Lithium(^{7}Li)				10.0

1) Solution also contains 1000 mg/L Chloride, 200 mg/L Carbon, and 100 mg/L Phosphorus and Sulfur, and 2mg/L Titanium.

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 7

ELEMENT CONCENTRATIONS IN INTERMEDIATE STANDARDS

ELEMENT	CONCENTRATION IN SOLUTION, mg/L				
	MS-MIX-Z	ICP-MS PQL Intermediate 1	ICP-MS PQL Intermediate 2	ICP-MS Method Tune Intermediate	ICP-MS ICSAB Intermediate
Aluminum	95.0	6.0			
Antimony			0.20		2
Arsenic		0.10			
Barium			0.20		2
Beryllium			0.20	10.0	2
Boron	5.0	0.8			2
Cadmium			0.20		
Calcium	500	2.0			
Chromium			0.60		
Cobalt			0.20	10.0	
Copper			0.20		
Indium				10.0	
Iron	500	2.0			
Lead			0.20	10.0	2
Lithium				10.0	
Magnesium	500	2.0		10.0	
Manganese			0.20		
Molybdenum		0.02	0.80		2
Nickel			0.20		
Potassium	500	20.0			
Selenium		0.10			2
Silicon	100	4.0			50
Silver			0.20		
Sodium	500	20.0			
Strontium	5.0		0.20		2
Thallium			0.40	10.0	2
Thorium		0.020			
Tin	5.0		0.20		2
Tungsten	5.0		1.0		2
Uranium		0.020			2
Vanadium			1.0		2
Zinc		0.20			

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 8

ELEMENT CONCENTRATIONS IN STOCK STANDARDS

Element	CONCENTRATION IN SOLUTION, mg/L					
	Instrument Calibration Standard 1 (Spex)	Instrument Calibration Standard 2 (Spex)	CL-ICS-1 (Spex)	CL-ICS-3 (Spex)	CL-ICS-4 (Spex)	CL-ICS-5 (Spex)
Aluminum	20.0		10.0			
Antimony	20.0		10.0			
Arsenic	20.0		10.0			
Barium	20.0		10.0			
Beryllium	20.0		10.0			
Boron						
Cadmium	20.0		10.0			
Calcium		1000		200.0		
Chromium	20.0		10.0			
Cobalt	20.0		10.0			
Copper	20.0		10.0			
Iron		1000		200.0		
Lead	20.0		10.0			
Magnesium		1000		200.0		
Manganese	20.0		10.0			
Molybdenum	20.0				10.0	10.0
Nickel	20.0		10.0			
Potassium		1000		200.0		
Selenium	20.0		10.0			
Silver	20.0		10.0			
Sodium		1000		200.0		
Strontium						10.0
Thallium	20.0		10.0			
Thorium	20.0				10.0	
Tin						10.0
Tungsten						
Uranium	20.0				10.0	
Vanadium	20.0		10.0			
Zinc	20.0		10.0			

TABLE 8 (continued)

ELEMENT CONCENTRATIONS IN STOCK STANDARDS

Element	CONCENTRATION IN SOLUTION, ug/L		
	6020ICS-0A[1] (Inorganic Ventures)		**ICP-MS-TS-2 (High Purity)**
Aluminum	1000		
Arsenic			
Cadmium			
Calcium	1000		
Chromium			
Cobalt			
Copper			
Iron	1000		
Magnesium	1000		
Manganese			
Molybdenum	20.0		
Nickel			
Potassium	1000		
Silver			
Sodium	1000		
Zinc			
Cerium			10000
Lithium			10000
Thallium			10000
Yttrium			10000

1) Solution also contains 10000 mg/L Chloride, 2000 mg/L Carbon, and 1000 mg/L Phosphorus and Sulfur, and 20 mg/L Titanium.

TABLE 9

REQUIRED ANALYTICAL SEQUENCE

Sequence Number	Standard/Sample	Purpose
1	Method Tuning Solution	Verify mass calibration and resolution
2	S0 (Calibration Blank)	Initial calibration
3	S1 (Calibration Standard)	Initial calibration
4	S2 (Calibration Standard)	Initial calibration
5	S3 (Calibration Standard)	Initial calibration
6	S4 (Calibration Standard)	Initial calibration
7	ICV (Initial Calibration Verification)	Check calibration accuracy
8	ICB (Initial Calibration Blank)	Check calibration accuracy
9	PQL (Practical Quantitation Limit)	Check calibration accuracy at low concentration
10	ICS-A (Interference Check Solution A)	Verify accuracy of mass correction equations
11	ICS-AB (Interference Check Solution AB)	Verify accuracy of mass correction equations
12	Rinse Blank	Clear system of high levels of interferents
13	CCV (Continuing Calibration Verification)	Check calibration stability
14	CCB (Continuing Calibration Blank)	Check calibration stability
15-24	Analyze up to 10 samples	
25	CCV (Continuing Calibration Verification)	Check calibration stability
26	CCB (Continuing Calibration Blank)	Check calibration stability
...	Continue analyzing sequences of up to 10 samples, followed by a CCV and a CCB	
...	After last analytical sample, analyze PQL , followed by a CCV and a CCB	

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 10

INSTRUMENT OPERATING CONDITIONS

Data Acquisition Program		Acquisition Mode	Spectrum
		Points per Mass	3
		Number of Replicates	3
		Detector Mode	Auto for all elements
		Integration Time per Point (for listed masses and their correction masses)	0.10 sec for Li, B, ^{29}Si, Sc, V, Cr, Mn, Ni, Cu, Zn, Y, Mo, Ag, In, Sn, Sb, Ba, Tb, W, Tl, Pb, Bi, Th, U
			0.30 sec for Be, As, Cd, Ge
			0.010 sec for Na, Al, K, ^{28}Si
			0.030 for Ca, Fe, Sr
			1.00 sec for Se
			0.050 sec for Mg, Co
		Spray Chamber Temperature	2° C
		Total Acquisition Time	113 sec for 3 replicates
Peristaltic Pump Program		Analysis Speed	0.12 rps
ISIS Pump Program	Before Acquisition	Uptake Speed	1.00 rps
		Uptake Time	25 sec
		Stabilization Time	120 sec
	After Acquisition (Probe Rinse)	Rinse Speed	1.00 rps
		Rinse Time (sample)	10 sec
		Rinse Time (standard)	10 sec
	After Acquisition (Rinse)	Rinse Vial	1
		Uptake Speed	1
		Uptake Time	25 sec
		Stabilization Time	150 sec
Calibration Curvefit		All quantitation masses	Y=ax+(blank)
		All internal standard masses	(Excluded)
		All interference correction masses	(Excluded)
Reporting Parameters		QC Reports	On-Printer
		All Other Reports	Off

TABLE 11

INSTRUMENT TUNE SPECIFICATIONS

Sensitivity	Li >5000 cts/0.1 sec/10 ppb
	Y >10,000 cts/0.1 sec/10 ppb
	Tl >5000 cts/0.1 sec/10 ppb
Precision	Li <8% RSD (0.1 sec integration time)
	Y <5% RSD (0.1 sec integration time)
	Tl <5% RSD (0.1 sec integration time)
Oxides	<1.0%
Doubly Charged (Ce^{++}/Ce^{+})	<2.0%
Background	Li <15 cps
	Y <15 cps
	Tl <15 cps
Mass Resolution	Width at 10% peak height: 0.7-0.8 amu
Mass Axis	Li ±0.1 amu of nominal mass
	Y ±0.1 amu of nominal mass
	Tl ±0.1 amu of nominal mass

TABLE 12

METHOD TUNE SPECIFICATIONS

Precision	≤5% RSD of 4 replicates
Mass Resolution	Width at 10% peak height: <0.9 amu
Mass Calibration	±0.1 amu of nominal mass

TABLE 13

REPORTED ISOTOPES AND INTERNAL STANDARDS

ELEMENT	MASS	INTERNAL STANDARD (mass)
Aluminum	27	Scandium (45)
Antimony	123	Terbium (159)
Arsenic	75	Germanium (72)
Barium	135	Terbium (159)
Beryllium	9	Lithium (6)
Boron	11	Lithium (6)
Cadmium	114	Germanium (72)
Calcium	44	Scandium (45)
Chromium	52	Germanium (72)
Cobalt	59	Germanium (72)
Copper	65	Germanium (72)
Iron	57	Germanium (72)
Lead	208	Bismuth (209)
Magnesium	25	Scandium (45)
Manganese	55	Germanium (72)
Molybdenum	98	Germanium (72)
Nickel	60	Germanium (72)
Potassium	39	Scandium (45)
Selenium	82	Germanium (72)
Silicon	29	Scandium (45)
Silver	107	Germanium (72)
Sodium	23	Scandium (45)
Strontium	88	Germanium (72)
Thallium	203	Bismuth (209)
Thorium	232	Bismuth (209)
Tin	118	Terbium (159)
Tungsten	182	Terbium (159)
Uranium	238	Bismuth (209)
Vanadium	51	Germanium (72)
Zinc	66	Germanium (72)

ATTACHMENT 1

HARDNESS BY CALCULATION

As referenced in "Standard Methods for the Examination if Water and Wastewater," Methods 2340 A & B, Hardness Introduction and Hardness by Calculation, American Public Health Association, 18th Edition, Revised 1992, total hardness is the sum of the calcium and magnesium concentrations, both expressed as calcium carbonate, in milligrams per liter.

Once the calcium and magnesium concentrations have been determined by EPA methods 6010, 6020, 200.7 or 200.8, the total hardness of an aqueous sample may be calculated as follows:

$$\text{Total Hardness, mg equivalent } CaCO_3/L = 2.497\ (Ca, mg/L) + 4.118\ (Mg, mg/L)$$

The calcium hardness of an aqueous sample may also be calculated as follows:

$$\text{Calcium Hardness, mg equivalent } CaCO_3/L = 2.497\ (Ca, mg/L)$$

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: SD-902
Revision History
Cover Page
Page 1

TITLE: **SAMPLE RECEIPT AND INTERNAL CONTROL**

Prepared By: Andrea Colby Date: 6/2002

Approved By:

Group Supervisor: Andrea Colby Date: 6/6/02

Lab Operations Mgr: J. C. Burton Date: 6/5/02

QA Officer: Deborah J. Nadeau Date: 6.6.02

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
04	Changed cover sheet, minor changes to sections 7.1, 7.6 7.7.4, 7.10 + 7.20. Complete rewrite of sections 7.11 + 7.12 to comply with new KIMS	DN	6.6.02	6.6.02
05	Added verbal date entry to KIMS. Added reference to immediate internal COC book. Added Department Manager reference. Added section 7.7.3. Updated new incoming	DN	05.04	05.04
06	Added procedure + logbook page for checking turbidity of drinking water samples. Changed wet chem shorts board to a book (included example page). Added custody procedures for food/micro. Added VOA soil freezer storage.	DN	01.26.04	01.26.04
07	Added instructions to create lettered labels. Changed sample locations to reflect new building. Removed Figures 8 and 10. Updated Table and Figures w/ current ones. Added wording to Sect. 7.7.5 to clarify how pH measurements are taken.	LAD	02/07	02/07
08	Added summary stating sample acceptance policy. Deleted all references to radiation checks (not performed). Add IR gun usage. Reorganized section 7.0 to prioritize time sensitive tasks. Added wireless thermometer monitoring. Updated SRCR. Other minor changes.	DN	05/09	05/09

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SD-902-08**, titled **Sample Receipt and Internal Control**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SD-902-08**, titled **Sample Receipt and Internal Control**.

Recipient: _____ Date: _____

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

1.0 SCOPE AND APPLICATION

Katahdin Analytical Services, Inc. requires the use of specific receiving, acceptance, identification, storage, and distribution procedures for samples it accepts for analyses. These procedures assure that:

- samples are uniquely identified,
- samples are protected from loss or damage,
- essential sample characteristics are preserved,
- any alteration of samples (e.g., filtration, preservation) is documented,
- the correct samples are analyzed, and
- a record of continuous sample custody and utilization is established.

The purpose of this SOP is to describe the procedures used for the receipt and tracking of samples received by Katahdin Analytical Services, Inc. (Katahdin).

1.1 Definitions

SDG: Sample Delivery Group – A group of samples to be reported as one data package.

1.2 Responsibilities

It is the responsibility of all Katahdin staff who receive samples or handle samples in the course of analysis to follow the procedures set forth in this SOP, to document their understanding of the procedures in their training files (refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability"), and to suggest changes and revisions when appropriate. All technical staff are responsible for monitoring their immediate areas, stopping an activity when a problem is detected or suspected, initiating corrective action when needed, documenting any actions taken, and notifying the appropriate individual (e.g., Department Manager, Operations Manager, QAO). The primary responsibility for implementing real-time corrective actions and maintaining an effective QA self-inspection system resides with Katahdin staff. When problems are identified Katahdin personnel are expected to attempt to resolve situations within the scope of their technical knowledge, and to seek assistance from peers and the Department Manager as necessary.

It is the responsibility of Department Managers to oversee the adherence to Katahdin QC practices and internal documentation of laboratory activities within their area, to take corrective actions where needed and communicate problems to the Operations Manager, QAO or Vice President/President when warranted.

It is the responsibility of the Operations Manager to oversee adherence to Katahdin QA/QC practices by all laboratory groups under his/her authority, to help identify

problems and assure resolution, to facilitate corrective action where needed, and to communicate problems and concerns to the QAO and Vice President/President.

It is the responsibility of the Quality Assurance Officer (QAO) to oversee adherence to this SOP, to conduct periodic audits of each laboratory, to track corrective action reports, resolution, and documentation, and to communicate concerns and report findings to the Vice President/President. The QA Officer shall function independently from laboratory operations and be able to evaluate data objectively and perform assessments without outside influence. The QA Officer has the authority to independently halt production operations (including data reporting) if warranted by quality problems.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical safety procedures and the Katahdin Environmental Health & Safety Manual and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Wastes generated during the receipt of samples must be disposed of in accordance with the Katahdin Environmental Health & Safety Manual and SOPs SD-903, "Sample Disposal" and CA-107, "The Management of Hazardous Waste as it Relates to the Disposal of Laboratory Process Waste, Reagents, Solvents and

Standards," current revisions. Expired standards are placed in the Katahdin hazardous waste storage area, and disposed of in accordance with these SOPs.

2.0 SUMMARY OF METHOD

Regulatory, program, and/or method requirements dictate the specifics of sample acceptance. These requirements include, but are not limited to, temperature upon receipt, chemical preservation, container type, sample amount, holding time considerations and complete and accurate documentation of all of these conditions, as well as sample identification. Katahdin's sample acceptance policy is to note any anomalies, discrepancies or non-compliances concerning the receipt of samples. The client is always notified with these issues to direct Katahdin on how and whether to proceed with analysis. All guidance from the client is recorded in the project phone logs and/or on the Sample Receipt Condition Report, which becomes part of the final report. Conditions or analyses performed which do not meet the necessary requirements are narrated or notated as described in the individual analytical SOPs.

3.0 INTERFERENCES

Not applicable.

4.0 APPARATUS AND MATERIALS

4.1 Thermometer – Oakton® Non-Contact Infrared Thermometer, or equivalent, capable of reading 0.1°C and digital probe style capable of reading 0.1°C (used for back-up).

4.2 Capillary tubes – 75 mm Hematocrit Tubes, disposable

4.3 Wide range pH test strips, pH 0 to 14 pH, EMD ColorpHast or equivalent.

4.4 Narrow range pH test strips, pH 0 to 2.5 pH, EMD ColorpHast or equivalent.

4.5 Narrow range pH test strips, pH 11 to 13 pH, EMD ColorpHast or equivalent.

5.0 REAGENTS

Preservatives - refer to Table 1, Sampling and Preservation Requirements, for specifics.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Refer to Table 1, Sampling and Preservation Requirements, for specifics.

7.0 PROCEDURES

PROCEDURES FOR SAMPLE CUSTODIAN

The following procedures include all steps to be completed for satisfactory receipt and acceptance of samples at Katahdin. These steps do not necessarily have to be performed in the exact order as described. Sample deliveries occur constantly throughout the day, so the sample custodian must multi-task and move back and forth between different procedures to accomplish the most critical tasks of checking receipt temperatures and checking for "RUSH" or quick hold time parameters.

7.1 When samples (except for non-environmental food samples) are dropped off, by either a delivery service (i.e. FEDEX or UPS) or by the client, the Chain-of-Custody (COC) should be signed immediately. The client (who is delivering or that has shipped samples with a delivery service) shall sign (at the lab upon delivery or prior to shipment of samples) that they have relinquished custody to the laboratory. The laboratory shall sign and record the date and time that custody is accepted. (Refer to Figures 1-3 for a Katahdin standard COC, a Katahdin Homeowner COC, and a Katahdin Food/Microbiology COC).

7.2 Cut custody seals and open all coolers. Remove the packets containing the client Chains-of-Custody (COCs).

7.3 Using the COCs, enter the date and time of sample receipt and the client name into the next available work order/login number in the sample receipt logbook (Figure 4). Initial each entry (line) to maintain a record of the individual who assigned each group of samples a discreet lab work order/login number. Record the assigned work order numbers in the appropriate space on the client COCs. Complete the log-in entry date and time once samples are logged in as described below.

7.4 Inventory the COCs for any "RUSH" or quick hold time analyses. Notify the appropriate section managers of these analyses. List any samples for analyses that have short hold times in the "Wet Chemistry Shorts and Rushes Logbook" (Figure 5) in the wet chemistry laboratory. Be sure to list the client, number of samples and date and time of the earliest sample. GC or GC/MS personnel must be informed when ENCORES are received so that they may be scheduled for extrusion. Microbiology personnel should also be informed of any microbiology samples that arrive. Parameters that routinely require short analytical hold times are:

Coliforms	Color	pH
Nitrate/Nitrite	Dissolved Oxygen	Turbidity
Ferrous iron	Orthophosphate	Hex. Chromium
MBAS	TBOD	Free CO_2
Sulfite	ENCORE soil samples	Settleable Solids
Odor	Residual Chlorine	CBOD

7.5 Inspect the condition of custody seals, cooler, ice condition and samples received. Note any non-intact conditions on the Sample Receipt Condition Report (SRCR - Figure 6). Notify the Katahdin project manager (PM) of any discrepancies or problems with sample receipt. The PM contacts the client as necessary. If breakage of a potentially hazardous sample is discovered, close and seal the packing container with all the samples inside and move to a hood in the organic extractions area or to the smaller hood in the login area if space permits. One of the three Katahdin Emergency Response Coordinators or the Katahdin Environmental Health & Safety Manager must be notified. Disposition of the broken and other possibly contaminated samples will be determined on a case-by-case basis in accordance with the laboratory's handling procedures for hazardous waste as outlined in the Katahdin Environmental Health & Safety Manual. Generally, when a sample has broken and has mixed with any ice in the cooler, that liquid will be poured off into 2 liter plastic containers and labeled as "do not use". These containers will be disposed of as soon as the disposition of the appropriate samples has been determined through analysis.

7.6 If there is no breakage of a potentially hazardous sample:

Check cooler temperatures using the IR thermometer assigned to the sample receipt area. If a cooler temperature blank is present, aim the IR gun at the temperature blank; otherwise aim the IR gun at any sample in the cooler if no temperature blank is present. Be sure that the IR gun is within 6 inches of the bottle and not aimed at a label on the bottle. Press the trigger on the handle and be sure the red dot is visible on the bottle surface. The IR gun has been set to read in degrees celcius. If checking the temperature of a plastic bottle, set the emissivity at 0.90. If checking the temperature of a glass bottle (either amber or clear), set the emissivity at 0.85. Refer to Figure 7 for manufacturer's instructions on changing the emissivity. Record the temperature on the Sample Receipt Condition Report. Receipt temperatures should be <6 °C, without freezing. Any temperature falling outside of this range must be noted on the SRCR and reported to the appropriate Katahdin project manager.

Note: Samples received for metals analysis only do not have to meet any temperature receipt requirements.

Note: A probe type thermometer is retained as back-up in case there is a problem with the IR thermometer.

7.7 Note the condition of the ice or ice packs. If the ice has melted and the temperature is out of acceptance criteria, note this on the SRCR. For samples that are hand delivered to the laboratory immediately after collection (i.e. sample collection times are <6 hours old), the temperature blank and/or cooler temperature will most likely not meet the acceptance criteria. The samples shall be considered acceptable if there is evidence that the chilling process has begun such as arrival on ice. Note this on the SRCR. If samples (that were just collected) have not arrived on ice, note this on the SRCR, and start the cooling process as soon as possible after arrival at the laboratory.

Note: All clients must be notified when samples are received that do not meet the appropriate temperature requirements. In these cases, certain regulatory requirements may not be met and may invalidate certain data.

7.8 Inventory the samples against the chain of custody (COC). If the COC is incomplete, the sample custodian must inform the appropriate Katahdin project manager (PM). The PM may make changes to correct or complete the COC, but all changes must be initialed and dated. Changes must be noted on the SRCR. Any discrepancies between the samples and the COC must also be noted on the SRCR.

7.9 Using the Sampling and Preservation Requirements Table (Table 1) as a reference, check if samples are in proper containers and received correct pretreatment (e.g., filtration, preservation) for the analyses requested. For aqueous parameters requiring preservation, check pH by inserting a clean capillary tube into the sample and dabbing the tube on wide range pH paper. If the pH is not clearly either less than 2 or greater than 12, the appropriate narrow range pH paper must be used. NOTE: The pH of volatile organic (VOA) samples is checked and recorded by the analyst after completion of analysis and not by sample receipt personnel. The used capillary tube is discarded and a new capillary tube is used for each sample.

Additional preservative is added to samples if the pH is not in the range specified in the Sampling and Preservation Requirements Table. No more than 10% of the original sample volume should be added as preservative. If the client has noted that the sample reacts violently (i.e., foams and bubbles) upon preservation, add no more preservative to the sample. Some clients may wish to be contacted if their samples are found to be improperly preserved. Record all preservation discrepancies on the Sample Receipt Condition Report including the lot number of the preservative added. If additional preservative is added, a sticker with the type of preservative must be placed on the sample container.

Note: Preservatives are obtained from the larger containers in the bottle preparation area.

Note: If samples are received unpreserved for 200.7 or 200.8 analysis, the samples must be preserved to pH <2 with nitric acid. Samples must be held for 16 hours after preservation before sample preparation can begin.

7.10 For samples requiring filtration as pretreatment (i.e. for dissolved metals), the work order/login numbers are recorded in the filtration logbook (see Figure 8). The samples are filtered by the Metals Group.

7.10.1 A 500 mL filter flask and filter funnel are acid rinsed three times in a 10% nitric acid bath, then three times with Laboratory Reagent Grade Water.

7.10.2 A vacuum pump is attached.

7.10.3 A 0.45 micron filter is rinsed three times with 5% nitric acid and three times with Laboratory Reagent Grade Water. The rinsate is discarded.

7.10.4 A sufficient sample aliquot is filtered and preserved with concentrated nitric acid to pH <2.

7.10.5 The bottles are labeled with the work order/login number and other sample information and stored at <6 ° C until the time of digestion.

7.11 Using the Sampling and Preservation Requirements Table (Table 1) as a reference, determine if sufficient volume of sample is present for analysis. Note discrepancies on the SRCR.

7.12 For drinking water samples, enter the appropriate information (work order, date, etc.) into the Measured Turbidity and Preservation of Incoming Samples Logbook. Inform the appropriate analyst of the sample. The turbidity must be measured prior to sample preparation. If the turbidity is <1 NTU, the sample does not have to be digested prior to metals analysis. If the turbidity is >1 NTU, the sample must be digested prior to metals analysis. The sample must be preserved after the turbidity measurement is taken. Record the appropriate information in the logbook (Figure 9).

7.13 Notify the PM immediately if there are any discrepancies or problems with sample receipt. The PM will contact the client for information and resolution as necessary.

7.14 Review any additional paperwork that accompanies the sample(s) submitted for analysis along with laboratory-generated information. This includes shipping forms, letters, chain-of-custody forms, sample labels, Incoming Sample Information Sheets (ISIS), quotes, memos, etc. These forms may provide details on specific client requests. The ISIS will provide information on specifics for log-in. Refer to Figure 10 for an example.

7.15 Resolve any questions or concerns raised by steps 7.1-7.14 by consulting the correspondence files or client services personnel or communicating directly with the

client. Note in the notes section of the SRCR any deviations from normal sample handling or analytical procedures (e.g., client requests analysis although hold-time expired).

7.16 When non-environmental food samples are delivered to the laboratory, they are taken immediately to the food/microbiology laboratory and stored in the refrigerators there. A copy of the Chain-of-Custody is left with the analysts. The original paperwork is forwarded to sample log in where the job is logged into the KIMS system.

7.17 The following information is documented via the Katahdin Information Management System (KIMS) and a work order/login COC report (Figure 11) is generated for the samples received:

7.17.1 Log onto KIMS by entering employee ID under "Username", employee specific password under "Password" and KIMS under "Database".

7.17.2 Once logged onto KIMS select "Sample Management" and then "Login".

7.17.3 Select "New" and the next available Login ID number will automatically be entered. Select "OK" and the Sample Definition screen will open.

Note: If a Work Order number has already been opened, select "change" and type in the appropriate number to access the information.

7.17.4 In the Sample Definition Screen, enter the following information.

Client ID -	Enter client sample description.
ReceiveDate -	Enter in date that samples were received in the lab in the format YY-Month-DD.
CollectDate -	Enter in date that samples were collected in the format YY-Month-DDTIME.
TAT -	Enter TAT for hardcopy report.
DueDate -	Due date will automatically be calculated based on calendar days.
VerbalDate -	Manually type in verbal due date.
QuoteRef -	Enter quote number if applicable.
Project -	Enter project number if applicable.
Account -	Enter client specific account number.

Account Name -	Account name will automatically be entered.
Collected By -	Enter name/initials of sampler listed on COC. If unknown, enter "Client".
Locator -	May be used for client ID information when requested by the project manager.
Site -	Enter project site name.
Description -	May be used for long client Ids when requested by the project manager.
Discount -	No entry-not currently used.
Priority -	No entry-not currently used.
Fact. -	No entry-not currently used.
Expected -	No entry-not currently used.
Comments -	Enter MS/MSD, verbal due date and any sample irregularities if applicable.
OrderDate -	Current date is automatically entered.
Matrix -	Enter sample matrix code where AQ = Aqueous SL = Solid, Soil, Sludge FP = Free Product WP = Wipe NOAQ = NonAqueous DW = Drinking Water SLD = Food Solid AR = Air SWAB = Swab SAL = Saline TIS = Tissue
Product Code -	Enter analysis code per test requested on COC.
Type -	Product code type will automatically be entered where S = Stand alone P = Parent C = Children
Fact. -	No entry-default is 1.

Price - This is left as is by sample log-in. During project management review of the work order, the prices are entered based on quotes or standard prices.

Cost - No entry needed.

Lev - No entry needed.

Type - Container type will automatically be entered.

Bot - Enter number of containers per test for printing of labels.

Login Info - Parameter Data Screen will open. Enter following information

KAS Proj. Manager-	Initials of Katahdin person overseeing the project.
Client PO#-	Client purchase order.
Project-	Project name.
Cooler Temperature-	Temperature blanks or cooler temps.
Delivery Services-	Method of delivery to the lab.
QC Level-	QC Level of report and regulatory agency (ie., IV NFESC).
SDG ID-	Sample Delivery Group ID if applicable.
SDG Status-	Begin, Continue or End.
Analysis Instructions-	PM will enter special instructions regarding project.
Report Instructions-	PM will enter special instructions regarding project.
Regulatory List-	Used for federal programs.
EDD Format-	Specific KAS EDD format.

Select "SAVE" and then "CANCEL".

Addresses - Select "Addresses" and the Address Links screen will open. The billing address is the default address of the account. Enter the client account code under "Project/Account" and select the report to contact under "Address Type". Select the appropriate boxes for report, report CC and invoice CC. Select "SAVE" and then "CLOSE".

Create Containers - Select "Create Containers". Letters will be assigned to each sample number. Select "OK" until letters have been assigned to each sample number. To manually assign letters, Select "Enter Container IDs" and "OK". Enter sample numbers including letters and select "OK", "Close", "Yes" to save changes, "Cancel" and "Cancel".

7.17.5 To print the login report, select "Reports", "Login" and "Login COC". Enter login number under "Login Number". Select "OK", "Run Report" and then "Print".

7.18 To print labels unique to each bottle, select "Reports", "Login" and "Labels". Enter login number under "Login/Prelogin", select "Background (IDXL)" and select F9 on keyboard under "Select Sample Label". Select "OK" and then "Print". After labels print out select "Cancel".

7.19 Affix permanent sample number labels to sample containers, assuring that sample IDs on labels correspond to sample bottle IDs. Do not obscure client ID on the bottles.

7.20 Place samples in their designated storage locations and log them in, noting initials, date and time, work order/login and sample numbers, and storage location on the internal laboratory chain of custody form (Figure 12). Place form in the appropriate binder in the log in area. Non-environmental food samples do not get an internal COC and are taken immediately to the food/microbiology lab for storage.

Storage location of the samples is determined by type of sample and/or type of analysis, as outlined below. Most samples are stored in the walk-in cooler, which is organized by test type and work order/login number.

Specific storage locations are described below.

7.20.1 Aqueous samples for wet chemistry (except hardness, see 7.19.4 below) - left aisle, both sides, as you enter walk-in cooler. TOC vials are to be stored in the trays designated for TOC samples.

7.20.2 Aqueous samples for organic extractions – right aisle, left side, as you enter walk-in cooler.

7.20.3 Non-aqueous samples (all analyses except volatile organics) - to the right and towards the back as you enter walk-in cooler. Non-aqueous samples for volatile organics are stored in "VOA Refrigerator 2" located in the Volatiles Laboratory.

7.20.4 Aqueous samples for metals and/or hardness analyses – right aisle, right side towards the front as you enter walk-in cooler.

7.20.5 Samples (aqueous and solid) for volatile organics analyses (VOA) – All aqueous samples and soil samples in VOA vials (preserved with methanol or sodium bisulfate) are stored in "VOA Refrigerator 1" in the Volatiles Laboratory. VOA soils in jars or ENCORE samplers are stored in "VOA Refrigerator 2" in the Volatiles Laboratory. VOA samples known or suspected to be hazardous (such that cross-contamination of other samples might occur) are placed in a "paint can" and stored in the walk-in.

7.20.6 Soil samples for volatile organics analyses (VOA) that are unpreserved or preserved with Laboratory Reagent Grade Water are stored in "VOA Freezer 1" in the volatiles laboratory.

7.21 Sample Receipt gives the Work order/login COC report and confirmation of the job, as logged-in, to the appropriate Katahdin project manager. All chain-of-custody and other receipt documentation must accompany the job. The project manager reviews the job for accuracy and completeness. Any unresolved issues should be resolved at this time. Any project or program specific forms should be included with the paperwork at this time. These forms may include CLP forms or state-specific forms. The project manager then dispatches the work order/login to the individual department worklists. The dispatched work order/login package is then filed in Data Management where the complete package will eventually be compiled.

7.22 The temperature of all sample storage refrigerators and freezers is recorded daily by assigned individuals. Notebooks containing a record of each refrigerator and freezer temperature history are used for this purpose and are maintained by the assigned individuals. Temperatures above or below the acceptance range are to be brought to the attention of a Department Manager, Operations Manager, or Quality Assurance Officer. Such an occurrence and the actions taken to correct it must be noted in the comments column of the temperature recording notebook next to the temperature measurement. (See Figure 13).

Additionally, temperatures of storage units are monitored continuously by wireless thermometers. A temperature is recorded electronically every 10 minutes. The QAO can generate a specified report as needed, including every reading or maximum/minimum temperatures for a given timeframe. These monitoring devices ensure continual compliance seven days per week. The data can be used to check for problems.

PROCEDURES FOR CHEMISTS

7.23 When removing a sample from its storage location, record on the laboratory internal chain-of-custody (from the appropriate department) the sample number, date and

time it was removed, chemist who removed it, and the analysis to be conducted or reason for removal.

7.24 If the samples have not been logged in yet and they need to be pulled in order to analyze short holding time parameters, the analyst taking the sample must use the designated logbook (Immediate Internal COC – Figure 14) to sign the samples out. Many circumstances lead to analysts having to pull samples before they are logged into the KIMS system. It is everyone's responsibility to ensure that all samples can be accounted for at all times. Failure to do so can create confusion and bottle necks for others trying to access the samples. Samples that are pulled before log-in must be returned to the designated bin in the sample receipt area.

7.25 If a sample is not consumed by an analysis, return the remaining sample to its assigned storage location and enter the date and time returned on the laboratory internal chain-of-custody record.

7.26 If analysis consumes the entire sample, indicate this on the laboratory internal chain-of-custody record.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Each thermometer used to monitor sample storage or cooler temperatures must be calibrated annually against a NIST traceable thermometer. The QAO is responsible for ensuring that the thermometer(s) are scheduled for annual calibration and for maintaining the calibration records. All other procedures and documentation listed in this SOP must be followed at all times.

9.0 METHOD PERFORMANCE

Not applicable.

10.0 APPLICABLE DOCUMENTS/REFERENCES

"Handbook for Analytical Quality Control in Water and Wastewater Laboratories," U.S. EPA EMSL Office of Research and Development, March 1979.

Code of Federal Regulations 40, Parts 136 and 141.

"Test Methods for Evaluating Solid Waste: Physical/Chemical Methods," SW-846 Chapters 1 & 2, USEPA, Third Edition, including Updates I, II, IIA, and IIB, III June, 1997.

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

Katahdin Analytical Services, Inc., Environmental Health & Safety Manual, current revision.

LIST OF TABLES & FIGURES

Table 1 Sampling and Preservation Requirements
Figure 1 Example of Standard Katahdin Chain-of-Custody Form
Figure 2 Example of Homeowner Katahdin Chain-of-Custody Form
Figure 3 Example of Food/Microbiology Katahdin Chain-of-Custody Form
Figure 4 Example of Sample Receiving Logbook
Figure 5 Example of Wet Chemistry Shorts and Rushes Logbook
Figure 6 Example of Katahdin Sample Receipt Condition Report
Figure 7 IR Thermometer Manufacturer's Instructions for Changing Emissivity
Figure 8 Example of Sample Filtration Logbook
Figure 9 Measured Turbidity and Preservation of Incoming Samples Logbook
Figure 10 Example of Laboratory Incoming Sample Information Sheet (ISIS)
Figure 11 Example Katahdin Work order/login COC Report
Figure 12 Example of Katahdin Internal Chain-of-Custody Form
Figure 13 Example of Refrigerator Temperature Logbook
Figure 14 Example of Immediate Internal COC Logbook

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

TABLE 1

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – AQUEOUS MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
GENERAL CHEMICAL ANALYSES					
Acidity	305.1	100 mL	P,G	1,2	14 days
Alkalinity-Manual Titrimetric	310.1	100 mL	P,G	1,2	14 days
Ammonia-Nitrogen with distill-Auto. Phenate	350.1	1 L	P,G	1,3	28 days
Ammonia-Nitrogen-Automated Phenate	350.1, 350.2	250 mL	P,G	1,3	28 days
Anions (Cl, Br, SO4, NO2, NO3)	300.0	250 mL	P, G	1	48hr/28days
Bicarbonate, Carbonate (see pH & alkalinity)	calc.				
Biochemical Oxygen Demand-Carbonaceous	405.1	1 L	P,G	1	48 hours
Biochemical Oxygen Demand-Total	405.1	1 L	P,G	1	48 hours
Bromide	320.1	500 mL	P,G	1	28 days
Chemical Oxygen Demand-Manual Colorimetric	410.4	100 mL	P,G	1,3	28 days
Chloride-Automated Ferricyanide	325.2	100 mL	P,G	1	28 days
Chlorine, Residual	SM4500-Cl G	100 mL	P,G	1,9	ASAP
Chromium, Hexavalent	SM3500Cr D / SW7196	200 mL	P,G	1,9	24 hours
Color, Apparent	110.2	100 mL	P,G	1,2	48 hours
Cyanide, Amenable-Spectrophotometric	335.1	250 mL	P,G	1,5	14 days
Cyanide, Total-Spectrophotometric	SM4500CN C 335.3, 335.4	250 mL	P,G	1,5	14 days
Dissolved Oxygen(Lab)-Membrane Electrode	360.1	500 mL	G	1	ASAP
Ferrous Iron - Colorimetric	SM3500-Fe D	250mL	P	1	24 hrs
Fluoride with distillation, Potentiometric ISE	SM4500F C/340.2	500 mL	P only	1	28 days
Fluoride, Potentiometric ISE	340.2	200 mL	P only	1	28 days
Free CO_2	SM4500-CO_2 C	250mL	P	1	24 hrs.
Hardness, Total-Manual Titrimetric	130.2,SM2340C	250 mL	P,G	4	6 months
MBAS, Extraction-Colorimetric	SM5540C	1 L	P,G	1	48 hours
Nitrate+Nitrite-Automated Cadmium Reduction	353.2	100 mL	P,G	1,3	28 days
Nitrate-Automated Cadmium Red./Diazotization	353.2	100 mL	P,G	1	48 hours
Nitrite-Automated Diazotization	353.2	100 mL	P,G	1	48 hours
Oil & Grease-Total Recoverable, Gravimetric N-Hexane extractable material N-Hexane extractable material w/ silica gel cleanup	1664	(2) 1 L	glass only	1,11	28 days
pH (Laboratory)	150.1	100 mL	P,G	1,2	24 hours
Phenolics, Total Recoverable-Manual 4AAP	420.1	1000 mL	glass only	1,3	28 days
Phosphate, Ortho- Ascorbic Acid	365.2	100 mL	P,G	1	48 hours
Phosphate,Total	365.4	100 mL	P,G	1,3	28 days
Solids-Filterable Residue (TDS),Gravimetric180	160.1	250 mL	P,G	1	7 days
Solids-Nonfilterable Residue (TSS)	160.2	500 mL	P,G	1	7 days
Solids-Settleable Solids (SS)	160.5	1 L	P,G	1	48 hours

TABLE 1 (cont.)

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – AQUEOUS MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
GENERAL CHEMICAL ANALYSES					
Solids-Total Solids	160.3	250 mL	P,G	1	7 days
Solids-Total Volatile (TVS)	160.4	250mL	P,G	1	7 days
Solids-Volatile Filterable Residue (VDS)	160.1/160.4	250 mL	P,G	1	7 days
Solids-Volatile Nonfilterable Residue (VSS)	SM 2540 F	500 mL	P,G	1	7 days
Specific Conductance-Wheatstone Bridge	120.1	100 mL	P,G	1,2	28 days
Sulfate-Turbidimetric	375.4	100 mL	P,G	1	28 days
Sulfide-Iodometric	376.1	500 mL	P,G	1,7	7 days
Sulfite-Titrimetric	377.1	500 mL	P,G	1,9	ASAP
Tannin/Lignin-Colorimetric	SM 5550 B	100 mL	P,G	1	7 days
TKN-Auto Block Digest, Spect.	351.2	100 mL	P,G	1,3	28 days
Total Inorganic Carbon	415.1	(2) 40 mL	VOA vial	1	28 days
Total Inorganic Carbon if with TOC	415.1	(2) 40 mL	VOA vial	1	28 days
Total Organic Carbon-Oxidation	415.1	(2) 40 mL	VOA vial	1,3	28 days
Total Organic Halogen	9020	500 mL	Amber Glass	1,3	28 days
Turbidity	180.1	100 mL	P,G	1	48 hours
ELEMENTAL ANALYSES					
Chromium, Hexavalent	7196/6010	500 mL	P,G	1,9	24 hrs
GFAA(Furnace) Elements	SM 3113/ 200 series	500 mL	P,G	4	6 months
ICP Elements	200.7/6010	500 mL	P,G	4	6 months
ICP MS Elements	200.8/6020	500 mL	P,G	4	6 months
Low Level Mercury	1631	500 mL	G	NA	90 days
Mercury	245.1/7470	500 mL	P,G	4	28 days
GC ORGANIC ANALYSES					
BTEX & MTBE	602 & 8021	(2) 40 mL	VOA vial	1,8,9	14 days(~)
EDB, DBCP & 1,2,3-TCP	504.1	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Extractable Petroleum Hydrocarbons	MADEP/EPH	(2) 1000 mL	Amber Glass	12	14days/40days
Formaldehyde	556	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Fuel Oil in Water	8015Modified	(2) 1000 mL	Amber Glass	1,8	7days/40days
Fuel Oil in Water	ME HETL 4.1.25	(2) 1000 mL	Amber Glass	1,8	7days/40days
Gasoline in Water	8015Modified	(2) 40 mL	VOA vial	1,8	14 days
Gasoline in Water	ME HETL 4.2.17	(2) 40 mL	VOA vial	1,8	14 days
Glycols	8015Modified	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Herbicides	8151	(2) 1000 mL	Amber Glass	1	7days/ 40days
Methane, Ethane & ethene	RSK 175	(2) 40 mL	VOA vial	1,8,9	14 days(~)
PCB's (& Congeners)	608 & 8082	(2) 1000 mL	Amber Glass	1	7days/40days

TABLE 1 (cont.)

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – AQUEOUS MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
GC ORGANIC ANALYSES					
Pesticides	608 & 8081	(2) 1000 mL	Amber Glass	1	7days/40days
Pesticides and PCB's	608 & 8081/8082	(2) 1000 mL	Amber Glass	1	7days/40days
Purgeable Aromatics	602 & 8021	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Purgeable Halocarbons	601 & 8021	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Purgeables, Total	601 & 602	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Purgeables, Total	8021	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Solvents (Direct Injection)	8015M	(2) 40 mL	VOA vial	1	14 days
Volatile Petroleum Hydrocarbons	MADEP/VPH	(2) 40 mL	VOA vial	11	14days
GC/MS ORGANIC ANALYSES					
Acid Extractables-Priority Pollutants	625	(2) 1000 mL	Amber Glass	1	7days/40days
Acid Extractables-TCL	8270	(2) 1000 mL	Amber Glass	1	7days/40days
Base Neutral Extract.-Priority Pollutants	625	(2) 1000 mL	Amber Glass	1	7days/40days
Base Neutral Extractables-TCL	8270	(2) 1000 mL	Amber Glass	1	7days/40days
Drinking Water Volatiles - Low Level	524.2	(3) 40 mL	VOA vial	1,8,9,10	14 days(~)
PCB Homologues	680	(2) 1000 mL	Amber Glass	1	7days/40days
Polyaromatic Hydrocarbons	8270/8270 SIM	(2) 1000 mL	Amber Glass	1	7days/40days
Semivolatile Extractables-Priority Pollutants	625	(2) 1000 mL	Amber Glass	1	7days/40days
Semivolatile Extractables-TCL	8270	(2) 1000 mL	Amber Glass	1	7days/40days
Volatile Organics	8260	(2) 40 mL	VOA vial	1,8,9	14 days(~)
Volatile Organics-Priority Pollutants	624	(2) 40 mL	VOA vial	1,8,9	14 days(~)
HPLC ANALYSES					
HPLC-Explosives	8330, 8332	(2) 1000 mL	Amber Glass	1	7days/40days
MICROBIOLOGICAL ANALYSES					
Coliform, Fecal	SM 9222D, SM 9213D Mod.	100 mL	P,G	1,6	6 hours
Coliform, Total	SM 9222B	100 mL	P,G	1,6	30 hours
Coliform and E-coli, Total	SM9223B/Colitag	100 mL	P,G	1,6	30 hours
E-coli	SM9213D, Colilert/Quantitray	100 mL	P,G	1,6	6 hours
Heterotrophic Plate Count	SM9215B SIMPLATE	100 mL	P,G	1,6	30 hours

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

TABLE 1 (cont.)

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – SOLID MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
GENERAL CHEMICAL ANALYSES		**4 oz=100 g**			
% Carbon	9060 mod.	4 oz	Soil Jar	1	28 days
Ammonia-Nitrogen-Automated Phenate	350.1 mod.	4 oz	Soil Jar	1	28 days (^)
Anions	9056	4 oz	Soil Jar	1	48hrs to 28 days from slurry (^)
Cation Exchange Capacity	9081	4 oz	Soil Jar	1	14days/7days (^)
Chloride-Automated Ferricyanide	9251/300.0	4 oz	Soil Jar	1	28days from slurry (^)
Cyanide, Amenable-Spectrophotometric	9012	4 oz	Soil Jar	1	14 days
Cyanide, Total-Spectrophotometric	9012	4 oz	Soil Jar	1	14 days
Fluoride, Potentiometric ISE	300.0 mod./340.2	4 oz	Soil Jar	1	28 days (^)
Lime Equivalency	310.1 mod.	4 oz	Soil Jar	1	28 days (^)
Nitrate+Nitrite-Automated Cadmium Reduction	300.0 mod./353.2	4 oz	Soil Jar	1	28 days (^)
Nitrate-Automated Cadmium Red./Diazotization	300.0 mod./353.2	4 oz	Soil Jar	1	48 hrs from slurry (^)
Nitrite-Automated Diazotization	300.0 mod./353.2	4 oz	Soil Jar	1	48 hrs from slurry (^)
Oil & Grease-Total Recoverable, Gravimetric N-Hexane extractable material N-Hexane extractable material w/ silica gel cleanup	9071	4 oz	Soil Jar	1	28 days (^)
Organic Nitrogen-Auto. Block Digest.,Spectro.	350.1/351.2 mod.	4 oz	Soil Jar	1	28 days (^)
pH (Laboratory)	9045	4 oz	Soil Jar	1	24 hours (^)
Phenolics, Total Recoverable-Manual 4AAP	Mod. 9065	4 oz	Soil Jar	1	28 days (^)
Phosphate, Ortho- Ascorbic Acid	300.0 mod./365.2	4 oz	Soil Jar	1	48 hrs from slurry (^)
Phosphate,Tot.-Auto Ascorbic Acid/Block Dig.	Mod. 365.4	4 oz	Soil Jar	1	28 days (^)
Solids-Ash	SM 2540 F	4 oz	Soil Jar	1	28 days (^)
Solids-Total Solids	CLP-CIP	4 oz	Soil Jar	1	28 days (^)
Solids-Volatile Solids	SM 2540 F	4 oz	Soil Jar	1	28 days (^)
Specific Conductance-Wheatstone Bridge	Mod. 9050	4 oz	Soil Jar	1	28 days (^)
Sulfate-Turbidimetric	9036/9038	4 oz	Soil Jar	1	28 days from slurry (^)
Sulfide-Iodometric	9030	4 oz	Soil Jar	1	7days from slurry (^)
Sulfide-Monier-Williams	40CFR-425	4 oz	Soil Jar	1	28 days (^)
Sulfite-Titrimetric	ASTM D3987/377.1 mod.	4 oz	Soil Jar	1	24 hrs from slurry (^)
TKN-Auto Block Digest,Spectro.	351.2 mod.	4 oz	Soil Jar	1	28 days (^)
Total Organic Halogen	9020/9021	4 oz	Soil Jar	1	28 days (^)
Total Petroleum Hydrocarbons-Extraction, IR	9071	4 oz	Soil Jar	1	28 days (^)
ELEMENTAL ANALYSES					
ICP Elements	6010	4 oz	Soil Jar	1	6 months
ICP MS ELements	6020	4 oz	Soil Jar	1	6 months
GFAA(Furnace) Elements	7000series	4 oz	Soil Jar	1	6 months
Mercury	7471	4 oz	Soil Jar	1	28 days

TABLE 1 (cont.)

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – SOLID MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
ELEMENTAL ANALYSES **(cont.)**		**4 oz=100 g**			
Chromium, Hexavalent	3060/7196	4 oz	Soil Jar	1	30dys/24hrs
GC ORGANIC ANALYSES					
BTEX & MTBE	8021	(2) 40 mL	VOA Vial	1	14 days
Explosives - HPLC	8330, 8332	4 oz	Soil Jar	1	14days/40days
Extractable Petroleum Hydrocarbons	MADEP/EPH	4 oz	Soil Jar	1	7days/40days
Fuel Oil	ME HETL 4.1.25	4 oz	Soil Jar	1	14days/40days
Fule Oil	8015 mod.	4 oz	Soil Jar	1	14days/40days
Gasoline	ME HETL 4.2.17	(2) 40 mL	VOA Vial	1	14 days
Gasoline	8015 mod.	(2) 40 mL	VOA Vial	1	14 days
Herbicides	8151	4 oz	Soil Jar	1	14days/40days
PCB's (& Congeners)	8082	4 oz	Soil Jar	1	14days/40days
PCB's in Oil	8082	4 oz	VOA Vial	1	40 days
Pesticides	8081	4 oz	Soil Jar	1	14days/40days
Pesticides and PCB's	8081/8082	4 oz	Soil Jar	1	14days/40days
Purgeable Aromatics	8021	(2) 40 mL	VOA Vial	1	14 days
Purgeable Halocarbons	8021	(2) 40 mL	VOA Vial	1	14 days
Purgeables, Total	8021	(2) 40 mL	VOA Vial	1	14 days
Solvents (Direct Injection)	8015M	(2) 40 mL	VOA Vial	1	14 days
Volatile Petroleum Hydrocarbons	MADEP/VPH	(2)40 mL	VOA vial	13	28days
HPLC ANALYSES					
HPLC-Explosives	8330, 8332	4 oz	Soil Jar	1	7days/40days
GC/MS ANALYSES					
Acid Extractables-Priority Pollutants	8270	4 oz	Soil Jar	1	14 days/40 days
Acid Extractables-TCL	8270	4 oz	Soil Jar	1	14 days/40 days
Base Neutral Extractables-Priority Pollutants	8270	4 oz	Soil Jar	1	14 days/40 days
Base Neutral Extractables-TCL	8270	4 oz	Soil Jar	1	14 days/40 days
Polyaromatic Hydrocarbons	8270/8270SIM	4 oz	Soil Jar	1	14 days/40 days
Semivolatile Extractables-Priority Pollutants	8270	4 oz	Soil Jar	1	14 days/40 days
Semivolatile Extractables-TCL	8270	4 oz	Soil Jar	1	14 days/40 days
Volatile Organics – High Soil (>200 ug/kg)	5035/8260	Please refer to Table 6-2	Encore or similar sampler or VOA Vial or soil jar	14	Extruded w/in 48 hrs. Analyzed w/in 14 days
Volatile Organics – Low Soil (<200 ug/kg)	5035/8260	Please refer to Table 6-2	Encore or similar sampler or VOA Vial	14 or 15	Extruded w/in 48 hrs. Analyzed w/in 14 days
Volatile Organics-Priority Pollutants	8260	(2) 40 mL	VOA Vial	1	14 days
Volatile Organics-TCL	8260	(2) 40 mL	VOA Vial	1	14 days

TABLE 1 (cont.)

SAMPLING AND PRESERVATION REQUIREMENTS

PARAMETER – SOLID MATRICES	METHOD	QUANTITY	CONTAINER	PRSV	HOLD TIME
RCRA - HAZARDOUS WASTE CHARAC.					
Corrosivity-pH	9045	4 oz	Soil Jar	1	24 hours (^)
Ignitability-Flash Point (closed cup)	1010	4 oz	Soil Jar	1	14 days (^)
Reactivity-Reactive Cyanide	7.3.3.2	4 oz	Soil Jar	1	14 days
Reactivity-Reactive Sulfide	7.3.4.1	4 oz	Soil Jar	1	7 days
TCLP					
TCLP Extraction-Volatile Organics	1311	100 g	Soil Jar	1	14 days
TCLP Extraction-Semivolatiles	1311	200 g	Soil Jar	1	14 days
TCLP Extraction-Pesticides & Herbicides	1311	400 g	Soil Jar	1	14 days
TCLP Extraction-Metals	1311	200 g	Soil Jar	1	28 days
TCLP Analysis-Volatile Organics	8260	see above	Soil Jar	1	14 days
TCLP Analysis-Metals	6010/6020	see above	Soil Jar	1	180 days
TCLP Analysis-Mercury	7470	see above	Soil Jar	1	28 days
TCLP Analysis-Semivolatiles	8270	see above	Soil Jar	1	7 days/40 days
TCLP Analysis-Pesticides	8081	see above	Soil Jar	1	7 days/40 days
TCLP Analysis-Herbicides	8151	see above	Soil Jar	1	7 days/40 days

METHODS OF PRESERVATION
1 = Cool at 4 Degrees Celsius
2 = Settled
3 = H2SO4 to pH<2
4 = HNO3 to pH<2
5 = NaOH to pH>12
6 = 1 mL 0.1M Na2S2O3 or 1 10 mg pellet
7 = 1 m/L 2NZnAc/L & NaOH
8 = 2 drops 1:1 HCl
9 = No headspace
10 = Na2S2O3, if chlorinated
11 = HCl to pH < 2
12 = 5 mL of HCL
13 = 15 mL of methanol
14 = methanol
15 = sodium bisulfate

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 1

EXAMPLE OF STANDARD KATAHDIN CHAIN-OF-CUSTODY FORM

Katahdin ANALYTICAL SERVICES
600 Technology Way
Scarborough, ME 04074
Tel: (207) 874-2400
Fax: (207) 775-4029

CHAIN of CUSTODY

PLEASE BEAR DOWN AND PRINT LEGIBLY IN PEN

Page ____ of ____

Client | Contact | Phone # ( ) | Fax # ( )

Address | City | State | Zip Code

Purchase Order # | Proj. Name / No. | Katahdin Quote #

Bill (if different than above) | Address

Sampler (Print / Sign) | Copies To:

LAB USE ONLY WORK ORDER #:
KATAHDIN PROJECT NUMBER _____

REMARKS: _____

SHIPPING INFO: ☐ FED EX ☐ UPS ☐ CLIENT

AIRBILL NO: _____

TEMP°C _____ ☐ TEMP BLANK ☐ INTACT ☐ NOT INTACT

ANALYSIS AND CONTAINER TYPE PRESERVATIVES

Sample Description	Date / Time coll'd	Matrix	No. of Cntrs.	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N	Filt. ☐Y ☐N
	/												
	/												
	/												
	/												
	/												
	/												
	/												
	/												
	/												
	/												
	/												
	/												
	/												
	/												
	/												
	/												

COMMENTS

Relinquished By: (Signature)	Date / Time	Received By: (Signature)	Relinquished By: (Signature)	Date / Time	Received By: (Signature)
Relinquished By: (Signature)	Date / Time	Received By: (Signature)	Relinquished By: (Signature)	Date / Time	Received By: (Signature)

THE TERMS AND CONDITIONS ON THE REVERSE SIDE HEREOF SHALL GOVERN SERVICES. EXCEPT WHEN A SIGNED CONTRACTUAL AGREEMENT EXISTS

ORIGINAL

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 2

EXAMPLE OF HOMEOWNER KATAHDIN CHAIN-OF-CUSTODY FORM

Katahdin
ANALYTICAL SERVICES

600 Technology Way
P.O. Box 540
Scarborough, ME 04070
Tel: (207) 874-2400 Fax: (207) 775-4029

Homeowner Chain of Custody

Client:	Contact:	Phone:	Fax:
Address:	City:	State:	Zip:
Purchase Order #:	Project Name/No.:	E-mail:	
Billing Address (if different):			
Sampler (Print/Sign):		Copies To:	

***Test results are for compliance and will be reported to the state (see statement below). yes no | Compliance samples must be received on ice.

Lab Use Only | Work Order #: | KAS Project Manager:

Shipping: UPS Fed-Ex Mail Drop-Off

Sample(s) Received on Ice? Yes No Temperature if Iced:

Sample Description (Sample Identification and/or Lot #)	Date Collected	Time Collected	No. of Cntrs.	Standard Homeowner	Arsenic	Total Coliforms	Lead	Safely Test – coliform & N+N	FHA/MSH	Fluoride	Uranium		What's Included in the Standard Test and the FHA/MSH Test.
													Standard Homeowner Total Coliform/e-coli Nitrate, Nitrite Chloride, pH Hardness Copper, Iron Manganese Sodium
													FHA/MSH Standard plus Lead Turbidity Color Odor

Relinquished By:	Date/Time:	Received By:	Relinquished By:	Date/Time:	Received By:

Per the National Environmental Laboratory Accreditation Program (NELAP) Standards, Katahdin is required to accept samples that have been properly preserved. All sample containers provided to you have been properly preserved, but the proper preservation also requires samples to be received at <6 degrees celcius. The Safe Drinking Water Act regulations only require this for compliance samples (i.e., results that are submitted to the state). By circling no for compliance (above), you acknowledge that the samples described above are not for compliance purposes, and thus may not meet the temperature receipt requirements.

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 3

EXAMPLE OF FOOD/MICROBIOLOGY KATAHDIN CHAIN-OF-CUSTODY FORM

Katahdin
ANALYTICAL SERVICES

600 Technology Way
P.O. Box 540
Scarbrough, ME 04070
Tel: (207) 874-2400 Fax: (207) 775-4029

Chain of Custody

Client: Contact: Phone: Fax:

Address: City: State: Zip:

Purchase Order #: Project Name/No.: E-mail:

Billing Address (if different):

Sampler (Print/Sign): Copies To:

Lab Use Only | Work Order #: KAS Project Manager:

Food & Microbiological Services

Shipping: UPS Fed-Ex Airbill No.:

Temperature:

Sample Description (Sample Identification and/or Lot #)	Date/Time Collected	Matrix	No. of Cntrs.	Plate Count (A/H/S)	Listeria	Yeast and Mold	Salmonella	E-Coli	E-Coli 0157:H7	Staph	Vibrio	Total Coliforms	Campylobacter	Shelf Life	Challenge Study		

Relinquished By:	Date/Time:	Received By:	Relinquished By:	Date/Time:	Received By:

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 4

EXAMPLE OF KATAHDIN SAMPLE RECEIPT LOGBBOK

KATAHDIN ANALYTICAL SERVICES, INC.

SAMPLE LOG IN

Date Received	Time Received	Date Logged In	Time Logged In	Work Order	Client	Initials
				SA 0094		
				SA 0095		
				SA 0096		
				SA 0097		
				SA 0098		
				SA 0099		
				SA 0100		
				SA 0101		
				SA 0102		
				SA 0103		
				SA 0104		
				SA 0105		
				SA 0106		
				SA 0107		
				SA 0108		
				SA 0109		
				SA 0110		
				SA 0111		
				SA 0112		
				SA 0113		
				SA 0114		
				SA 0115		
				SA 0116		
				SA 0117		
				SA 0118		
				SA 0119		
				SA 0120		
				SA 0121		
				SA 0122		
				SA 0123		
				SA 0124		

Signed By:_____ Date:_____

Reviewed By:_____ Date:_____

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 5

EXAMPLE OF WET CHEMISTRY SHORTS AND RUSHES LOGBOOK

WET CHEMISTRY SHORTS & RUSHES **Receipt Date:**

HOLDING TIME					Immediate			24 Hr		48 Hr								
Work Order / Client	Matrix	Earliest Sampling Date	Earliest Sampling Time	Rush Parameters	pH	DO	Sulfite	Fe +2	Cr+6	Total BOD	Carbon. BOD	Color	Nitrate	Nitrite	OPO4	Set. Solids	Turbidity	Comments (Quick TAT, MS/MSD, etc.)

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 6

EXAMPLE OF SAMPLE RECEIPT CONDITION REPORT FORM

Katahdin Analytical Services, Inc. **Sample Receipt Condition Report**

Client:	KAS PM:	Sampled By:
Project:	KIMS Entry By:	Delivered By:
KAS Work Order#:	KIMS Review By:	Received By:
SDG #:	Cooler: _____ of _____	Date/Time Rec.:

Receipt Criteria	Y	N	EX*	NA	Comments and/or Resolution
1. Custody seals present / intact?					
2. Chain of Custody present in cooler?					
3. Chain of Custody signed by client?					
4. Chain of Custody matches samples?					
5. Temperature Blanks present? If not, take temperature of any sample w/ IR gun.					Temp (°C):
Samples received at <6 °C w/o freezing?					Note: Not required for metals analysis.
Ice packs or ice present?					The lack of ice or ice packs (i.e. no attempt to begin cooling process) may not meet certain regulatory requirements and may invalidate certain data.
If not, has the cooling process begun (i.e. ice or packs present) and sample collection times <2hrs., but samples are not yet cool?					Note: No cooling process required for metals analysis.
6. Volatiles free of headspace: **Aqueous:** No bubble larger than a pea					
Soil/Sediment: Received in airtight container?					
Received in methanol?					
Methanol covering soil?					
7. Trip Blank present in cooler?					
8. Proper sample containers and volume?					
9. Samples within hold time upon receipt?					
10. Aqueous samples properly preserved? Metals, COD, NH3, TKN, O/G, phenol, TPO4, N+N, TOC, DRO, TPH – pH <2					
Sulfide - >9					
Cyanide – pH >12					

* Log-In Notes to Exceptions: document any problems with samples or discrepancies or pH adjustments

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 7

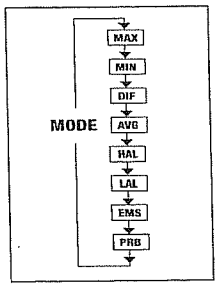
IR THERMOMETER MANUFACTURER'S INSTRUCTIONS FOR CHANGING EMISSIVITY

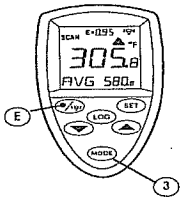
MODE Button Functions

Your infrared thermometer measures Maximum (MAX), Minimum (MIN), Differential (DIF)*, and Average (AVG)** temperatures each time you take a reading. This data is stored and can be recalled with the MODE button (3) until a new measurement is taken. (See "Hold and Recall" for information on how to recall stored data.) When the trigger is pulled again, the unit will begin measuring in the last mode selected. Pressing the MODE button also allows you to access the High Alarm (HAL), Low Alarm (LAL), Emissivity (EMS), Probe temperature (PRB—only available when the probe is connected), and Data logger (LOG). Each time you press MODE, you advance through the mode cycle. The diagram shows the sequence of functions in the Mode cycle.

Note: PRB (probe) is only available in the MODE loop when the contact probe is connected to the unit.

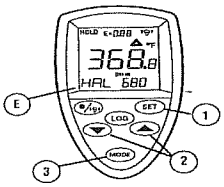
*DIF shows the difference between the maximum and minimum temperatures measured.
**AVG shows the average temperature reading for each time the trigger is pulled or the unit is locked on.

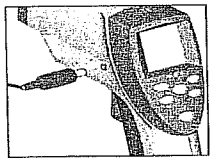


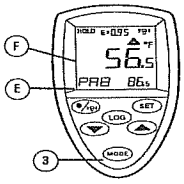


Selecting a Function

To Select the MAX, MIN, DIF, or AVG mode, pull the trigger. While holding the trigger, press the MODE button (3) until the appropriate code appears in the lower left corner of the display (E). Each time you press MODE, you advance through the MODE cycle. The MODE cycle is shown above.







Setting the High Alarm, Low Alarm, and Emissivity

To set values for the High Alarm (HAL), Low Alarm (LAL), and Emissivity, pull the trigger or press the MODE button (3) to activate the display. Press the MODE button until the appropriate code appears in the lower left corner of the display (E). Use the up and down keys (2) to adjust the desired values. To activate the alarms, press SET (1). To deactivate the alarms, press SET again.

Using a Probe (PRB)

Connect the probe to the input on the side of the unit (as shown). PRB automatically appears in the lower left corner of the display (E, below). The probe temperature is shown in the lower right part of the display. The current infrared temperature continues to show in the center of the display (F). While the probe is connected, you may still cycle through the mode functions by pressing MODE (3).

Note: PRB is only available in the MODE loop when a probe is connected to the unit; the probe temperature will not activate the high alarm or low alarm.

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 8

EXAMPLE OF KATAHDIN SAMPLE FILTRATION LOGBOOK

KATAHDIN ANALYTICAL SERVICES, INC.
Sample Filtration Logbook

Katahdin Sample No.	Site ID	Filtration Requested By:		Filtered and Preserved By:		
List Individually	(Optional)	Initials	Date	Initials	Date	Time

Reviewed and Approved by: _____ Date: _____

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 9

MEASURED TURBIDITY AND PRESERVATION OF INCOMING SAMPLES LOGBOOK

KATAHDIN ANALYTICAL SERVICES

Measured Turbidity and Preservation of Incoming Samples

KAS Lab Sample ID	Measured Turbidity (NTU)	Turbidity Date	Turbidity Analyst	Preservation Date	Preservation Time	Preservation Analyst
WW6757-9A	0.22	12-19-06		12/14/06	~~1430~~ 1400	DM
↓ -10A	0.18	↓		↓	↓	↓
WW6758-1A	4.80	↓		↓	↓	↓
WW6776-1A	0.68	12-20-06		12/20/06	1000	DM
WW6855-1A	0.92	12-26-06		12/26/06	1600	DM
WW6860-1A	2.40	↓		↓	↓	↓
SA0027-1A	0.52	1-3-07		01/03/07	1445	DM
SA0028-1A	0.72	1-3-07		↓	↓	↓
SA0087-1A	0.26	1-5-07		01/05/07	1645	DM
SA0088-1A	10.4	1-5-07		↓	↓	↓
SA0110-1A	0.47	1-8-07		01/08/07	1430	DM
SA0109-1A	0.32	1-8-07		↓	↓	↓
SA0138-1A	0.12	1-09-07		01/09/07	1700	DM
SA0262-1A	0.14	1-18-07		01/18/07	0930	DM
SA0263-1A	0.33	1-18-07		↓	↓	↓
SA0267-1A	0.30	1-18-07		01/18/07	0950	DM
SA0313-1A	0.58	1-22-07		01/22/07	1030	↓
SA0316-1A	1.70	1-22-07		01/22/07	1345	DM

REVIEWED BY: DATE:

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 10

EXAMPLE OF LABORATORY INCOMING SAMPLE INFORMATION SHEET (ISIS)

KATAHDIN ANALYTICAL SERVICES, INC. – INCOMING SAMPLE INFORMATION SHEET

CLIENT:	ATTENTION:
ADDRESS:	
TELEPHONE:	FAX:
OTHER CLIENT CONTACTS:	
REPORT CC:	PM/SALES CONTACT:
PO #:	CLIENT ACCOUNT:
KAS QUOTE #:	KIMS QUOTE #:
PROJECT NAME:	KIMS PROJECT REF.:
SDG:	APPLICABLE CERTIFICATION:
RECEIPT DATE:	MEANS OF DELIVERY:

METHOD OR KIMS PRODUCT	MATRIX	# OF SAMPLES	SPECIAL INSTRUCTIONS (I.E LIMITS, PREPS)

SUBCONTRACT PARAMETER	SUBCONTRACT LABORATORY

QC LEVEL:	EDD:
RUSH/VERBAL TAT:	HARDCOPY TAT:
HISTORY:	
OTHER:	

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 11

EXAMPLE OF KATAHDIN WORK ORDER/LOGIN COC REPORT

Katahdin
ANALYTICAL SERVICES

Katahdin Analytical Services
Login Chain of Custody Report (lno1)
Jan. 26, 2007
03:51 PM

Page: 1 of 1

Login Number: SA0395
Account:KATAHD001 Web
Katahdin Analytical Services

Project:

Primary Report Address:
Leslie Dimond
Katahdin Analytical Services
600 Technology Way
P.O. Box 540
Scarborough,ME 04070

Primary Invoice Address:
Accounts Payable
Katahdin Analytical Services
600 Technology Way
P.O. Box 540
Scarborough,ME 04070

Report CC Addresses:
Invoice CC Addresses:

Login Information

ANALYSIS INSTRUCTIONS	:
CHECK NO.	:
CLIENT PO#	:
COOLER TEMPERATURE	: n/a
DELIVERY SERVICES	: In House
EDD FORMAT	:
MAIL DATE	:
PM	: LAD
PROJECT NAME	: QC Holding Blanks
QC LEVEL	: I
REGULATORY LIST	:
REPORT INSTRUCTIONS	:
SDG ID	:
SDG STATUS	:

Laboratory Sample ID	Client Sample Number	Collect Date/Time	Receive Date	PR	Verbal Date	Due Date	Comments
SA0395-1	WHITE FRIDGE	26-JAN-07 15:50	26-JAN-07			08-FEB-07	
Matrix	*Product*	*Hold Date (shortest)*	*Bottle Type*		*Bottle Count*		
Aqueous S	SW8260FULL5ML	09-FEB-07			2		
SA0395-2	BLUE FRIDGE	26-JAN-07 15:50	26-JAN-07			08-FEB-07	
Matrix	*Product*	*Hold Date (shortest)*	*Bottle Type*		*Bottle Count*		
Aqueous S	SW8260FULL5ML	09-FEB-07			2		

Total Samples: 2 ***Total Analyses:*** 2

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 12

EXAMPLE OF LABORATORY INTERNAL CHAIN-OF-CUSTODY FORM

Katahdin Analytical Services, Inc.
Internal Custody Record

EXTRACTIONS (AQ) Work Order #:_____

Sample Numbers Involved	Placed in refrigeration by: Initials
	Date
	Time

Sample Numbers	Analysis	Removed			Returned			Consumed?	
		Initials	Date	Time	Initials	Date	Time	Yes	No

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 13

EXAMPLE OF REFRIGERATOR TEMPERATURE LOGBOOK

KATAHDIN ANALYTICAL SERVICES, INC.

SAMPLE RECEIPT WALK-IN TEMPERATURE LOG

Corrective Action: Note in the "comments" column and notify the QAO or supervisor; document corrective actions taken and return to control.

Thermometer Location		Sample Receipt Walk-in 1	Comments
Acceptance Criteria		2 to 6 °C	
Date	Initials	Temp (°C)	
02/2/07	ND4	3.7	
02/05/07	DWM	5.8	
02/06/07	DWM	5.8	
02/07/07	DWM	5.8	
02/08/07	DWM	3.8	
02/09/07	DWM	3.5	
02/12/07	DWM	3.5	
02/13/07	DWM	3.5	

TITLE: SAMPLE RECEIPT AND INTERNAL CONTROL

FIGURE 14

EXAMPLE OF IMMEDIATE INTERNAL COC LOGBOOK

KATAHDIN ANALYTICAL SERVICES, INC.
INTERNAL CUSTODY RECORD FOR IMMEDIATES

CLIENT	PROJECT	CLIENT ID &/or WORK ORDER #	ANALYSIS	OUT date/time	IN date/time	INIT	Consumed?
Jacobs		WW4813-1A, -2A	ICP	9/13/06 0930 →	0935	DJJ	yes no
Jacobs		WW4883-1A	ICP	9/16/06 0900 →	1000	DJJ	yes no
CES		WW4965	BOD	9/20/06 0920	9/20/06 1200	CP	yes no
CCAR		WW4969	BOD	9/20/06 1000	↓	CP	yes no
GEMF		WW4970	BOD	↓	↓	CP	yes no
Jacobs		WW4962-1A, -2A	ICP	9/20/06 0900 →	1000	DJJ	yes no
Irving		WW4994	BOD	9/21/06 1000	9/21/06 1015	CP	yes no
Highmer		WW4992	BOD	9/21/06 1015		CP	yes no
NATIONAL		WW5000	TS, PERCHLORE PH, SP.COND	9/21/06 1100	9/21/06 1257	[illegible]	yes no
W+C		WW5001	BOD	9/21/06 1300		CP	yes no
Aliens		WW5016	NO3	9/22/06 1300 NO3	09/22/06 1100	[illegible]	yes no
RASSOM		WW5010	↓	↓ ↓	↓	↓	yes no
EcoMaine		WW5029	BOD	9/22/06 1100		CP	yes no

QAQC143

0000095

TITLE: **SAMPLE DISPOSAL**

Prepared By: _____ Date: 2/01

Approved By:

Group Supervisor: _____ Date: _____

Operations Manager: _____ Date: 2/01

QA Officer: Deborah J. Nadeau Date: 2·01

General Manager: _____ Date: 2/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Format changes, ~~added pollution prevention~~, added updated log book and greater detail on disposal.	DN	2·01	2/01
02	Major rewrite to include more detail on hazardous waste regulations + to reflect current practices.	DN	02/05	02/05
03	Rewrite of section 7 to comply with current practices in new facility. Updated Figures 1 to 3.	DN	02·08	02·08
04	Added elementary neutralization to section 7.0. Other minor edits.	DN	05·09	0509

TITLE: SAMPLE DISPOSAL

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SD-903-04**, titled **SAMPLE DISPOSAL**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SD-903-04**, titled **SAMPLE DISPOSAL**.

Recipient: _____ Date: _____

TITLE: SAMPLE DISPOSAL

1.0 SCOPE AND APPLICATION

Katahdin Analytical Services, Inc. requires strict adherence to specific procedures for the disposal of samples. The procedures are designed to categorize waste materials, provide for their safe and timely disposal and to ensure compliance with local and federal regulations pertaining to disposal of chemicals and environmental samples. Any other means of disposal not described in this SOP is prohibited without consent from the Katahdin Environmental Health & Safety Officer and/or the Katahdin Environmental Compliance Officer.

The purpose of this SOP is to describe the procedures utilized by Katahdin Analytical personnel for the disposal of samples. These procedures apply to the disposal of all samples received or processed by Katahdin. Refer to the current revision of Katahdin SOP CA-107 regarding the disposal of spent preparation and analysis reagents, standards, sample extracts, distillates, or digestates.

1.1 Definitions

Hazardous Waste – A "Solid Waste" which displays a hazardous characteristic or is specifically listed as hazardous waste.

Solid Waste – Any discarded material that is not excluded from the definition of hazardous waste.

Discarded Material – Material that is abandoned, recycled or inherently waste-like.

Waste (State of Maine) –

- Any useless, unwanted, or discarded substance or material, whether or not such substance or material has any other future use.
- Any substance or material that is spilled, leaked, pumped, poured, emptied or dumped onto the land or into the water or ambient air.
- Materials which are used in a matter constituting disposal, burned for energy recovery, reclaimed, or accumulated speculatively.

Ignitable Hazardous Waste – EPA Waste Code D001

- Liquids with a flash point less than 140°F or 60°C.
- Solids capable of spontaneous combustion under normal temperature and pressure.
- Ignitable compressed gas.
- Oxidizers.

Corrosive Hazardous Waste - Liquids with a pH less than or equal to 2.0 or greater than or equal to 12.5. EPA waste code D002.

Reactive Hazardous Waste – EPA waste code D003.

- A material that reacts violently with water.
- A material that generates toxic gases or fumes.
- Explosives.

Toxic Hazardous Waste – A material that exceeds certain concentration levels based on the toxicity characteristic leaching procedure (TCLP). See Figure 3 for the chemicals and concentration levels covered under this definition.

Listed Wastes – Lists of chemicals that are considered hazardous based on the following criteria

- Virgin chemical or unused product.
- Sole active ingredient.
- Single substance spill debris.

Listed wastes are divided into 5 subcategories

- F-wastes – Describe hazardous waste from non-specific sources usually containing halogenated and non-halogenated solvents.
- K-wastes – Describe hazardous wastes created by specific processes.
- U-wastes – Describe toxic or non-acute hazardous wastes.
- P-wastes – Describe acute hazardous wastes. (Note: Maine considers a material to be a P-listed waste if it contains 10% or more of any P-listed chemical.
- State listed wastes – Maine lists any material with a concentration of greater than 50 ppm Polychlorinated Biphenyls (PCB) as a hazardous waste.

Organics hit – A liquid sample containing greater than 1 mg/L of organic contaminants or a soil sample containing greater than 20 mg/kg of organic contaminants.

1.2 Responsibilities

Only designated analysts/technicians trained in these procedures may dispose of samples or analytical by-products. Each analyst or technician must be familiar with Katahdin Analytical safety procedures. Gloves, safety glasses, lab coats and/or other protective clothing must be worn at all times.

It is the responsibility of the designated Katahdin personnel involved in the disposal of samples to read and understand this SOP, to adhere to the procedures outlined, to properly document their activities in the appropriate lab notebook and file the necessary manifests and reports to outside agencies in the required manner. Refer to

Katahdin SOP QA-805, "Personnel Training & Documentation of Capability," current revision.

It is the responsibility of the Department Managers to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

It is the responsibility of the Katahdin Environmental Health & Safety Officer (EHSO) to manage the proper classification and disposal of samples. Katahdin is responsible for regulatory compliance of Katahdin's waste storage areas (less than 90 day storage). The EHSO ensures compliance of the waste storage areas with applicable state and federal regulations. The EHSO is responsible for providing the appropriate training to all individuals involved in the proper classification and/or disposal of samples. The EHSO is responsible for working with the Laboratory Operations Manager/Environmental Compliance Officer to help identify problems and assure resolution, to facilitate corrective action where needed, and to communicate unresolved problems and concerns to the Laboratory Vice President.

It is the responsibility of the Operations Manager/Environmental Compliance Officer to oversee adherence to Katahdin sample disposal and hazardous waste practices by all laboratory groups under his/her authority, to help identify problems and assure resolution, to facilitate corrective action where needed, and to communicate problems and concerns to the EHSO and/or the Laboratory Vice President.

It is the responsibility of the Laboratory Vice President to provide the necessary resources to meet the regulatory requirements of proper classification and disposal of samples.

2.0 SUMMARY OF METHOD

Not applicable.

3.0 INTERFERENCES

Not applicable.

4.0 APPARATUS AND MATERIALS

Not applicable.

TITLE: SAMPLE DISPOSAL

5.0 REAGENTS

Not applicable.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Not applicable.

7.0 PROCEDURES

7.1 Sample purging is the removal of samples from laboratory refrigerated storage. Sample storage areas where samples are removed (purged) from include wet chemistry, organic extractables, metals, volatiles, total organic carbon and soils. Wet chemistry, aqueous metals, organic extractables, total organic carbon, and soils can all be found in the walk-in refrigerator. Aqueous and soil volatiles can be found in the volatiles laboratory refrigerators/freezer.

7.2 Samples are purged from storage, after analysis and reporting, on a routine basis to make room for incoming samples. Samples are to be kept in storage for a duration of 30 days past the report mailed date. Some samples must be kept for 60 or 90 days beyond the report mailed date, depending on specific client requests and contracts.

7.3 The first step in disposing of samples is to generate a disposal list. The disposal list contains sample analysis information stored in the Katahdin Information Management System (KIMS). The analytical data for the samples is compared to the hazardous waste criteria specified in 40CFR Part 261 and to local wastewater discharge criteria. Refer to Figure 4 for 40 CFR Part 261 Characteristic Hazardous Waste Criteria. Based on this comparison, the report displays information on the classification/category for disposal of each sample. The disposal report should be reviewed against the data reports for accuracy. Refer to Figure 2 for an example of a KIMS generated disposal list. The primary disposal categories listed in the report are: non-hazardous, high organics, high metals, flashpoint, high mercury, high PCBs, and high cyanide. Katahdin has established 14 waste stream profiles with a 3rd party waste transporter/waste disposal firm for sample disposal based on these categories. As required, new or special temporary waste profiles are established based on the characteristics of samples.

7.4 Sorting through samples and preparing them for disposal is a crucial quality checkpoint. Samples put into the incorrect waste stream could not only produce adverse environmental effects, but, could also interrupt the 3rd party's waste treatment efficiency, or endanger an individual handling the waste stream. Therefore, when sorting through samples pay close attention to which waste stream each sample falls into.

7.5 Once you are ready to dispose of the samples of interest (the oldest samples that have been purged), these samples must be sorted, logged, and the classification/category (sample knowledge) information recorded.

Sample storage times (as listed in section 7.2) and space should be taken into consideration when purging samples. It is important to make room for future samples, but to make sure that samples are not purged too early. Samples should be pulled from the walk-in or the volatiles refrigerators to make room for new samples. When purging, chose a section that needs extra space the most and remove the oldest samples.

Safety glasses, nitrile gloves, lab coat, and a splash apron must be worn when handing samples during disposal

7.6 Remove the designated purge samples from the shelf one by one and line them up on the countertop in the log-in area. Generally, removing two cartloads at a time is a good amount to purge at one time. For volatile samples in 40mL vials, 5 or 6 vial trays should be purged at a time. Samples should be lined up across the counter with the earliest sample to the left and building up to the right, organizing the samples according to work order and sample number. After the samples are lined up, they should be recorded in the Sample Disposal Logbook (SDL). Refer to Figure 1 for an example SDL page. The location the samples were removed from should also be recorded. Sample storage areas are recorded with the following designations:

VOA (Aq)	Aqueous Volatiles(VOA)
VOA (SL)	Solid Volatiles(VOA)
M	Metals
EXT	Extractables (Organic)
TOC	Total Organic Carbon
WC	Wet Chemistry
S	Soils

7.7 The next step is to use the sample disposal list to determine the earliest release date of the reports and to determine each samples appropriate waste classification/characterization. As stated in section 7.3, the primary disposal categories listed in the report are: non-hazardous, high organics, high metals, flashpoint, high mercury, high PCBs, and high cyanide.

Using the information from the KIMS disposal list, record the appropriate classification for each sample in the SDL. If multiple categories are identified as being present then a single category is selected as controlling. The order of precedence is PCB's, metals and then organics. If another scenario is found, the individual should bring it to the EHSO for a determination of the acceptable waste stream designation or a determination that it should be lab packed separately.

If samples have been sorted that have not been in storage for the 30 days beyond the release date (60 or 90 for certain clients), then these samples need to be placed back in storage and it should be noted in the SDL.

7.8 As stated above, a sample may be categorized into a waste stream based upon the analytes it contains as determined by laboratory testing. In addition, many samples are also categorized as hazardous waste based upon the preservative that they contain. Since many samples contain preservatives, caution must be used when dumping samples. It is also important to ensure that the sample container is empty. This can be accomplished by holding the container upside down and shaking gently until liquid is no longer observed coming out of the container.

7.9 Once waste categories have been determined and entered into the SDL, The following waste categories are disposed of as follows:

7.9.1 Dumping non-hazardous samples (as determined by laboratory testing)

Non-hazardous samples (non-preserved) are poured directly into the sink in the warehouse.

Non-hazardous solid samples are disposed of with the general trash, which is picked up by commercial trash collectors and ultimately disposed of in a waste-to-energy incinerator.

Sample containers from non-hazardous samples are disposed of with the general trash.

7.9.2 Dumping Samples with high Organics (as determined by laboratory testing)

Aqueous samples get dumped into waste stream "K". Containers are disposed of with general trash. Solid samples are placed into waste stream "I" with their containers. The disposal date is recorded in the SDL.

7.9.3 Dumping samples high in metals, including mercury (as determined by the by laboratory testing)

Aqueous samples get disposed of in waste stream "A". Containers are disposed of with general trash. Solid samples are placed in waste stream "L" with their containers. The disposal date is recorded in the SDL.

7.9.4 Dumping Acidic Samples that do not contain any other hazardous waste constituents (as determined by the acidic preservative or by laboratory testing)

Refer to section 7.10 below.

7.9.5 Dumping Basic samples (as determined by the basic preservative or by laboratory testing)

Aqueous samples get disposed of in waste stream "NHi". Containers are disposed of with general trash. The disposal date is recorded in the SDL.

7.9.6 Dumping samples with high PCBs (as determined by laboratory testing)

Aqueous samples are disposed of in waste stream "Q". Containers are disposed of with general trash. Solid samples get disposed of in waste stream "F" with their containers. The disposal date is recorded in the SDL.

7.9.7 Dumping samples with low flashpoints (as determined by laboratory testing)

Aqueous samples are disposed of in waste stream "O". Containers are disposed of with general trash. Solid samples get disposed of in waste stream "I" with their containers. The disposal date is recorded in the SDL.

7.9.8 Dumping samples with high cyanide (as determined by laboratory testing)

Aqueous samples are disposed of in waste stream "NHi". Containers are disposed of with general trash. Solid samples should be set aside for labpack. The disposal date is recorded in the SDL.

7.9.9 Miscellaneous Disposal (as determined by the preservative)

Sodium Bisulfate: Sodium Bisulfate often comes in vials, but may also come in the 2-4oz glass jars. Dump the Sodium Bisulfate out of the container into waste stream "A". There may be remaining soil left in the sample container. The soil's waste stream and dump date will be dictated by the SDL. The disposal date is recorded in the SDL.

Methanol / Free Products: This often comes in vials, but may also come in the 2-4oz glass jars. Dump the methanol out of the container into the mix-flammables accumulation. When this satellite accumulation container gets full it can be dumped into the "O" waste stream. There may be remaining soil left in the sample container. The soil's waste stream and dump date will be dictated by the SDL. Lastly, samples marked "free product" on the Katahdin sample ID label can be dumped into the mixed flammables stream. The disposal date is recorded in the SDL.

7.10 Pursuant to Maine DEP regulations, Katahdin has the necessary agreements, processes and documentation in place to neutralize samples without a license. Refer to the current revision of the Katahdin Environmental Health & Safety Manual for additional information. Generally, the following procedures are followed.

7.10.1 Samples that have been determined to be hazardous due **solely** to the corrosivity characteristic are neutralized using sodium hydroxide pellets. In the warehouse, samples are emptied into a five gallon heavy duty carboy to about 60% capacity. The carboy is kept in a secondary container. Sodium hydroxide pellets are added slowly to the carboy (about 5 grams at a time) and stirred with a long glass stirring rod. The pH is checked with pH paper.

7.10.2 This process is continued until the pH is between 7 and 8. This normally takes about 30-40 grams of sodium hydroxide pellets, but may vary depending on the buffering capacity of the individual samples.

7.10.3 The carboy is emptied into the sink in the warehouse. The tap water is run at the same time as the neutralized material is disposed of. An eyewash station and spill material is located at this sink.

7.10.4 All neutralization activities are documented, including the date and time of neutralization, the name of the person doing the neutralizing, the amount of neutralized liquid discharged, details on the inspection of the drain area and the date and nature of any significant repairs or corrective actions. This documentation is maintained by the EHSO. Refer to Figure 5 for an example logbook page of neutralization documentation.

7.11 Every 3 to 5 weeks a pickup of hazardous waste is scheduled with the 3rd party waste transporter/waste disposal firm. An inventory is faxed to the transporter summarizing the number of drums and waste streams/profiles. As required, a "lab pack" of expired chemicals or orphan samples is organized as necessary. A designated individual, with applicable Hazardous Waste (RCRA) and Department of Transportation (DOT) training, oversees the waste pickup and signs the hazardous manifests and land ban documentation. Within 7 days a copy is forwarded to the Maine Department of Environmental Protection (MEDEP) and the environmental agency in the designation state (if required by that state). Once the report is received at the disposal facility a copy is returned to KATAHDIN and the MEDEP.

7.12 Prior to March 31 of each year, the laboratory prepares the Annual Hazardous Waste Report (i.e., MEDEP modified EPA Form 8700-13A) as required by MEDEP Hazardous Waste Management Rules. The complete report is reviewed by the Katahdin Environmental Compliance Officer and then forwarded to the following address:

Maine Department of Environmental Protection
Bureau of Remediation & Waste Management
State House Station #17
Augusta, ME. 04333
Attn: Annual Hazardous Waste Report

TITLE: SAMPLE DISPOSAL

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

On a daily basis, a designated individual performs quality checks in all hazardous waste storage areas. The daily check documentation is located in login. Any discrepancy is copied to the Operations Manager and the Katahdin Vice President for corrective action. Refer to the current revision of Katahdin SOP CA-107, *The Management of Hazardous Waste as it Relates to the Disposal of Laboratory Process Waste, Reagents, Solvents & Standards*, for more information. Refer to Figure 3 for a copy of the daily check documentation.

9.0 METHOD PERFORMANCE

Not applicable.

10.0 APPLICABLE DOCUMENTS/REFERENCES

USEPA Code of Federal Regulations, 40 CFR Part 261.

Maine Department of Environmental Protection (ME DEP) Hazardous Waste Management Rules

ME DEP modified EPA Form 8700-13A

LIST OF TABLES AND FIGURES

Figure 1 Example of Sample Disposal Logbook
Figure 2 Example of KIMS Generated Waste Disposal Report
Figure 3 Example Of Hazardous Waste Area Daily Check Documentation
Figure 4 Characteristic Toxic Hazardous Waste and TCLP concentrations
Figure 5 Example of Elementary Neutralization Logbook

TITLE: SAMPLE DISPOSAL

FIGURE 1

EXAMPLE OF SAMPLE DISPOSAL LOGBOOK (SDL)

KATAHDIN ANALYTICAL SERVICES, INC. –SAMPLE STORAGE/DISPOSAL LOGBOOK

WORK ORDER/ SAMPLE NUMBERS	DEPARTMENT	EARLIEST RELEASE DATE	< CRITERIA	SAMPLE KNOWLEDGE									DATE DISPOSED	INITIALS
			CLEAN	WL	ORG	METS	CN	FP	HG	PCBS				
SAS783-1	WC	10-17-07	✓										1-22-08	GN
SAS786-1		10-17-07	✓											
SAS787-1,2,4		10-17-07	✓											
SAS790-1		10-19-07		✓										
SAS793-1		10-17-07	✓											
SAS795-1-9		10-23-07	✓											
SAS797-1		10-23-07	✓											
SAS798-1,2		10-25-07			✓									
SAS799-1-5		10-31-07	✓											
SAS804-1,2		10-25-07	✓											
SAS809-1,2		10-23-07	1			2				2				
SAS810-1-4		10-23-07	✓											

QAQC354

0000022

TITLE: SAMPLE DISPOSAL

FIGURE 2

EXAMPLE OF KIMS GENERATED WASTE DISPOSAL REPORT

SAMPLE DISPOSAL REPORT

Query by: Login SA6501 to SA7000
Date : 15-JAN-08

Sample	SDG	Status	Mail Date	Parameter	Value		
SA6605-1		NEED	12/02/07				
SA6606-1		NEED	12/02/07				
SA6607-1		NEED	11/15/07				
SA6608-1		NEED	12/06/07	ORG	1.17	MG/L	(HIGH)
SA6608-1		NEED	12/06/07				
SA6608-2		NEED	12/06/07	AA	13	MG/KG	(HIGH)
SA6609-1		NEED	11/26/07				
SA6609-1		NEED	11/26/07				
SA6610-1		NEED	11/30/07				
SA6611-1	FCS-020	NEED	12/07/07				
SA6611-2	FCS-020	NEED	12/07/07				
SA6611-3	FCS-020	NEED	12/07/07				
SA6611-4	FCS-020	NEED	12/07/07				
SA6611-5	FCS-020	NEED	12/07/07				
SA6611-6	FCS-020	NEED	12/07/07				
SA6611-7	FCS-020	NEED	12/07/07				
SA6611-8	FCS-020	NEED	12/07/07				
SA6612-1	NSA-030	NEED	12/07/07				
SA6612-2	NSA-030	NEED	12/07/07				
SA6612-3	NSA-030	NEED	12/07/07				
SA6612-4	NSA-030	NEED	12/07/07	ORG	1.70735	MG/L	(HIGH)
SA6612-5	NSA-030	NEED	12/07/07	ORG	1.0481	MG/L	(HIGH)

Page 24 of 99

TITLE: SAMPLE DISPOSAL

FIGURE 3

EXAMPLE OF HAZARDOUS WASTE STORAGE AREA DAILY CHECK

Daily Checklist for
HAZARDOUS WASTE STORAGE AREA

Week of: 1-28, 20 03

Item/Day:	Monday	Tuesday	Wednesday	Thursday	Friday
1. Are containers closed? (Except when waste is being added)	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
2. Are containers properly labeled with a hazardous waste label?	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
3. Do you have access to each container and can you read the label? (36" aisle?)	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
4. Is each container marked with the date storage began?	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
5. Are the dates on the containers less than 90 days old?	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
6. Is container free of dents, bulges, rust, spills or leaks?	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
7. Are all containers on a firm working surface?	Yes / No	Yes / No	Yes / No	Yes / No	Yes / No
8. Inspection by: Name (No Initials)	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
9. Time of Inspection	16:35	15:00	14:45	14:15	16:25
10. Verification of Inspection (Name/Date)	[illegible] 1-28-03	[illegible] 1-29-03	[illegible] 1-30-03	[illegible] 1-31-03	[illegible] 1-02-03

Deficiency noted:					
Corrective action:					
By (Name/Date):					

QAQC315

0000060

TITLE: SAMPLE DISPOSAL

FIGURE 4

CHARACTERISTIC TOXIC HAZARDOUS WASTE AND TCLP CONCENTRATIONS

Chemical Name	CAS Number	Waste Code	TCLP conc. liquid	Equivalent conc. In Soil
Arsenic	7440-38-2	D004	5.0 mg/L	100 mg/kg
Barium	7440-39-3	D005	100 mg/L	2000 mg/kg
Cadmium	7440-43-9	D006	1.0 mg/L	20 mg/kg
Chromium	7440-47-3	D007	5.0 mg/L	100 mg/kg
Lead	7439-92-1	D008	5.0 mg/L	100 mg/kg
Mercury	7439-97-6	D009	0.2 mg/L	4 mg/kg
Selenium	7782-49-2	D010	1.0 mg/L	100 mg/kg
Silver	7440-22-4	D011	5.0 mg/L	20 mg/kg
Endrin	72-20-8	D012	0.02 mg/L	0.4 mg/kg
Lindane	58-89-9	D013	0.4 mg/L	8 mg/kg
Methoxychlor	72-43-5	D014	10 mg/L	200 mg/kg
Toxaphene	8001-35-2	D015	0.5 mg/L	10 mg/kg
2,4-D	94-75-7	D016	10 mg/L	200 mg/kg
2,4,5-TP (Silvex)	93-72-1	D017	1.0 mg/L	20 mg/kg
Benzene	71-43-2	D018	0.5 mg/L	10 mg/kg
Carbon Tetrachloride	56-23-5	D019	0.5 mg/L	10 mg/kg
Chlordane	57-74-9	D020	0.03 mg/L	0.6 mg/kg
Chlorobenzene	108-90-7	D021	100 mg/L	2000 mg/kg
Chloroform	67-66-3	D022	6.0 mg/L	120 mg/kg
o-Cresol	95-48-7	D023	200 mg/L	4000 mg/kg
m-Cresol	108-39-4	D024	200 mg/L	4000 mg/kg
p-Cresol	106-44-5	D025	200 mg/L	4000 mg/kg
Cresol	1319-77-3	D026	200 mg/L	4000 mg/kg
1,4-Dichlorobenzene	106-46-7	D027	7.5 mg/L	150 mg/kg
1,2-Dichloroethane	107-06-2	D028	0.5 mg/L	10 mg/kg
1,1-Dichloroethylene	75-35-4	D029	0.7 mg/L	14 mg/kg
2,4-Dinitrotoluene	121-14-2	D030	0.13 mg/L	2.6 mg/kg
Heptachlor	76-44-8	D031	0.008 mg/L	0.16 mg/kg
Hexachlorobenzene	118-74-1	D032	0.13 mg/L	2.6 mg/kg
Hexachlorobutadiene	87-68-3	D033	0.5 mg/L	10 mg/kg
Hexachloroethane	67-72-1	D034	3.0 mg/L	60 mg/kg
Methyl Ethyl Ketone	78-93-3	D035	200 mg/L	4000 mg/kg
Nitrobenzene	98-95-3	D036	2.0 mg/L	40 mg/kg
Pentachlorophenol	87-86-5	D037	100 mg/L	2000 mg/kg
Pyridine	110-86-1	D038	5.0 mg/L	100 mg/kg
Tetrachloroethylene	127-18-4	D039	0.7 mg/L	14 mg/kg
Trichloroethylene	79-01-6	D040	0.5 mg/L	10 mg/kg

FIGURE 4, cont'd

CHARACTERISTIC TOXIC HAZARDOUS WASTE AND TCLP CONCENTRATIONS

Chemical Name	CAS Number	Waste Code	TCLP conc. liquid	Equivalent conc. In Soil
2,4,5-Trichlorophenol	95-95-4	D041	400 mg/L	8000 mg/kg
2,4,6-Trichlorophenol	88-06-2	D042	2.0 mg/L	40 mg/kg
Vinyl Chloride	75-01-4	D043	0.2 mg/L	4.0 mg/kg

TITLE: SAMPLE DISPOSAL

FIGURE 5

EXAMPLE OF ELEMENTARY NEUTRALIZATION LOGBOOK

Katahdin Analytical Services, Inc. – Elementary Neutralization Logbook

Date: 3-4-09		**Time:** 12:00	**Analyst:** GN
# of gallons neutralized	Final pH	Condition of drain and sink area before and after neutralization.	Significant Repairs or Corrective Actions
5	5	good	
6	7	good	
6	5	good	
6	6	good	
2	8	good	

25
240

Date: 3-10-09		**Time:** 13:45	**Analyst:** GN
# of gallons neutralized	Final pH	Condition of drain and sink area before and after neutralization.	Significant Repairs or Corrective Actions
6	7	good	
6	6	good	
5	6	good	
6	8	good	
6	5	good	
6	8	good	
5	5	good	
6	7	good	
3	5	good	

49
289

Leslie Dimond

From: Moreno, Pati (NFESC) [pati.moreno@navy.mil]
Sent: Tuesday, March 31, 2009 4:13 PM
To: Leslie Dimond
Subject: RE: Navy Assessment - Katahdin
Signed By: There are problems with the signature. Click the signature button for details.

Ms. Dimond,

This email addresses the status of Katahdin Analytical Services, located at 600 Technology Way, Scarborough, Maine in the Navy Environmental Restoration (ER) Quality Assurance (QA) Program as administered by the Naval Facilities Engineering Service Center (NFESC).

NAVSEA 04XQ conducted a laboratory assessment of Katahdin Analytical Services of Scarborough, Maine as a support service to the Naval Facilities Engineering Service Center (NFESC). The general conclusion of the assessment is that the laboratory has successfully completed the evaluation for the parameters summarized in NAVSEA 04XQ letter Ser 04XQ (LABS)/205 dated August 7, 2007; these results are applicable to the Navy ER QA Program administered by NFESC. Based on the outcome of the assessment, a re-evaluation of your laboratory under the Navy ER QA Program will be due not later than June 4, 2009.

The outcome of this assessment does not guarantee the delivery of any analytical samples, and is facility specific (i.e. is applicable to the location specified and is not applicable to an affiliated or subcontract laboratory). The Navy reserves the right to conduct additional laboratory assessments. The outcome of the assessment may result in the addition or removal of parameters listed in the original scope of review, or reclassification from successful to unsuccessful.

Sincerely,
Pati Moreno
NFESC ER QA Program Coordinator
PH: (805) 982-1659 Fax: (805) 982-4304
Email: pati.moreno@navy.mil

No virus found in this incoming message.
Checked by AVG - www.avg.com
Version: 8.0.238 / Virus Database: 270.11.32/2030 - Release Date: 03/31/09 13:05:00

Leslie Dimond

From: Moreno, Pati (NFESC) [pati.moreno@navy.mil]
Sent: Tuesday, December 02, 2008 11:44 AM
To: Leslie Dimond
Subject: RE: Navy Assessment - Katahdin

Ms. Dimond,

This email addresses the status of Katahdin Analytical Services, located at 600 Technology Way, Scarborough, Maine in the Navy Environmental Restoration (ER) Quality Assurance (QA) Program as administered by the Naval Facilities Engineering Service Center (NFESC).

NAVSEA 04XQ conducted a laboratory assessment of Katahdin Analytical Services of Scarborough, Maine as a support service to the Naval Facilities Engineering Service Center (NFESC). The general conclusion of the assessment is that the laboratory has successfully completed the evaluation for the parameters summarized in NAVSEA 04XQ letter Ser 04XQ (LABS)/205 dated August 7, 2007; these results are applicable to the Navy ER QA Program administered by NFESC. Based on the outcome of the assessment, a re-evaluation of your laboratory under the Navy ER QA Program will be due not later than April 4, 2009.

The outcome of this assessment does not guarantee the delivery of any analytical samples, and is facility specific (i.e. is applicable to the location specified and is not applicable to an affiliated or subcontract laboratory). The Navy reserves the right to conduct additional laboratory assessments. The outcome of the assessment may result in the addition or removal of parameters listed in the original scope of review, or reclassification from successful to unsuccessful.

Sincerely,
Pati Moreno
NFESC ER QA Program Coordinator
PH: (805) 982-1659 Fax: (805) 982-4304
Email: pati.moreno@navy.mil
No virus found in this incoming message.
Checked by AVG - http://www.avg.com
Version: 8.0.176 / Virus Database: 270.9.12/1824 - Release Date: 12/2/2008 9:31 AM

DEPARTMENT OF THE NAVY

NAVAL FACILITIES ENGINEERING SERVICE CENTER
1100 23RD AVE
PORT HUENEME CA 93043-4370

IN REPLY REFER TO:

NFESC 413
August 23, 2007

Ms. Leslie Dimond
Quality Assurance Officer
Katahdin Analytical Services
340 Country Road No. 5
Westbrook, ME 04098

Dear Ms. Dimond,

This correspondence addresses the status of Katahdin Analytical Services of Westbrook, Maine in the Navy Environmental Restoration (ER) Quality Assurance (QA) Program as administered by the Naval Facilities Engineering Service Center (NFESC).

Your laboratory is accepted to perform sample analysis for the methods listed in Table 1. The period of acceptance expires September 30, 2008. This acceptance does not guarantee the delivery of any analytical samples. Acceptance is facility specific and can not be transferred to an affiliated or subcontract laboratory.

The Navy's assessment included a review of the laboratory's QA manual, selected standard operating procedures (SOPs) and SOP master list, list of major analytical instrumentation, performance test (PT) results and onsite assessment documentation[1].

The Navy reserves the right to conduct additional laboratory assessments or to suspend or revoke acceptance status for any or all of the listed parameters if deemed necessary.

Table 1

METHOD	PARAMETER	MATRIX
300 Series/9056	Anions: Bromide, Chloride, Fluoride, Nitrate, Nitrite, Orthophosphate, Phosphorus, Sulfate, Sulfide, Sulfite	Water/Solid
8260B	Volatile Organic Compounds	Water/Solid
8270C	Semivolatile Organic Compounds	Water/Solid
8081A	Organochlorine Pesticides	Water/Solid
8330	Explosives	Water/Solid
8082	Polychlorinated Biphenyls (PCBs)	Water/Solid

[1] The State of Florida conducted the onsite on August 28-30, 2006 to assess laboratory conformance with National Environmental Laboratory Accreditation Conference (NELAC) requirements. A supplementary Navy onsite was conducted May 8, 2007.

6010B/7000A	TAL Metals: Aluminum, Antimony, Arsenic, Barium, Beryllium, Cadmium, Calcium, Chromium, Cobalt, Copper, Iron, Lead, Magnesium, Manganese, Mercury, Molybdenum, Nickel, Potassium, Selenium, Silver, Sodium, Thallium, Vanadium, and Zinc	Water/Solid
6020	TAL Metals: Aluminum, Antimony, Arsenic, Barium, Beryllium, Cadmium, Calcium, Chromium, Cobalt, Copper, Iron, Lead, Magnesium, Manganese, Molybdenum, Nickel, Potassium, Selenium, Silver, Sodium, Thallium, Vanadium, and Zinc	Water/Solid
7196	Chromium VI	Water/Solid
1664	Total Petroleum Hydrocarbons (TPH)	Water/Solid
8011	1,2 Dibromoehtane and 1,2 Dibromo-3-Chloropropane by Microestraction and Gas Chromatograph	Water/Solid
8015M	Total Petroleum Hydrocarbons – Gasoline Range Organics (GRO) Diesel Range Organics (DRO)	Water/Solid
9012	Cyanide	Water/Solid

Acceptance for use for parameters not identified on the table will be determined by Navy project personnel.

The laboratory should notify NFESC if there are parameters not presented on Table 1 that the laboratory expects to run on a routine basis in support of Navy installation restoration projects. In these circumstances the laboratory's capability to run the tests will be reviewed and the table will be modified accordingly.

Questions concerning the information provided should be directed to the NFESC IR QA Program coordinator, Ms. Patricia Moreno at (805) 982-1659, or via email at pati.moreno@navy.mil.

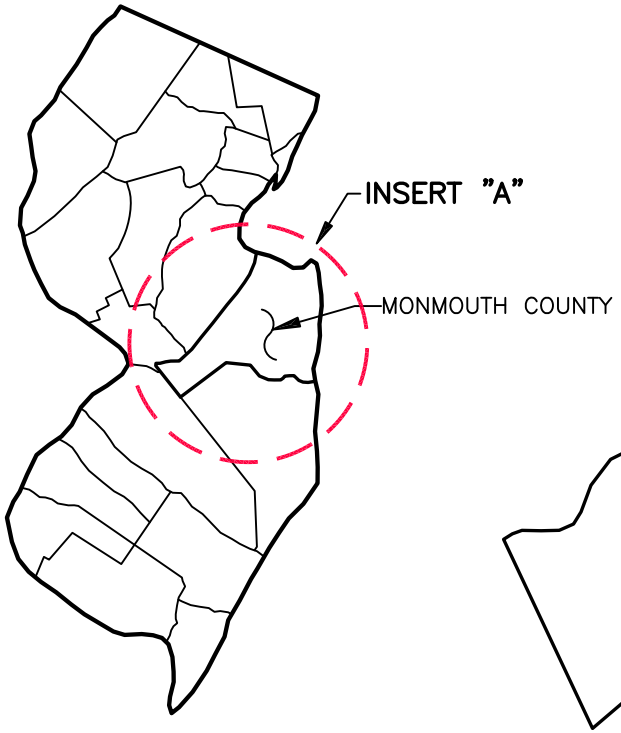
Sincerely,

Robert J. Kratzke
Supervisor, Consultation/Information
Management Branch

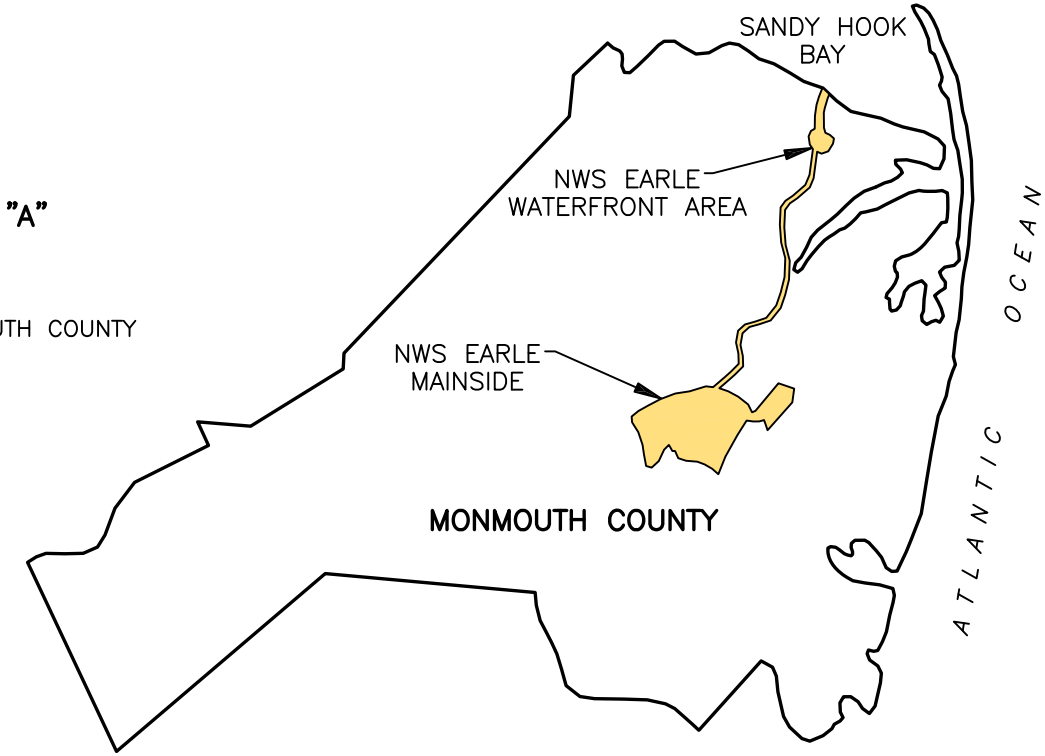
APPENDIX C

FIGURES

112G02091/0320/112G02091CM02.DWG 07/22/10 MKB



STATE OF NEW JERSEY



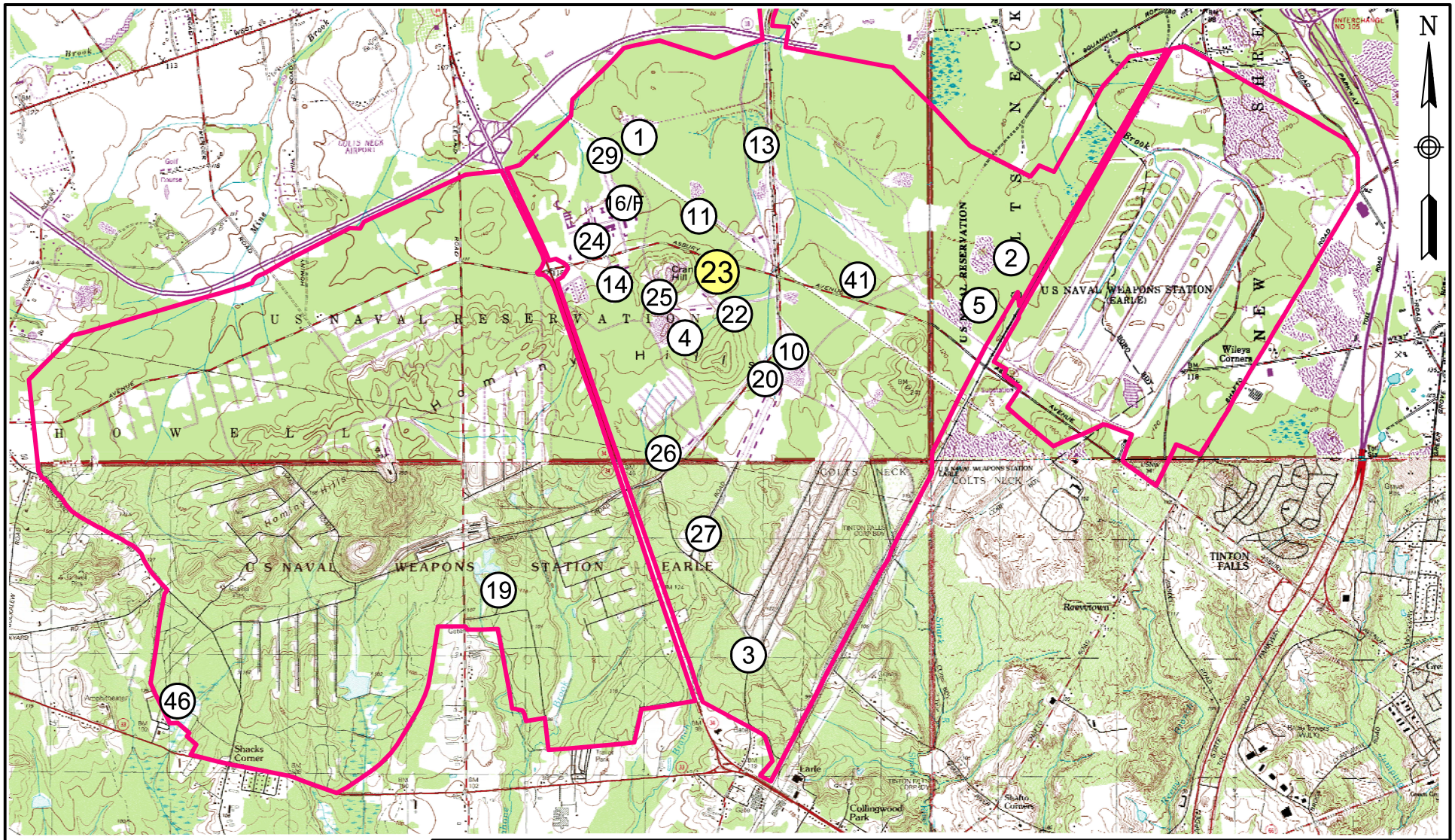
INSERT "A"

TETRA TECHNUS, INC.

SITE LOCATION MAP
NAVAL WEAPONS STATION EARLE
COLTS NECK, NEW JERSEY

SCALE	NOT TO SCALE
FILE	112G02091CM02
REV 0	DATE 07/22/10
FIGURE NUMBER	FIGURE 10–1

112G02091\0320\112G02091CM03.DWG 07/22/10 MKB



0 4,000 8000

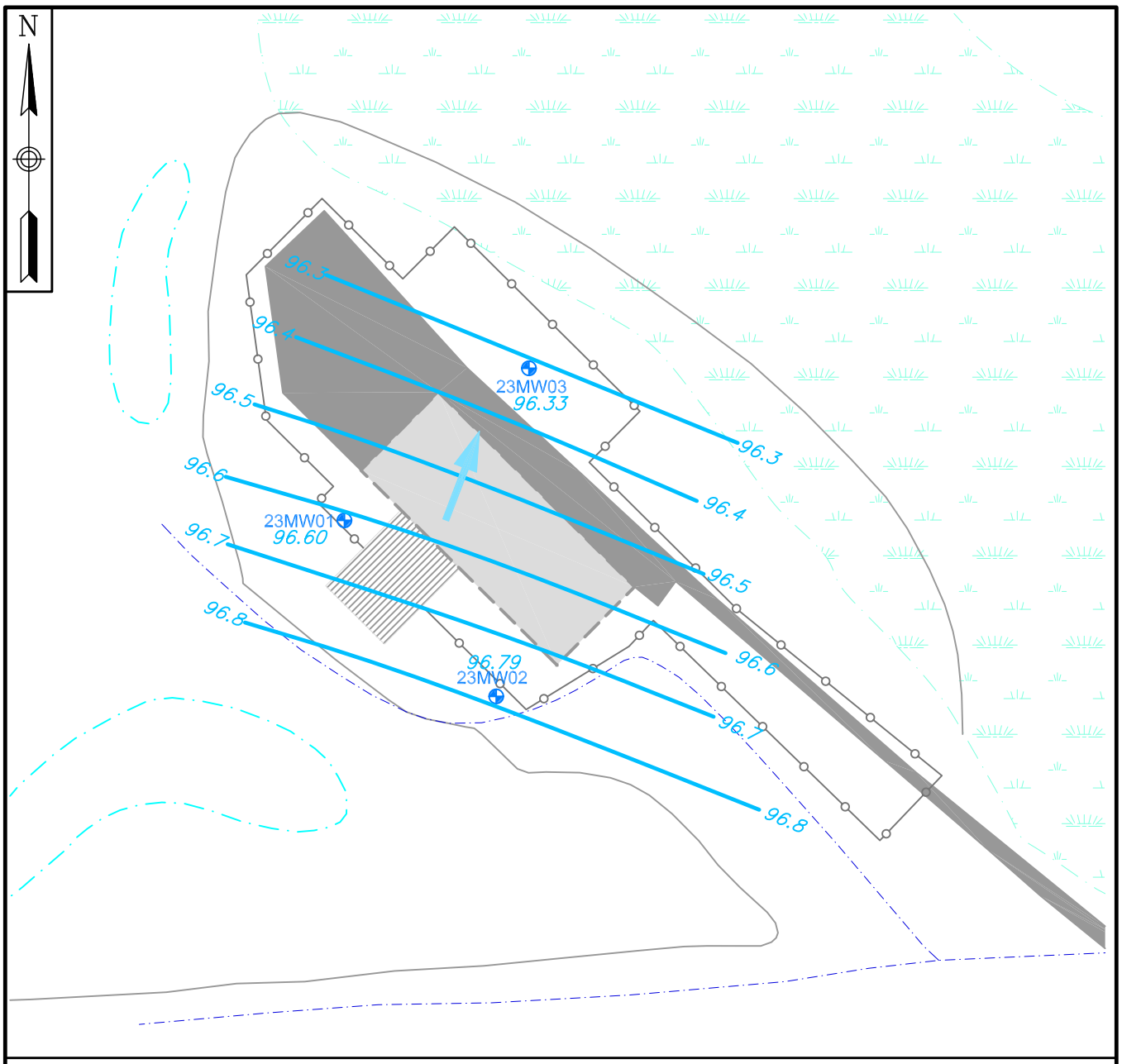
SCALE IN FEET

TETRA TECH NUS, INC.

SITE 23 LOCATION MAP
NAVAL WEAPONS STATION EARLE
COLTS NECK, NEW JERSEY

SCALE	AS NOTED
FILE	112G02091CM03
REV 0	DATE 07/22/10
FIGURE NUMBER	FIGURE 10–2

112G02091\0320\112G02091GM02.DWG 05/24/10 MKB



LEGEND

- MONITORING WELL
- AREA EXCAVATED
- FOOTPRINT OF FORMER BUILDING D-5 (REMOVED 2003)
- ASPHALT PAVING
- 96 — GROUNDWATER CONTOUR (FEET, MSL) (DASHED WHERE INFERRED)
- 96.46 GROUNDWATER ELEVATION (FEET, MSL)
- GROUNDWATER FLOW DIRECTION

SOURCE:
FWENC, 1997. CLOSE-OUT REPORT, REMOVAL ACTIONS AT SITES 22, 23, AND 27, NAVAL WEAPONS STATION EARLE, COLTS NECK, NEW JERSEY LANGHORNE, PENNSYLVANIA. FEBRUARY 14

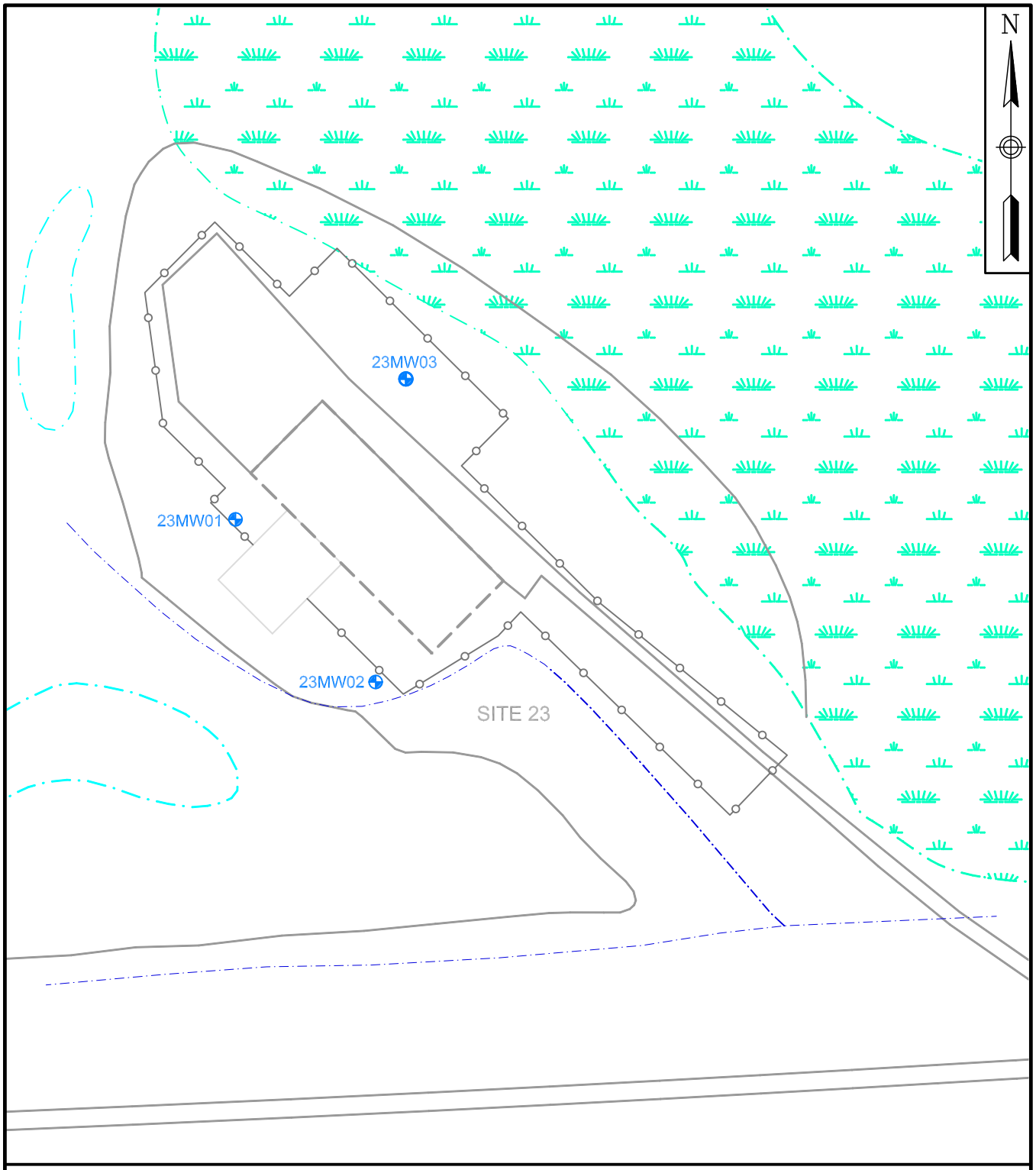
0 80 160
SCALE IN FEET

TETRA TECH NUS, INC.

SITE LAYOUT MAP
SITE 23
NAVAL WEAPONS STATION EARLE
COLTS NECK, NEW JERSEY

SCALE	AS NOTED
FILE	112G02091GM02
REV	0
DATE	05/24/10
FIGURE NUMBER	FIGURE 10-3

112G02091\0320\112G02091GM01.DWG 05/24/10 MKB



LEGEND

MONITORING WELL

WETLANDS

0 100 200

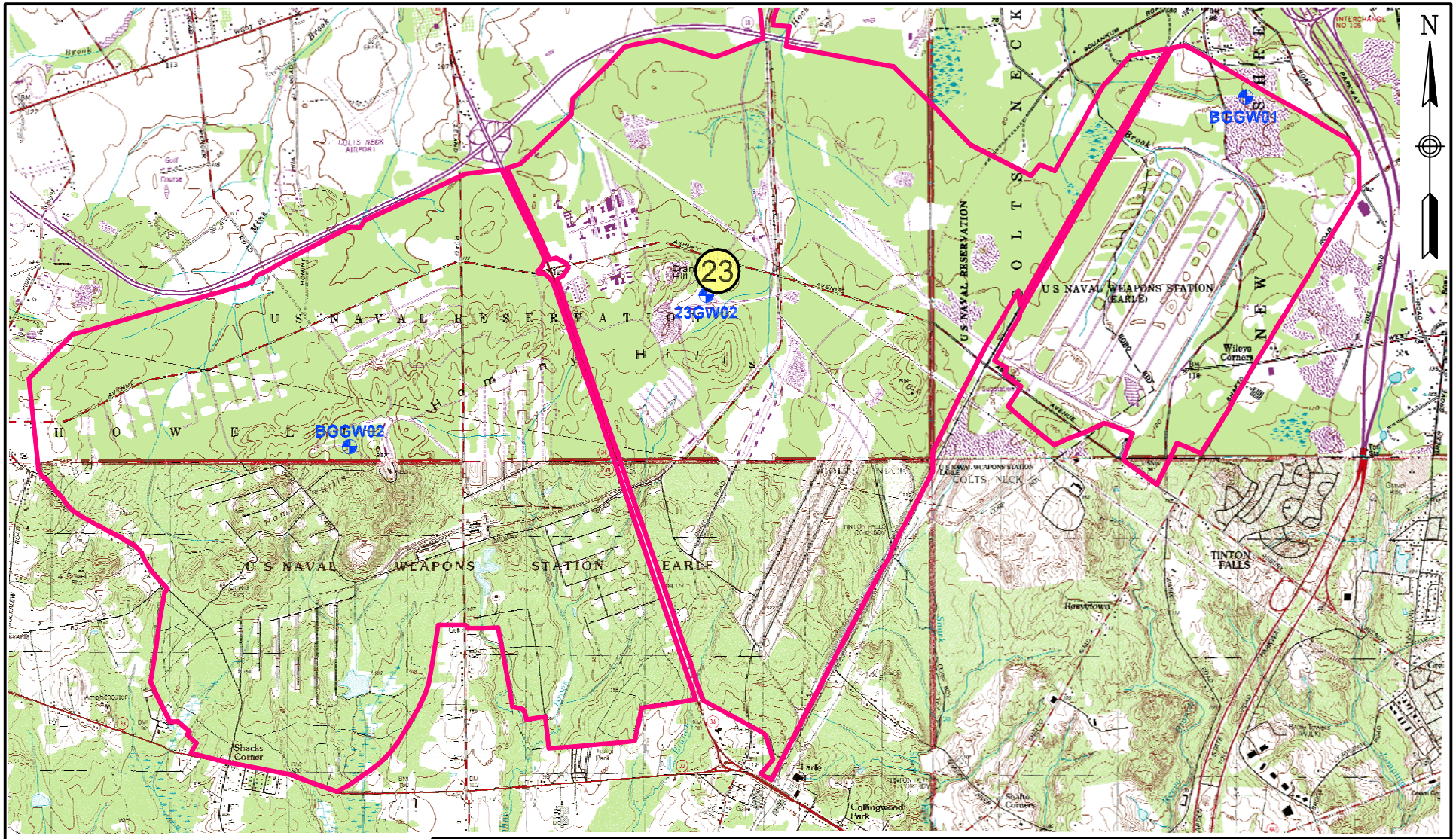
SCALE IN FEET

TETRA TECHNUS, INC.

MONITORING WELL LOCATION MAP
SITE 23
NAVAL WEAPONS STATION EARLE
COLTS NECK, NEW JERSEY

SCALE	AS NOTED
FILE	112G02091GM01
REV	0
DATE	05/24/10
FIGURE NUMBER	FIGURE 14–1

112G02091\0310\112G02091CM02-2.DWG 07/07/10 MKB



LEGEND

BACKGROUND GROUNDWATER SAMPLE LOCATION

0 4,000 8000

SCALE IN FEET

TETRA TECH NUS, INC.

BACKGROUND MONITORING WELLS
NAVAL WEAPONS STATION EARLE
COLTS NECK, NEW JERSEY

SCALE	AS NOTED
FILE	112G02091CM02-2
REV	0
DATE	07/07/10
FIGURE NUMBER	FIGURE 14-2