# Comprehensive Long-term Environmental Action Navy

N00639.AR.000845
MILLINGTON SUPPACT
5090.3a

CONTRACT NUMBER N62472-04-D-0055
CONTRACT NUMBER N62472-03-D-0057

Rev. 1
12/09

# Sampling and Analysis Plan (Field Sampling Plan and Quality Assurance Project Plan) for Small Arms Range Site Inspections

## Naval Support Activity Mid-South
## Millington, Tennessee

## Contract Task Order 0107
## Contract Task Order F275

## December 2009

NAVFAC
Naval Facilities Engineering Command
Midwest
201 Decatur Avenue, Building 1A
Great Lakes, Illinois 60088

TETRA TECH NUS, Inc.

**SAP Worksheet #1 – Title and Approval Page**

**(UFP-QAPP Manual Section 2.1)**

# SAMPLING AND ANALYSIS PLAN
## (Field Sampling Plan and Quality Assurance Project Plan)
## for
## Small Arms Range Site Inspections
## Naval Support Activity Mid-South
## Millington, Tennessee

December 2009

**Prepared for:**
Naval Facilities Engineering Command Midwest
201 Decatur Avenue, Building 1-A
Great Lakes, Illinois 60088-2801

**Prepared by:**
Tetra Tech NUS, Inc.
661 Andersen Drive
Foster Plaza 7
Pittsburgh, Pennsylvania 15220

**Prepared under:**
Contract Number N62467-04-D-0055
Contract Task Order 0107 and
Contract Number N62472-03-D-0057
Contract Task Order F275

**Review Signatures**

16 DEC 09
LAWSON ANDERSON — DATE
PROJECT MANAGER
TETRA TECH NUS, INC.
MEMPHIS, TENNESSEE

16 DEC 09
KELLY CARPER — DATE
CLEAN QA MANAGER
TETRA TECH NUS, INC.
PITTSBURGH, PENNSYLVANIA

**Approval Signatures**

HICKEY.HOWARD.M.1287981070
HOWARD HICKEY — DATE
REMEDIAL PROJECT MANAGER
NAVFAC MW

BOWERS.KENNETH.A.1230092474
KEN BOWERS — DATE
NAVY CHEMIST
NAVFAC OAD

FOR 12/16/09
DAVID CRISWELL — DATE
BRAC ENVIRONMENTAL COORDINATOR
BRAC PMO

TETRA TECH

PITT-2-10-028

February 17, 2010

Project No. 112G01506

Mr. David Criswell
U.S. Navy
BRAC Program Management Office Southeast
4130 Faber Place Drive, Suite 202
North Charleston, South Carolina 29405

Subject: CLEAN Contract Number N62467-04-D-0055
Contract Task Order 0107

Reference: Final Sampling and Analysis Plan (Field Sampling Plan and Quality Assurance Project Plan) for Small Arms Range Site Inspections
Naval Support Activity Mid-South
Millington, Tennessee

Dear Mr. Criswell:

Enclosed are one hard copy and two CDs of the subject Sampling and Analysis Plan (SAP) in Unified Federal Policy (UFP) format. Per your request, hard copies have been forwarded on behalf of the Navy to the Tennessee Department of Environment and Conservation (TDEC) and U.S. Environmental Protection Agency (USEPA) Region 4.

This UFP SAP has been prepared under two separate CLEAN contracts because the Seven Small Arms Range sites at NSA Mid-South are located on both active (E,RN-funded) and closed/transferred (BRAC-funded) portions of the installation. Copies of this UFP SAP have also been provided to Mr. Howard Hickey at Naval Facilities Engineering Command (NAVFAC) Midwest under the E,RN-funded contract.

Please contact me (email lawson.anderson@tetratech.com) or Mr. Geoff Pope (email geoff.pope@tetratech.com) at (901) 523-9500 regarding any questions or comments.

Sincerely,

Lawson Anderson

Lawson Anderson, CHMM
Task Order Manager

LMA/mlg
Enclosures (3)

cc: Howard Hickey, NAVFAC Midwest (letter, 2 hard copies, 2 CDs)
Jim Heide, NSA Mid-South (letter, 1 hard copy, 2 CDs)
Charlie Burroughs, TDEC (letter, 2 hard copies)
Greg Fraley, USEPA Region 4 (letter, 1 hard copy)
Chris Pike, Tetra Tech (letter, 1 CD)
John Trepanowski, Tetra Tech (letter, 1 CD)
Ralph Basinski, Tetra Tech (letter only)
Geoff Pope, Tetra Tech (letter, 4 hard copies)
Glenn Wagner, Tetra Tech (letter, 1 CD)
File copy – CTO 0107 (letter only)

Tetra Tech NUS, Inc.
661 Andersen Drive, Pittsburgh, PA 15220-2745
Tel 412.921.7090 Fax 412.921.4040 www.ttnus.com

TETRA TECH

PITT-02-10-027

February 17, 2010

Project No. 112G01506

Mr. Greg Fraley
U.S. Environmental Protection Agency Region 4
Atlanta Federal Center
61 Forsyth Street
Atlanta, Georgia 30303-8960

Subject: CLEAN Contract Number N62467-04-D-0055
Contract Task Order 0107

Reference: Final Sampling and Analysis Plan (Field Sampling Plan and Quality Assurance Project Plan) for Small Arms Range Site Inspections
Naval Support Activity Mid-South
Millington, Tennessee

Dear Mr. Fraley:

On behalf of the Department of the Navy, Tetra Tech is forwarding one hard copy of the subject Sampling and Analysis Plan (SAP) in Unified Federal Policy (UFP) format. This Final UFP SAP has been prepared under two separate Navy CLEAN contracts because the Seven Small Arms Range sites at NSA Mid-South are located on both active (E,RN-funded) and closed/transferred (BRAC-funded) portions of the installation. Copies of this Final UFP SAP have also been provided to Mr. Charlie Burroughs of the Tennessee Department of Environment and Conservation under the E,RN-funded contract.

Please contact me (email lawson.anderson@tetratech.com) or Mr. Geoff Pope (email geoff.pope@tetratech.com) at (901) 523-9500 regarding any questions or comments.

Sincerely,

Lawson Anderson

Lawson Anderson, CHMM
Task Order Manager

LMA/mlg
Enclosure

cc: Howard Hickey, NAVFAC Midwest
David Criswell, BRAC PMO Southeast
Jim Heide, NSA Mid-South
Charlie Burroughs, TDEC
Chris Pike, Tetra Tech
John Trepanowski, Tetra Tech
Ralph Basinski, Tetra Tech
Geoff Pope, Tetra Tech
Glenn Wagner, Tetra Tech
File copy – CTO 0107

Tetra Tech NUS, Inc.
661 Andersen Drive, Pittsburgh, PA 15220-2745
Tel 412.921.7090 Fax 412.921.4040 www.ttnus.com

# EXECUTIVE SUMMARY

Tetra Tech NUS, Inc. (Tetra Tech) has prepared this Sampling and Analysis Plan (SAP) (Field Sampling Plan (FSP) and Quality Assurance Project Plan (QAPP)) for Small Arms Range Site Inspections (SIs) under two Comprehensive Long-Term Environmental Action Navy (CLEAN) Contracts. CLEAN Contract No. N62467-04-D-0055, Contract Task Order (CTO) 0107 (i.e., "the BRAC CTO") includes the two Munitions Response Program (MRP)-eligible sites on former Navy property - the Rifle Range and Horse Stables Skeet Range #2, which total 43.7 acres. CLEAN Contract No. N62472-03-D-0057, CTO F275 (i.e., "the ER,N CTO") includes the five MRP sites within the current installation boundary, Pistol Range, Trap/Skeet Ranges #1 and #2, Aircraft Firing Range, and Horse Stables Skeet Range #1, which total 91.8 acres.

Naval Support Activity (NSA) Mid-South is located in the Town of Millington in Shelby County, Tennessee, approximately 20 miles north of Memphis, 180 miles west of Nashville, and 7 miles east of the Mississippi River. It encompasses 1,600 acres and serves as one of the largest inland Navy installations in the world. As of April 2002, the base employed 1,100 people including enlisted personnel, officer personnel, and civilians. Seven Navy MRP-eligible sites were identified at NSA Mid-South during a Preliminary Assessment (PA) completed by Malcolm-Pirnie, Inc., in August 2005, including Pistol Range, Rifle Range, Aircraft Firing Range, Trap/Skeet Ranges #1 and #2, Horse Stables Skeet Range #1, and Horse Stables Skeet Range #2. Trap/Skeet Range #2 was active during the PA and was not included; therefore, only six sites were included in the PA Report (Malcolm Pirnie, 2005). Trap/Skeet Range #2 was later inactivated and was then added to the list of inactive ranges, bringing the total to seven.

As a result of the Navy's munitions training activities, Munitions Constituents (MC) may be present at the seven sites located at NSA Mid-South. Munitions and Explosives of Concern (MEC) are not suspected to be present. The term MEC includes Discarded Military Munitions (DMM), Unexploded Ordnance (UXO), and MC in high enough concentrations to pose an explosive hazard. The Navy is following the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) of 1980 process for the investigation of these sites.

The Department of Defense (DoD) has established a separate program to address closed military ranges known as the Munitions Response Program (MRP), and is following the CERCLA process for the MRP. The CERCLA process is similar to the Resource Conservation and Recovery Act (RCRA) corrective action process. Munitions SIs at these seven inactive ranges are being conducted under CERCLA. These sites were not identified when the most recent RCRA Permit for NSA Mid-South, which became effective on April 24, 1998, was issued. Since then, Malcolm Pirnie conducted the PA, which correlates with the information gathering/initial evaluation phase of a RCRA Facility Assessment (RFA). The results

and recommendations of the PA were used to develop the field program for the SIs, which is described in this Unified Federal Policy (UFP) SAP. The CERCLA SIs correlate with the sampling phase of the RFA. The need for advancement to the CERCLA Remedial Investigation (RI) Phase, which correlates with the RCRA Facility Investigation (RFI) phase, for one or more of the MRP sites will be determined based on the results of the SIs.

The August 2005 PA report identified six "other than operational" small arms and trap/skeet ranges or Munitions Response Areas/Sites (MRA/Ss) at NSA Mid-South requiring further investigation. Closed, transferred, and transferring military ranges and sites not located on an operational range are considered other than operational. The MRA/Ss described in the NSA Mid-South PA Report are indicated on Figure ES.1. The PA used five primary sources of information to support the facility data collection effort, including historical archives, personal interviews, installation data repositories (including the Administrative Record), visual surveys, and off-facility data sources and repositories such as local libraries and museums.

The primary objective of these MRP MRA/Ss SIs, is to determine if further CERCLA response actions or RIs are appropriate for any of the sites identified in the PA. The SIs consider the background information provided in the PA and collect supplemental site-specific environmental data to further define the nature and extent of MC at the seven sites discussed above. The SIs are a secondary component of the CERCLA site evaluation process and are not intended to be a full-scale study of the nature and extent of contamination. The SIs will consist of focused sampling and analysis programs to identify potential contaminants based on information in the PA and various Navy and regulatory guidance for investigation of small arms and skeet ranges.

The information provided in the worksheets included in this SAP was developed based on the results of the project scoping meetings attended by the planning team, which consists of representatives of the Navy, United States Environmental Protection Agency (USEPA), United States Geological Survey (USGS) Tennessee Department of Environment and Conservation (TDEC), EnSafe, Inc., and Tetra Tech (see Worksheet #9 for attendees). Worksheet #10 contains the problem statement and general Conceptual Site Models (CSMs) for the skeet ranges and small arms ranges. The CSMs were used as the basis for the development of the Data Quality Objectives (DQOs), which are contained in Worksheet #11. The remaining worksheets describe the SI sampling, analytical, and data evaluation procedures including quality requirements.

**SAP Worksheets** **Page**

SAP Worksheet #1 -- Title and Approval Page ..... 1
SAP Worksheet #2 -- SAP Identifying Information ..... 10
SAP Worksheet #3 -- Distribution List ..... 13
SAP Worksheet #4 -- Project Personnel Sign-Off Sheet ..... 15
SAP Worksheet #5 -- Project Organizational Chart ..... 16
SAP Worksheet #6 -- Communication Pathways ..... 17
SAP Worksheet #7 -- Personnel Responsibilities and Qualifications Table ..... 19
SAP Worksheet #8 -- Special Personnel Training Requirements Table ..... 24
SAP Worksheet #9 -- Project Scoping Session Participants Sheet ..... 25
SAP Worksheet #10 – Conceptual Site Model and Problem Definition ..... 29
SAP Worksheet #11 -- Project Quality Objectives/Systematic Planning Process ..... 39
SAP Worksheet #12 -- Measurement Performance Criteria Table - Field Quality Control Samples ..... 44
SAP Worksheet #13 -- Secondary Data Criteria and Limitations Table ..... 45
SAP Worksheet #14 -- Summary of Project Tasks ..... 46
SAP Worksheet #15 -- Reference Limits and Evaluation Table ..... 50
SAP Worksheet #16 -- Project Schedule / Timeline Table (optional format) ..... 53
SAP Worksheet #17 -- Sampling Design and Rationale ..... 55
SAP Worksheet #18.1 -- Sampling Locations and Methods/SOP Requirements Table ..... 60
SAP Worksheet #18.2 -- Sampling Locations and Methods/SOP Requirements Table ..... 64
SAP Worksheet #18.3 -- Sampling Locations and Methods/SOP Requirements Table ..... 68
SAP Worksheet #18.4 -- Sampling Locations and Methods/SOP Requirements Table ..... 72
SAP Worksheet #18.5-- Sampling Locations and Methods/SOP Requirements Table ..... 76
SAP Worksheet #18.6-- Sampling Locations and Methods/SOP Requirements Table ..... 80
SAP Worksheet #19 -- Analytical SOP Requirements Table - Empirical Laboratories ..... 84
SAP Worksheet #19 -- Analytical SOP Requirements Table (GPL Laboratories) ..... 85
SAP Worksheet #20 -- Field Quality Control Sample Summary Table ..... 86
SAP Worksheet #21 -- Project Sampling SOP References Table ..... 88
SAP Worksheet #22 -- Field Equipment Calibration, Maintenance, Testing, and Inspection Table ..... 89
SAP Worksheet #23 -- Analytical SOP References Table - Empirical Laboratories ..... 90
SAP Worksheet #23 -- Analytical SOP References Table - GPL Laboratories ..... 92
SAP Worksheet #24 -- Analytical Instrument Calibration Table - Empirical Laboratories ..... 93
SAP Worksheet #24 -- Analytical Instrument Calibration Table - GPL Laboratories ..... 95
SAP Worksheet #25 -- Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table – Empirical Laboratories ..... 96
SAP Worksheet #25 -- Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table – GPL Laboratories ..... 98
SAP Worksheet #26 -- Sample Handling System ..... 99
SAP Worksheet #27 – Sample Custody Requirements Table ..... 100
SAP Worksheet #28 -- Laboratory QC Samples Table - Empirical Laboratories ..... 103
SAP Worksheet #28 -- Laboratory QC Samples Table - GPL Laboratories ..... 109
SAP Worksheet #29 -- Project Documents and Records Table ..... 112
SAP Worksheet #30 -- Analytical Services Table ..... 113
SAP Worksheet #31 -- Planned Project Assessments Table ..... 114
SAP Worksheet #32 -- Assessment Findings and Corrective Action Responses ..... 115
SAP Worksheet #33 -- QA Management Reports Table ..... 116
SAP Worksheet #34 -- Verification (Step I) Process Table ..... 117
SAP Worksheet #35 -- Validation (Steps IIa and IIb) Process Table ..... 118
SAP Worksheet #36 –Analytical Data Validation (Steps IIa and IIb) Summary Table ..... 121
SAP Worksheet #37 -- Usability Assessment ..... 122

**Figures**

| | |
|---|---|
| ES.1 | Site Map |
| 10.1 | Range/Site Details – Pistol Range |
| 10.2 | Range/Site Details – Rifle Range |
| 10.3 | Range/Site Details – Aircraft Firing Range |
| 10.4 | Range/Site Details – Trap/Skeet Ranges #1 and #2 |
| 10.5 | Range/Site Details – Horse Stables Skeet Range #1 |
| 10.6 | Range/Site Details – Horse Stables Skeet Range #2 |
| 17.1 | Proposed Sample Locations – Pistol Range |
| 17.2 | Proposed Sample Locations – Rifle Range |
| 17.3 | Proposed Sample Locations – Aircraft Firing Range |
| 17.4 | Proposed Sample Locations – Trap/Skeet Ranges #1 and #2 |
| 17.5 | Proposed Sample Locations – Horse Stables Skeet Range #1 |
| 17.6 | Proposed Sample Locations – Horse Stables Skeet Range #2 |

**Appendices**

| | |
|---|---|
| A | Standard Operating Procedures |
| B | Laboratory Standard Operating Procedures |
| C | Field Forms |

# ACRONYMS

| | |
|---|---|
| AOC | Area of Concern |
| AR | Administrative Record |
| ARAR | Applicable or Relevant and Appropriate Requirement |
| BCT | Base Cleanup Team or BRAC Cleanup Team |
| BEC | BRAC Environmental Coordinator |
| BERA | Baseline Environmental Risk Assessment |
| bgs | Below ground surface |
| BRAC | Base Realignment and Closure |
| CA | Corrective Action |
| CCB | Continuing Calibration Blank |
| CCC | Calibration Check Compound |
| CCV | Continuing Calibration Verification |
| CEC | Cation exchange capacity |
| CERCLA | Comprehensive Environmental Response, Compensation, and Liability Act |
| CHMM | Certified Hazardous Materials Manager |
| CIH | Certified Industrial Hygienist |
| CLEAN | Comprehensive Long-Term Environmental Action Navy |
| COC | Contaminant of concern |
| COPC | Chemical of potential concern |
| CSM | Conceptual Site Model |
| CSP | Certified Safety Professional |
| CTO | Contract Task Order |
| CWAP | Comprehensive Work Approval Process |
| DFTPP | Decafluorotriphenylphosphine |
| DoD | Department of Defense |
| DMM | Discarded Military Munitions |
| DPT | Direct-push Technology |
| DQI | Data quality indicator |
| DQO | Data Quality Objective |
| DVM | Data Validation Manager |
| E/A&H | Ensafe/Allen and Hoshall |
| EISOPQAM | Environmental Investigations Standard Operating Procedures and Quality Assurance Manual |
| ER,N | Environmental Restoration, Navy |
| ESSL | Ecological Soil Screening Level |

| | |
|---|---|
| FOL | Field Operations Leader |
| FTMR | Field Task Modification Request |
| GC/MS | Gas chromatography-mass spectrometry |
| GIS | Geographical information system |
| GPS | Global positioning system |
| HASP | Health and Safety Plan |
| HP | High-Pressure |
| HPLC | High-Pressure Liquid Chromatography |
| HSM | Health and Safety Manager |
| ICB | Initial Calibration Blank |
| ICP | Inductively Coupled Plasma |
| ICV | Initial Calibration Verification |
| IDW | Investigation-derived waste |
| IR | Installation Restoration |
| IS | Internal Standard |
| LCS | Laboratory Control Sample |
| LCSD | Laboratory Control Sample Duplicate |
| LIMS | Laboratory information management system |
| MC | Munitions Constituents |
| MDL | Method Detection Limit |
| MEC | Munitions and Explosives of Concern |
| mg/kg | Milligram per kilogram |
| MIS | Multi-incremental Sampling |
| mm | Millimeter |
| MPC | Measurement Performance Criterion |
| MRP | Munitions Response Program |
| MRA/S | Munitions Response Areas or Sites |
| MS/MSD | Matrix spike/matrix spike duplicate |
| NAVFAC MW | Naval Facilities Engineering Command Midwest |
| NC | Nitrocellulose |
| NCP | National Oil and Hazardous Substances Pollution Contingency Plan |
| NELAP | National Environmental Laboratory Accreditation Program |
| NFA | No Further Action |
| NFESC | Naval Facilities Engineering Service Center |
| NG | Nitroglycerin |
| NOSSA | Naval Ordnance Safety and Security Activity |
| NSA | Naval Support Activity |

| | |
|---|---|
| ORNL | Oak Ridge National Laboratory |
| PA | Preliminary Assessment |
| PAH | Polynuclear aromatic hydrocarbon |
| PAL | Project Action Limit |
| PA | Preliminary Assessment |
| PM | Project Manager |
| PMO | Project Management Office |
| POC | Point of Contact |
| PPE | Personal protective equipment |
| PPV | Public-Private Venture |
| PT | Proficiency testing |
| QA | Quality assurance |
| QAM | Quality Assurance Manager |
| QAO | Quality Assurance Officer |
| QAPP | Quality Assurance Project Plan |
| QC | Quality control |
| QL | Quantitation limit |
| %R | Percent Recovery |
| %RSD | Percent relative standard deviation |
| RF | Response factor |
| RCRA | Resource Conservation and Recovery Act of 1976 |
| RFA | RCRA Facility Assessment |
| RFI | RCRA Facility Investigation |
| RI | Remedial Investigation |
| RPD | Relative Percent Difference |
| RPM | Remedial Project Manager |
| RSLs | USEPA Residential Regional Screening Levels |
| SAP | Sampling and Analysis Plan |
| SDG | Sample Delivery Group |
| SE | Southeast |
| SIs | Site Investigations |
| SIM | Selective Ion Monitoring |
| SOP | Standard Operating Procedure |
| SPCC | System Performance Check Compound |
| SQL | Structured Query Language |
| SSO | Site Safety Officer |
| SWMU | Solid Waste Management Unit |

| | |
|---|---|
| TDEC | Tennessee Department of Environment and Conservation |
| TOC | Total organic carbon |
| TOM | Task Order Manager |
| Tetra Tech | Tetra Tech NUS, Inc. |
| UFP | Uniform Federal Policy |
| USEPA | United States Environmental Protection Agency |
| USGS | United States Geological Survey |
| UXO | Unexploded ordnance |
| XRF | X-ray fluorescence |

**SAP Worksheet #2 -- SAP Identifying Information**

| | |
|---|---|
| **Site Name/Number:** | **NSA Mid-South Millington, Tennessee** |
| **Operable Unit:** | **N/A** |
| **Contractor Name:** | **Tetra Tech NUS, Inc.** |
| **Contract Number:** | **N62467-04-D-0055/N62472-03-D-0057** |
| **Contract Title:** | **CLEAN IV** |
| **Work Assignment Number (optional):** | **CTO 0107/F275** |

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

2. Identify regulatory program: Munitions Response Program (MRP); National Oil and Hazardous Substances Pollution Contingency Plan (NCP); Comprehensive Environmental Response Compensation and Liability (CERCLA); Resource Conservation and Recovery Act (RCRA)

3. This SAP is a project-specific SAP.

4. List dates of scoping sessions that were held:

| Scoping Session | Date |
|---|---|
| Kick-off meeting for development of conceptual site models (CSMs) and Data quality objectives (DQOs) | September 14 and 15, 2008 |
| Scoping Meeting to review CSMs and DQOs | February 4 and 5, 2009 |

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

| Title | Date |
|---|---|
| Preliminary Assessment (PA) (Malcolm Pirnie) | August 2005 |
| Hydrogeology and Ground-Water Quality at Naval Support Activity Memphis, Millington, Tennessee (Carmichael, Parks, Kingsbury, and Ladd) | 1997 |
| Hydrogeology of Post-Wilcox Group Stratigraphic Units in the Area of the Naval Air Station Memphis, Near Millington, Tennessee (Kingsbury and Carmichael) | 1995 |
| Groundwater Monitoring Well Management Plan, Revision 8 (EnSafe) | 2005 |

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

United States Environmental Protection Agency (USEPA) Region 4 – Regulatory Oversight; TDEC – Regulatory Oversight; and the City of Millington.

7. Lead organization: Naval Facilities Engineering Command Midwest (NAVFAC MW) – Property Owner (ER,N sites); Navy Base Realignment and Closure (BRAC) Program Management Office (PMO) Southeast (SE) – Property Owner (BRAC sites)

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:

| UFP-SAP Worksheet No. | Required Information | Crosswalk to Related Information |
|---|---|---|
| **A. Project Management** | | |
| *Documentation* | | |
| 1 | Title and Approval Page | NA |
| 2 | Table of Contents<br>SAP Identifying Information | NA |
| 3 | Distribution List | NA |
| 4 | Project Personnel Sign-Off Sheet | NA |
| *Project Organization* | | |
| 5 | Project Organizational Chart | NA |
| 6 | Communication Pathways | NA |
| 7 | Personnel Responsibilities and Qualifications Table | NA |
| 8 | Special Personnel Training Requirements Table | NA |
| *Project Planning/ Problem Definition* | | |
| 9 | Project Planning Session Documentation (including Data Needs tables)<br>Project Scoping Session Participants Sheet | NA |
| 10 | Problem Definition, Site History, and Background.<br>Site Maps (historical and present) | NA |
| 11 | Site-Specific Project Quality Objectives | NA |
| 12 | Measurement Performance Criteria Table | NA |
| 13 | Sources of Secondary Data and Information<br>Secondary Data Criteria and Limitations Table | NA |
| 14 | Summary of Project Tasks | NA |
| 15 | Reference Limits and Evaluation Table | NA |
| 16 | Project Schedule/Timeline Table | NA |
| **B. Measurement Data Acquisition** | | |
| *Sampling Tasks* | | |
| 17 | Sampling Design and Rationale | NA |
| 18 | Sampling Locations and Methods/SOP Requirements Table<br>Sample Location Map(s) | NA |
| 19 | Analytical Methods/SOP Requirements Table | NA |
| 20 | Field Quality Control Sample Summary Table | NA |
| 21 | Project Sampling SOP References Table<br>Sampling SOPs | NA |
| 22 | Field Equipment Calibration, Maintenance, Testing, and Inspection Table | NA |
| *Analytical Tasks* | | |
| 23 | Analytical SOPs<br>Analytical SOP References Table | NA |
| 24 | Analytical Instrument Calibration Table | NA |
| 25 | Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table | NA |
| *Sample Collection* | | |
| 26 | Sample Handling System, Documentation Collection, Tracking, Archiving and Disposal<br>Sample Handling Flow Diagram | NA |
| 27 | Sample Custody Requirements, Procedures/SOPs Sample Container Identification<br>Example Chain-of-Custody Form and Seal | NA |

| UFP-SAP Worksheet No. | Required Information | Crosswalk to Related Information |
|---|---|---|
| *Quality Control Samples* | | |
| 28 | QC Samples Table<br>Screening/Confirmatory Analysis Decision Tree | NA |
| *Data Management Tasks* | | |
| 29 | Project Documents and Records Table | NA |
| 30 | Analytical Services Table<br>Analytical and Data Management SOPs | NA |
| **C. Assessment Oversight** | | |
| 31 | Planned Project Assessments Table<br>Audit Checklists | NA |
| 32 | Assessment Findings and Corrective Action Responses Table | NA |
| 33 | QA Management Reports Table | NA |
| **D. Data Review** | | |
| 34 | Verification (Step I) Process Table | NA |
| 35 | Validation (Steps IIa and IIb) Process Table | NA |
| 36 | Validation (Steps IIa and IIb) Summary Table | NA |
| 37 | Usability Assessment | NA |

**SAP Worksheet #3 – Distribution List**

(UFP-QAPP Manual Section 2.3.1)

| Name of SAP Recipient | Title/Role | Organization | Telephone Number (Optional) | E-Mail Address or Mailing Address | Document Control Number (Optional) |
|---|---|---|---|---|---|
| Howard Hickey | Remedial Project Manager (RPM) | NAVFAC MW | 847-688-2600 x243 | howard.hickey@navy.mil | NA |
| David Criswell | BRAC Environmental Coordinator (BEC) | BRAC PMO SE | 843-743-2130 | david.criswell@navy.mil | NA |
| Jim Heide | Installation Restoration (IR) Coordinator (Acting) | NSA Mid-South | 901-874-5461 | jim.heide@navy.mil | NA |
| Ursula Robinson | Assistant IR Coordinator | NSA Mid-South | 901-874-5918 | ursula.robinson@navy.mil | NA |
| Bonnie Capito | Administrative Record/Librarian | NAVFAC Atlantic | 757 322-4785 | bonnie.capito@navy.mil | NA |
| Lawson Anderson | Project Manager (PM) | Tetra Tech | 901-523-9500 | lawson.anderson@tetratech.com | NA |
| Geoff Pope | Deputy PM | Tetra Tech | 901-523-9500 | geoff.pope@tetratech.com | NA |
| Kelly Carper | CLEAN (PMO) – BRAC Site Quality Assurance Manager (QAM) | Tetra Tech | 412-921-7273 | kelly.carper@tetratech.com | NA |

| Name of SAP Recipient | Title/Role | Organization | Telephone Number (Optional) | E-Mail Address or Mailing Address | Document Control Number (Optional) |
|---|---|---|---|---|---|
| Dr. Tom Johnston | CLEAN PMO – ER,N Site QAM | Tetra Tech | 412-921-8615 | tom.johnston@tetratech.com | NA |
| John Trepanowski | CLEAN Program Manager - ER,N Sites | Tetra Tech | 610-491-9688 | john.trepanowski@tetratech.com | NA |
| Debbie Humbert | CLEAN Program Manager - BRAC Sites | Tetra Tech | 412-921-8968 | debra.humbert@tetratech.com | NA |
| Roger Donovan | Point of Contact (POC) for Site Investigation Activities | TDEC | 615-532-0864 | roger.donovan@state.tn.us | NA |
| Charlie Burroughs | TDEC Program Manager, Land Division | TDEC | 615-532-0863 | charles.burroughs@state.tn.us | NA |
| Greg Fraley | USEPA Region 4 RPM | USEPA | 404-562-8544 | greg.fraley@epa.gov | NA |
| Fred Grosskopf | Field Operations Leader (FOL)/Site Safety Officer (SSO)/Senior Geologist | Tetra Tech | 832-251-6058 | fred.grosskopf@tetratech.com | NA |
| Leanne Ganser | Project Chemist | Tetra Tech | 412-921-7090 | leanne.ganser@tetratech.com | NA |
| Kim Kostzer | Laboratory PM | Empirical Laboratories | 615-345-1115 | kkostzer@empirlabs.com | NA |
| Chino Ortiz | Laboratory PM | GPL Laboratories | 301-694-5310 | ortiz@gplab.com | NA |

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

(UFP-QAPP Manual Section 2.3.2)

| Name | Organization/Title/Role | Telephone Number (optional) | Signature/E-Mail receipt | SAP Section Reviewed | Date SAP Read |
|---|---|---|---|---|---|
| Fred Grosskopf | Tetra Tech/FOL/SSO/Senior Geologist | 832-251-6058 | | All | 12-Feb-10 |
| Matt Soltis | Tetra Tech/Health and Safety Manager (HSM) | 412-921-8912 | | Health and Safety Plan (HASP) | 12 FEB-'10 |
| Joe Samchuck | Tetra Tech/Data Validation Manager (DVM) | 412-921-8510 | | Worksheets #12, 14, 15, 19, 20, 23-28, 30, 34-37 | 12 FEB-10 |
| Leanne Ganser | Tetra Tech, Project Chemist | 412-921-8148 | | Worksheets #12, 14, 15, 19, 20, 23-28, 30, 34-37 | 12 FEB-10 |
| Kim Kostzer | Empirical Laboratories PM | 615-345-1115 | SEE EMAIL RECEIPT | Worksheets #12, 14, 15, 19, 20, 23-28, 30, 34-37 | 15-FEB-10 |
| Chino Ortiz | GPL Laboratories PM | 301-694-5310 | SEE E-MAIL RECEIPT | Worksheets #12, 14, 15, 19, 20, 23-28, 30, 34-37 | 15-FEB-10 |

## Rumer, Chris

**From:** Kim Kostzer [kkostzer@empirlabs.com]
**Sent:** Monday, February 15, 2010 11:08 AM
**To:** Rumer, Chris
**Subject:** NSA Mid-South UFP-SAP approval

Chris,

I have reviewed and approve of the analytical worksheets for NSA Mid-South UFP-SAP.

Kim Kostzer

**WE CURRENTLY HAVE EXCESS CAPACITY FOR THE FIRST QUARTER OF 2010 * SPECIAL PRICING AVAILABLE * CALL US NOW * 1-800-345-1113**

**Kim Kostzer**
**Project Manager**
**Empirical Laboratories, LLC**
**621 Mainstream Dr, Suite 270 I Nashville, TN 37228 I www.empirlabs.com I Main: 615.345.1115 ext. 240 I Toll Free: 877.345.1113 I Fax: 615.846.5426**

**Recipient of the 2008 Region IV (Southeastern US) Subcontractor of the Year from the Small Business Administration.**
**Celebrating over 40 years of excellence, Empirical Laboratories is certified as a Woman-Owned Small Business and a Small Disadvantaged Business. Come visit our website at www.empirlabs.com today.**

## Rumer, Chris

**From:** Virginia Zusman [zusman@gplab.com]
**Sent:** Monday, February 15, 2010 4:18 PM
**To:** Rumer, Chris
**Cc:** Chino Ortiz
**Subject:** NSA Mid-South UFP-SAP forms

Chris,

I have reviewed the UFP-SAP forms 12, 14, 15, 19, 20, 23-28, 30, and 34-37 for Centauri Labs (formerly GPL Laboratories) for Nitroglycerine analysis by method 8330B; and approve all of them, with the exception of the required sample volume for this analysis for soil on Form 19.

Thank you,
Virginia Zusman

**Virginia Zusman** | Director of Quality Assurance
Centauri Labs | 7210 Corporate Court | Frederick, MD 21703
Tel: 301-694-5310 extension 215
vzusman@centaurilabs.com | www.centaurilabs.com

Confidentiality Notice: Any documents accompanying this email contain confidential information which is legally privileged. This information is intended only for the use of the recipient named above. If you received this e-mail in error, please notify the sender immediately. You are hereby notified that any disclosure, copying, or distribution of the contents of this email without the written consent of the sender is prohibited.

## Rumer, Chris

**From:** Virginia Zusman [zusman@gplab.com]
**Sent:** Monday, February 15, 2010 2:11 PM
**To:** Rumer, Chris
**Subject:** FW: NSA Mid-South UFP-SAP
**Attachments:** image002.gif

---

**From:** Virginia Zusman [mailto:zusman@gplab.com]
**Sent:** Monday, February 15, 2010 12:39 PM
**To:** 'Chris.Rumer@tetratech.com'
**Cc:** Chino Ortiz
**Subject:** FW: NSA Mid-South UFP-SAP

Mr. Rumer,

I do not know if either Chino or Dave Howell have already communicated this, but we will need at least 1 kg of soil sample to perform the Nitroglycerine by method 8330B. The 8330B grinding process requires a much higher amount of sample.

If you have any questions, please let me know.

Virginia

**Virginia Zusman** | Director of Quality Assurance
Centauri Labs | 7210 Corporate Court | Frederick, MD 21703
Tel: 301-694-5310 extension 215
vzusman@centaurilabs.com | www.centaurilabs.com

---

**From:** Chino Ortiz [mailto:ortiz@gplab.com]
**Sent:** Monday, February 15, 2010 12:05 PM
**To:** 'Virginia Zusman'
**Subject:** FW: NSA Mid-South UFP-SAP

---

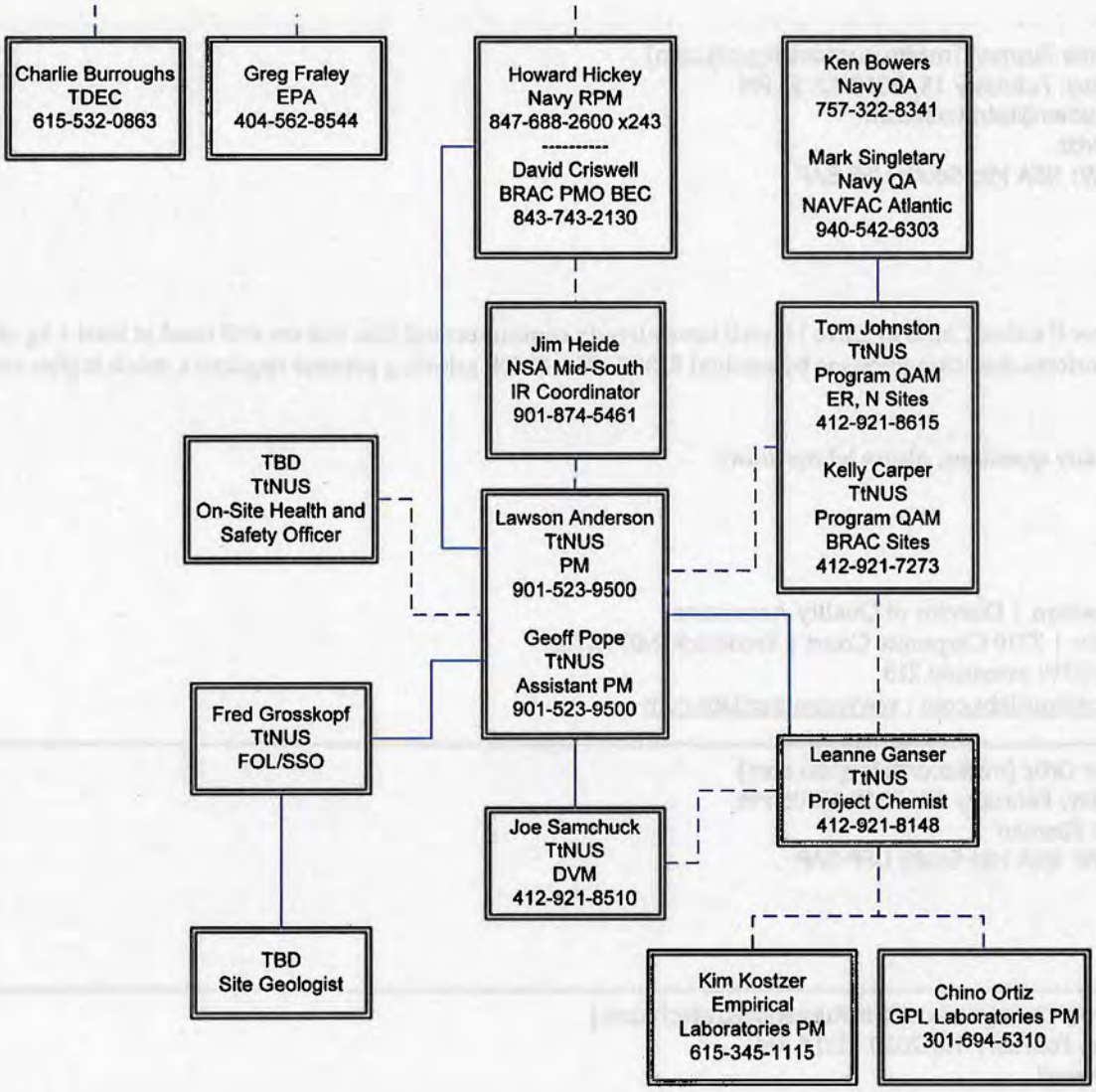
**From:** Rumer, Chris [mailto:Chris.Rumer@tetratech.com]
**Sent:** Friday, February 12, 2010 11:14 AM
**To:** David Howell
**Cc:** cortiz@centaurilabs.com
**Subject:** RE: NSA Mid-South UFP-SAP

Yes. I apologize for that. Please find attached the UFP-SAP for Small Arms Ranges for NSA Mid-South.

Thanks,
Chris

## SAP Worksheet #5 -- Project Organizational Chart

(UFP-QAPP Manual Section 2.4.1)



**SAP Worksheet #6 – Communication Pathways**

(UFP-QAPP Manual Section 2.4.2)

| Communication Driver | Responsible Party and Affiliation | Name | Phone Number and/or E-Mail | Procedure (timing, pathway to & from, etc.) |
|---|---|---|---|---|
| MEC Find | Field Staff, Tetra Tech | TBD | TBD | Field staff will inform Tetra Tech Unexploded Ordinance (UXO) Manager and Base POC.<br>Tetra Tech UXO Manager will inform Tetra Tech PM.<br>Base POC will make base emergency notifications.<br>Tetra Tech PM will inform Navy RPM/BEC.<br>Navy RPM/BEC will inform Naval Ordnance Safety and Security Activity (NOSSA). |
| | PM, Tetra Tech | Lawson Anderson | 901-523-9500 | |
| | BEC, BRAC PMO SE | David Criswell | 843-743-2130 | |
| | RPM (ER,N), NAVFAC MW | Howard Hickey | 847-688-2600 x243 | |
| | IR Coordinator, NSA Mid-South | Jim Heide | 901-874-5461 | |
| SAP Amendment | BEC, BRAC PMO SE | David Criswell | 843-743-2130 | Within 24-hours, the FOL will inform the Tetra Tech PM via Field Task Modification Request (FTMR) form; PM will notify RPM/BEC. |
| | RPM (ER,N), NAVFAC MW | Howard Hickey | 847-688-2600 x243 | |
| Change in Schedule | PM, Tetra Tech | Lawson Anderson | 901-523-9500, x214 | Tetra Tech will Inform Navy within 2 weeks via schedule impact letter as soon as impact is realized. |
| Field Issues Resulting in Changes in Field Work Scope | FOL/SSO, Tetra Tech | Fred Grosskopf | 832-251-6058 | FOL will inform PM within 8 hours; PM will inform RPM/BEC within 1 business day; RPM/BEC will issue scope change if warranted; scope change to be implemented before work is executed. Document via FTMR form. |
| | PM, Tetra Tech | Lawson Anderson | 901-523-9500, x214 | |
| | DVM, Tetra Tech | Joe Samchuck | 412-921-8510 | |

| Communication Driver | Responsible Party and Affiliation | Name | Phone Number and/or E-Mail | Procedure (timing, pathway to & from, etc.) |
|---|---|---|---|---|
| Recommendation to stop work and initiate work upon corrective action | FOL, Tetra Tech | Fred Grosskopf | 832-251-6058 | Within 1 hour, the responsible party will inform subcontractors, the Navy, and project team. |
| | TOM, Tetra Tech | Lawson Anderson | 901-523-9500, x214 | |
| | QAM, Tetra Tech | Kelly Carper | 412-921-7273 | |
| | HSM, Tetra Tech | Matt Soltis | 412-921-8912 | |
| | BEC, BRAC PMO SE | David Criswell | 843-743-2130 | |
| | RPM (ER,N), NAVFAC MW | Howard Hickey | 847-688-2600 x243 | |
| Field or laboratory data quality issue | Laboratory PM (Empirical) | Kim Kostzer | 615-345-1115 | Within 1 business day, the responsible party will notifiy the Tetra Tech Project Chemist.<br><br>Data Validation Staff and Tetra Tech PM will be notified if necessary |
| | Laboratory PM (GPL) | Chino Ortiz | 601-694-5310 | |
| | Project Chemist, Tetra Tech | Leanne Ganser | 412-921-8148 | |

## SAP Worksheet #7 -- Personnel Responsibilities and Qualifications Table

(UFP-QAPP Manual Section 2.4.3)

| Name | Title/Role | Organizational Affiliation | Responsibilities | Education and/or Experience Qualifications (Optional) |
|---|---|---|---|---|
| Lawson Anderson | PM | Tetra Tech | Oversees project, financial, schedule, and technical day-to-day management of the project.<br>• Ensures timely resolution of project-related technical, quality, and safety questions associated with Tetra Tech operations.<br>• Functions as the primary Tetra Tech interface with the Navy RPM/BEC, NSA Mid-South, Tetra Tech field and office personnel, and laboratory POCs.<br>• Ensures that Tetra Tech health and safety issues related to this project are communicated effectively to all personnel and off-site laboratories.<br>• Monitors and evaluates all Tetra Tech subcontractor performance.<br>• Coordinates and oversees work performed by Tetra Tech field and office technical staff (including data validation, data interpretation, and report preparation).<br>• Coordinates and oversees maintenance of all Tetra Tech project records.<br>• Coordinates and oversees review of Tetra Tech project deliverables.<br>• Prepares and issues final Tetra Tech deliverables to the Navy. | B.S. Environmental Science; CHMM; 25 years environmental experience. |

| Name | Title/Role | Organizational Affiliation | Responsibilities | Education and/or Experience Qualifications (Optional) |
|---|---|---|---|---|
| Fred Grosskopf | FOL SSO | Tetra Tech | Supervises, coordinates, and performs field sampling activities.<br>• Ensures that all health and safety requirements unique to the SIs are implemented.<br>• Functions as the on-site communications link between field staff members, NSA Mid-South, and the Tetra Tech PM.<br>• Alerts off-site analytical laboratories of any special health and safety hazards associated with environmental samples.<br>• Oversees the mobilization and demobilization of all field equipment and subcontractors.<br>• Coordinates and manages the field technical staff.<br>• Adheres to the work schedules provided by the Tetra Tech PM.<br>• Ensures the proper maintenance of site logbooks, field logbooks, and field recordkeeping.<br>• Initiates FTMRs (field change orders) when necessary.<br>• Identifies and resolves problems in the field, resolves difficulties via consultation with NSA Mid-South, implements and documents corrective action procedures, and provides communication between the field team and project management. | B.S. Geology, M.S. in Hydrogeology, 25 years environmental experience. |

| Name | Title/Role | Organizational Affiliation | Responsibilities | Education and/or Experience Qualifications (Optional) |
|---|---|---|---|---|
| Kelly Carper and Dr.Tom Johnston | QAM | Tetra Tech | Review SAP, oversee preparation of lab scopes, coordinate with labs, and conduct data quality reviews. Ensure quality aspects of the CLEAN program.<br>• Develop, maintain, and monitor quality assurance (QA) policies and procedures.<br>• Provide training to Tetra Tech staff in QA/quality control (QC) policies and procedures.<br>• Conduct system and performance audits to monitor compliance with environmental regulations, contractual requirements, SAP requirements, and corporate policies and procedures.<br>• Audit project records.<br>• Monitor subcontractor quality controls and records.<br>• Assist in the development of corrective action plans and ensuring correction of non-conformances reported in internal or external audits.<br>• Ensure that this SAP meets Tetra Tech, Navy, USEPA, and TDEC requirements.<br>• Oversees the responsibilities of the Tetra Tech Project QA/QC Advisor.<br>• Prepare QA reports for management. | Ms. Carper - B.S. Biology, 17 years environmental experience.<br><br>Dr. Johnston - PhD Analytical Chemistry, 30 years environmental experience. |

| Name | Title/Role | Organizational Affiliation | Responsibilities | Education and/or Experience Qualifications (Optional) |
|---|---|---|---|---|
| Leanne Ganser | Project Chemist | Tetra Tech | Coordinates analyses with lab chemists, ensures that lab scopes are followed, QAs data packages, and communicates with Tetra Tech staff.<br>• Ensures that the project meets objectives from the standpoint of laboratory performance.<br>• Provides technical advice to the Tetra Tech team on matters of project chemistry.<br>• Monitors and evaluates subcontractor laboratory performance.<br>• Ensures timely resolution of laboratory-related technical, quality, or other issues effecting project goals.<br>• Functions as the primary interface between the subcontracted laboratories and the Tetra Tech PM.<br>• Coordinates and oversees work performed by the subcontracted laboratories.<br>• Oversees the completion of Tetra Tech data validation.<br>• Coordinates and oversees review of laboratory deliverables.<br>• Recommends appropriate laboratory corrective actions. | B.S. Environmental Science, M.S. Marine, Estuarine, and Environmental Science, 4 years environmental experience. |
| Joseph Samchuck | DVM | Tetra Tech | Responsible for QA of data validation deliverables. | B.S. Chemistry, MBA, M.S. Finance, 23 years environmental experience. |

| Name | Title/Role | Organizational Affiliation | Responsibilities | Education and/or Experience Qualifications (Optional) |
|---|---|---|---|---|
| Matt Soltis | HSM | Tetra Tech | Oversees CLEAN Health and Safety Program.<br>• Provides technical advice to the Tetra Tech PM on matters of health and safety.<br>• Oversees the development and review of the HASP.<br>• Conducts health and safety audits.<br>• Prepares health and safety reports for management. | B.S. Industrial Safety Sciences; CIH; CSP; 24 years environmental experience. |
| Kim Kostzer | Laboratory PM | Empirical Laboratories | Coordinates analyses with lab chemists, ensures that the scope is followed; QAs data packages, and communicates with Tetra Tech staff. | Can be provided upon request. |
| Chino Ortiz | Laboratory PM | GPL Laboratories | Coordinates analyses with lab chemists, ensures that the scope is followed, QAs 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 is also responsible for SSO duties. This action will be performed only as credentials, experience, and availability permit.

## SAP Worksheet #8 -- Special Personnel Training Requirements Table

(UFP-QAPP Manual Section 2.4.4)

There are no special training requirements.

**SAP Worksheet #9 -- Project Scoping Session Participants Sheet**

(UFP-QAPP Manual Section 2.5.1)

| | |
|---|---|
| **Project Name:** MRP Site Inspections<br><br>**Projected Sampling Date(s):** October 2009 (tentative)<br><br>**Project Manager:** Lawson Anderson | **Site Name:** NSA Mid-South<br><br>**Site Location:** Millington, TN |

**Date of Session:** September 15 and 16, 2008
**Scoping Session Purpose:** Data Quality Objectives Session

| Name | Title | Affiliation | Phone # | E-Mail Address | Project Role |
|---|---|---|---|---|---|
| Lawson Anderson | PM | Tetra Tech | 901-523-9500 | lawson.anderson@tetratech.com | Management |
| Geoff Pope | Deputy PM | Tetra Tech | 901-523-9500 | geoff.pope@tetratech.com | Management |
| Howard Hickey | RPM | NAVFAC MW | 847-688-2600 x243 | howard.hickey@navy.mil | Management |
| Kathy Stewart | RPM | BRAC PMO SE | 843-743-2134 | kathy.stewart@navy.mil | Management |
| Ralph Basinski | MRP Coordinator | Tetra Tech | 412-921-8308 | ralph.basinski@tetratech.com | MRP Specialist |
| Rob Williamson | IR Coordinator | NSA Mid-South | 901-874-5467 | rob.williamson@navy.mil | Support |

Comments/Decisions: A site walk was conducted by session participants, after which the DQO session was held. CSMs were reviewed and the seven-step DQO process was then conducted for each site. Worksheet assignments were distributed to team members.

Action Items: Complete worksheets and text for work plan and implement review process.

Consensus Decisions: Worksheets will be completed by the most appropriate personnel and then reviewed by technical personnel and the Tetra Tech PM.

**SAP Worksheet #9 -- Project Scoping Session Participants Sheet**

(UFP-QAPP Manual Section 2.5.1)

| | |
|---|---|
| **Project Name:** MRP Site Inspections<br><br>**Projected Sampling Date(s):** October 2009 (tentative)<br><br>**Project Manager:** Lawson Anderson | **Site Name:** NSA Mid-South<br><br>**Site Location:** Millington, TN |

**Date of Session:** February 4 and 5, 2009
**Scoping Session Purpose:** Worksheet and Data Quality Objective Process Review

| Name | Title | Affiliation | Phone # | E-Mail Address | Project Role |
|---|---|---|---|---|---|
| Geoff Pope | Deputy PM | Tetra Tech | 901-523-9500 | geoff.pope@tetratech.com | Management |
| Rachael Bailey | Environmental Scientist | Tetra Tech | 901-523-9500 | rachael.bailey@tetratech.com | Management |
| Howard Hickey | RPM | NAVFAC MW | 847-688-2600 x243 | howard.hickey@navy.mil | Management |
| Kathy Stewart | RPM | BRAC PMO SE | 843-743-2134 | kathy.stewart@navy.mil | Management |
| Ralph Basinski | MRP Coordinator | Tetra Tech | 412-921-8308 | ralph.basinski@tetratech.com | MRP Specialist |
| Rob Williamson | IR Coordinator | NSA Mid-South | 901-874-5467 | rob.williamson@navy.mil | Support |
| Roger Donovan | POC for SIs Activities | TDEC | 615-532-0864 | roger.donovan@state.tn.us | Regulatory Review |
| Charles Burroughs | Alternate POC for SIs Activities | TDEC | 615-532-0863 | charles.burroughs@state.tn.us | Regulatory Review |
| Joseph Beutler | Environmental Specialist | NAVFAC MW | 847-688-2600 x322 | joseph.beutler@navy.mil | Management |
| Greg Fraley | USEPA Region 4 RPM | USEPA | 404-562-8544 | greg.fraley@epa.gov | Regulatory Review |
| Jack Carmichael | Hydrogeologist | USGS | 615-837-4704 | jkcarmic@usgs.gov | Technical Review |

Comments/Decisions: See meeting minutes.

Action Items: See meeting minutes.

Consensus Decisions: See meeting minutes

**Draft Meeting Minutes**
**Munitions Response Program Site Investigation Project Planning**
**BCT Meeting**
**NSA Mid-South**

Date: Wednesday, February 4, 2009
Time: 8:30 a.m. to 4:00 pm, CDT
Location: Environmental Conference Room NSA Mid-South

1. Attendees

| | | | |
|---|---|---|---|
| Ben Brantley | EnSafe | bbrantley@ensafe.com | 901-337-7962 |
| Corey Coleman | EnSafe | ccoleman@ensafe.com | 901-372-7962 |
| Howard Hickey | NAVFAC MW | howard.hickey@navy.mil | 847-815-6719 |
| Joseph Beutler | NAVFAC MW | joseph.beutler@navy.mil | 847-688-2600 x322 |
| Kathy Stewart | BRAC PMO SE | kathryn.stewart@navy.mil | 843-743-2134 |
| Rob Williamson | NSA Mid-South | rob.williamson@navy.mil | 901-874-5467 |
| Gregory Fraley | USEPA | fraley.gregory@epa.gov | 404-562-8544 |
| Jack Carmichael | USGS | jkcarmic@usgs.gov | 615-837-4704 |
| Roger Donovan | TDEC | roger.donovan@state.tn.us | 615-532-0864 |
| Charles Burroughs | TDEC | charles.burroughs@state.tn.us | 615-532-0863 |
| Ralph Basinski | Tetra Tech | ralph.basinski@tetratech.com | 412-251-9736 (c)<br>412-921-8308 (o) |
| Geoff Pope | Tetra Tech | geoff.pope@tetratech.com | 901-523-9500 |
| Rachael Bailey | Tetra Tech | rachael.bailey@tetratech.com | 901-523-9500 |
| Ronnie Moody | SpectraTech | rmoody@spectratechinc.com | 865-256-8830 |
| Paul Gentry | SpectraTech | pgentry@spectratechinc.com | 865-748-7700 |

The February 4, 2009 Project Planning meeting was held to review the UFP SAP Process and reach a consensus regarding key issues of the SI process. The significant items discussed and agreed upon during the Planning meeting were as follows:

- USEPA National SSL tables will be used for contaminant screening.

- Lead was selected to be the main COC driving regulatory determination for each site, and any subsequent impacts from other inorganic COCs are accepted to be co-located with lead-impacted soil.

- All decisions regarding the presence or absence of contamination or the need for further action will be based on lead; therefore, lead will be the only inorganic analyzed for during the SIs.

- The action level for determining whether concentrations of lead in soil at the MRP sites pose an unacceptable risk to human health was established as 400 milligrams/kilogram (mg/kg).

- The portion of shot fall zone of the Trap/Skeet Range #2 that falls within the boundaries of the SWMU 2 Landfill will be removed from the area to be investigated during the SIs because land use controls for the SWMU 2 landfill are in place that prohibit penetrating the landfill cap.

**SAP Worksheet #10 – Conceptual Site Model and Problem Definition**

(UFP-QAPP Manual Section 2.5.2)

## 10.0 INTRODUCTION

Naval Support Activity (NSA) Mid-South is located in the Town of Millington in Shelby County, Tennessee, approximately 20 miles north of Memphis, 180 miles west of Nashville, and 7 miles east of the Mississippi River. The facility encompasses 1,600 acres and serves as one of the largest inland Navy installations in the world. As of April 2002, the base employed 1,100 people including enlisted personnel, officer personnel, and civilians. Seven MRP-eligible sites were identified at NSA Mid-South, including, Pistol Range, Rifle Range, Aircraft Firing Range, Trap/Skeet Ranges #1 and #2, and Horse Stables Skeet Range #1, Horse Stables Skeet Range #2. Trap/Skeet Range #2 was active during the Preliminary Assessment (PA) and was not included; therefore, only six sites were included in that report (Malcolm Pirnie, 2005). Trap/Skeet Range #2 was later inactivated and was then added to the list of inactive ranges, bringing the total to seven. The historic information presented in the following subsections is a summary of the information presented in the 2005 PA.

To facilitate discussion, the seven MRP-eligible sites have been divided into the following two groups:

- Firing Ranges – Rifle Range, Pistol Range, and Aircraft Firing Range.
- Skeet Ranges – Trap/Skeet Ranges #1 and 2, Horse Stables Skeet Range #1, Horse Stables Skeet Range #2.

## 10.1 Firing Ranges

This section discusses the former firing ranges, including the Pistol Range, Rifle Range, and Aircraft Firing Range.

### 10.1.1 Site Location, History, and Physical Features

#### 10.1.1.1 Pistol Range

The former Pistol Range consists of approximately 0.5 acre from the firing line to a containment berm located behind the former target areas and is located near the southern border of the installation, east of Singleton Avenue, off the Perimeter Security Patrol Road. The Pistol Range was constructed after 1948 at the former location of a skeet range and was in use until 1994.

Property records indicate that the range, when active, consisted of a berm, shooting stations, targets, and an armory. The firing line maintained 16 firing positions with targets that were operated via a manual, mechanical cable system from behind the firing lines. This included five target lines, one each at 7 yards, 15 yards, 25 yards, 1,000 inches and 50 yards. According to information presented in the PA, the targets present at the pistol range were made of paper and secured to aboveground posts. A U-Shaped berm, approximately 10-15 feet tall and 500 feet long encompassed the area behind and on both sides of the target lines. Munitions use was limited to small arms ammunition, including 0.22-caliber, 0.30-caliber, 9-mm, 0.38-caliber, and 0.45-caliber ammunition. The current conditions of the former Pistol Range are shown on Figure 10.1.

A visual inspection conducted during the PA identified expended ammunition on the surface of the berm. Additionally, a site visit was conducted by TtNUS and NAVFAC MW personnel on September 15, 2008, with expended rounds visually identified on the surface of the containment berm. Also identified at the base of the containment berm were 40-mm training rounds; although, subsequent discussions with NAVFAC MW and NSA Mid-South personnel could not determine how or why the 40-mm rounds were present. NSA Mid-South personnel suggested that the vegetation present on the berm be cleared by the Public Works Department prior to conducting the SI to aid in identification of additional expended ammunition. During the September 2008 site visit, only three of the five historic target lines were readily identifiable.

10.1.1.2 Rifle Range

The former Rifle Range consists of approximately 11 acres from the firing line to a containment berm formerly located behind the target area and is located near the northern border of the installation, east of Attu Extension and south of Kerrville Rosemark Road, adjacent to a military housing complex. The range was built in 1942 and after closure of the range in the mid-1960's, soils from the containment berm were used for site work during construction of the NSA Mid-South Naval Hospital in 1965. A review of historic records and previous environmental reports available do not indicate remediation of the Rifle Range either before or after removal of the berm.

Property records indicate that the range, when active, consisted of a berm, shooting stations, targets, target house, and three magazines. This included three firing lines (one each at 50, 100, and 200 yards). A containment berm, approximately 450 feet long, was located behind the targets. According to conversations with NSA Mid-South personnel present when the Rifle Range was operational, the targets were wooden in composition and were raised and lowered from a concrete vault located in front of the containment berm. Munitions use was limited to small arms ammunition, including 0.22-caliber,

0.30-caliber and 0.50-caliber machine gun ammunition. The current conditions of the former Rifle Range are shown on Figure 10.2.

A visual inspection conducted during the PA did not identify any MC between the areas of the former firing lines and former containment berm. Additionally, a site visit was conducted by TtNUS and NAVFAC MW personnel on September 15, 2008, with no expended munitions fragments visually noted at the Rifle Range. Even though the target area and berm were previously removed, the concrete pads marking the locations of the former firing points are still present and were noted during the September 2008 site visit.

#### 10.1.1.3 Aircraft Firing Range

The former Aircraft Firing Range consists of approximately 8.5 acres located near the southern border of the installation, west of Singleton Avenue, directly south of "B" Street. The Aircraft Firing Range was constructed in 1942 and was used for bore sightings of fixed-wing machine guns on aircraft through World War II. Later, the range was converted to an aircraft maintenance garage and remained in use until 1995 when the Army Reserve began using the area. A containment berm was previously associated with the Aircraft Firing Range but was removed prior to use of the area by the Army Reserve. A review of historic records and previous environmental reports available (including the PA) was not able to identify when the berm was removed or where the soil was transported.

Property records indicate that the range, when active, consisted of the Machine Gun Test Building, which later became an aircraft maintenance garage, Instruction Building, offices, and two berms. According to information presented in the PA, the targets were typically wooden stands covered with cloth placed in front of the containment berms. The containment berms, each approximately 200 feet long, were located south of, and parallel to, the former building. Munitions use was limited to small arms ammunition, including 0.30- and 0.50-caliber machine gun ammunition. The current conditions of the former Aircraft Firing Range are shown on Figure 10.3.

A visual inspection conducted during the PA did not identify any MC between the areas of the former firing lines and former containment berms. A site visit was conducted by TtNUS and NAVFAC MW personnel on September 15, 2008, with no expended munitions fragments visually noted at the former Aircraft Firing Range.

### 10.1.2 Conceptual Site Model for Firing Ranges

#### 10.1.2.1 Potential or Known Contaminants

Lead is assumed to be the primary inorganic munition constituent (MC) of concern because it was the primary component of the spent munitions used during operation of each small arms range; and because of its documented toxicity to both human and ecological receptors. The other inorganic MCs that comprised the ammunition used at the firing ranges (e.g., antimony, zinc, copper, arsenic and tin) were traditionally present in much smaller fractions than lead and are accepted to be historically co-located with the lead. Therefore, the BCT decided that lead is the only inorganic MC that will be investigated under this SI (see Worksheet #9 meeting minutes). Nitroglycerin (NG), in the form of smokeless powder and ejection of unburned propellants, is another MC associated with firing ranges; and is suspected, along with lead from lead-azide initiating compound, to be present at firing points.

By design, firing ranges are directed fire training areas with containment berms behind the targets that stop the munitions expended on the range. As previously stated, the targets present at each of the firing ranges were located on various types of aboveground stands. Therefore, it is assumed that munitions fragments impacted the containment berms after passing through the targets or fell onto surrounding ground. As a result, it is expected that any MC present would have only penetrated into the first foot (0-1 foot depth interval) of the surficial soil and containment berms at each firing range. Expended ammunition, as previously discussed in Sections 10.1.1.1, 10.1.1.2, and 10.1.1.3, was trapped by the target berms (no longer present on the Rifle or Aircraft Firing Ranges) and could still be present along the range floor immediately in front of the Pistol Range target berm and former berm locations of the Rifle and Aircraft Firing Ranges. Expended munitions that did not impact the containment berms may have travelled a much farther distance (i.e., the Surface Danger Zone (SDZ), as indicated in Figures 10.1, 10.2, and 10.3). Given the large areas of undeveloped land that are encompassed by the SDZs outside of the current NSA Mid-South property boundaries, this SI will only be focusing on impacted soil within the defined boundaries of each range, as indicated on Figures 10.1, 10.2, and 10.3.

#### 10.1.2.2 Migration Pathways

As a result of frost heave, MC may become exposed at the ground surface and become a potential risk to both human and ecological receptors. MC, if present in surface soil, could potentially migrate to subsurface soil and subsequently infiltrate to the shallow water-bearing zones in the loess (present across most of NSA Mid-South) or alluvium (restricted to stream valleys, such as Big Creek) where it could potentially migrate with the groundwater flow. The loess, comprised of silt, silty clay, clay, and minor amounts of sand, is present at the Rifle Range, while alluvium, which is alleviated or reworked

loess, is present at the Pistol Range and Aircraft Firing Range. The upper alluvium is comprised primarily of silt and silty clay overlying sand and gravel (EnSafe, 2005).

Groundwater in the shallow water-bearing zone at NSA Mid-South is generally found at a depth of approximately five to 15 feet bgs. Groundwater in the loess is typically perched and discontinuous. When present, it moves slowly downward to recharge the underlying fluvial deposits and laterally to discharge locally to nearby streams, drainage ditches and surface water bodies. In addition, the loess, due to the low permeability of the silt, generally retards the downward movement to the underlying fluvial deposits (Carmichael, Parks, Kingsbury, and Ladd, 1997).

While the upper portions of the alluvium contain water, the chemical and physical properties of the silt/silty clay soils affect the permeability and downward migration of chemical constituents. Any potential contaminants entering the shallow water-bearing zone would be expected to primarily migrate laterally towards Big Creek Drainage Canal, the lowest hydraulic point in the area (Carmichael, Parks, Kingsbury, and Ladd, 1997). The leaching process for transferring metals from projectiles to soils is slow because of the relatively low surface area exposed to the elements and the virtual insolubility of the metals in the environment. While precipitation infiltration could mobilize lead into subsurface soil and shallow groundwater, given the near immobility of lead in soil and the depth to groundwater, significant migration of MCs is not expected. Therefore, the scope of this SI is limited to surface soil characterization.

10.1.2.3 Receptors, Exposure Pathways, and Future Land Use

Current human receptors include Navy personnel, Navy-escorted visitors (e.g., contractors conducting environmental or ecological surveys), and trespassers (e.g., authorized personnel who may wander outside of designated areas). The facility's current land uses are expected to remain unchanged into the foreseeable future; therefore, all current potential receptors are also considered potential future receptors. In addition, future contractors hired to maintain grounds and perform other maintenance tasks such as sewer repair may be exposed to MC from former range activities. Current and future ecological receptors include the current flora (predominantly grassland species) and fauna (large mammals such as deer, small mammals such as rabbits, reptiles/amphibians, and bird species) present at the sites.

Unexpended munitions that may be present at the site may become exposed and present an explosive risk to fauna and human receptors (e.g., groundskeepers or other personnel crossing the site). Fragments from small arms munitions present at the site may also become exposed and present a risk to receptors.

Future land use for the Firing Ranges is undetermined; however, NSA Mid-South is expected to remain an active Naval installation for the foreseeable future and the current land use for each site is as follows:

- The Pistol Range is unused land.

- The Rifle Range was given back to the community through BRAC in 1999, and the Navy currently leases the site from the City of Millington for use as a grazing area and stables for horses.

- The Aircraft Firing Range site is currently used as motor pool parking and equipment storage by the Army Reserve.

A potential future land use for the sites is residential; however the facility and ranges are expected to remain active for the foreseeable future.

### 10.1.3 Problem Statement

As a result of historical small arms range activities, MC consisting of NG and lead may be present in surface soil at the small arms range sites. Surface soil, which is the environmental medium most likely to be contaminated from past site operations, needs to be investigated during the SI to determine whether MC are present at concentrations that could present unacceptable human health risk and if an RI of the site is required.

## 10.2 Skeet Ranges

This section discusses the former skeet ranges, including Trap/Skeet Ranges #1 and #2, Horse Stables Skeet Range #1, and Horse Stables Skeet Range #2.

### 10.2.1 Site Location, History, and Physical Features

#### 10.2.1.1 Trap/Skeet Ranges #1 and #2

Former Trap/Skeet Ranges #1 and #2 consist of approximately 79 acres, located near the southern border of the installation, east of Singleton Avenue, off the Perimeter Security Patrol Road, adjacent to the former Pistol Range. The ranges were built around 1943, with Trap/Skeet Range #1 having an unrecorded closure date sometime after World War II and Trap/Skeet Range #1 closing in 2005. No remedial efforts are documented for closure of the ranges.

Property records indicate that the range, when active, had a firing line, skeet office and armory, two clay pigeon storage buildings, an ammunition storage building, Instruction Building, and shooting stations and targets. Munitions use was limited to small arms ammunition, primarily 12- and 20-gauge shotgun shells.

The current conditions of the Former Trap Skeet Ranges #1 and #2 are shown on Figure 10.4. Of note is that Solid Waste Management Unit (SWMU) 2, a closed solid waste landfill that served NSA Mid-South, is located within the defined boundaries of the Former Trap Skeet Ranges #1 and #2. To maintain the integrity of the landfill cap, investigation of the portion of SWMU 2 that lies within the boundaries of the Former Trap Skeet Ranges #1 and #2 has been removed from this SI.

A visual inspection conducted during the PA found no evidence of the former Trap Skeet Ranges #1 and #2, as the concrete firing lines and all structures associated with the ranges had previously been removed. The PA visual inspection did observe broken skeet targets present on the ground surface. A site visit conducted by TtNUS and NAVFAC MW personnel on September 15, 2008; with no expended munitions fragments or skeet targets visually noted at the former Trap Skeet Ranges #1 and #2.

10.2.1.2 Horse Stables Skeet Range #1

The former Horse Stables Skeet Range #1 consists of approximately 36 acres on the northern portion of the installation, east of Attu Street Extension, adjacent to the Rifle Range. The ranges were built in 1945 and closed in the summer of 1952 for the construction of a Navy family housing area. No remedial efforts are documented for the closure of the range.

Property records indicate that the range had a magazine, shed, a skeet tower, a firing arch, and high and low houses. The magazine, a shed and skeet tower, were used also for the adjacent skeet range. Munitions use was limited to small arms ammunition, primarily 12- and 20-gauge shotgun shells. The current conditions of the former Horse Stables Skeet Range #1 are shown on Figure 10.5.

A visual inspection conducted during the PA found no evidence of the former Horse Stables Skeet Range #1, as the structures formerly associated with the range were believed to have been removed prior to construction of the residential development. A site visit was conducted by TtNUS and NAVFAC MW personnel on September 15, 2008; with no expended munitions fragments or skeet targets visually noted at the former Horse Stables Skeet Range #1.

10.2.1.3 Horse Stables Skeet Range #2

The former Horse Stables Skeet Range #2 consists of approximately 42 acres, on the northern portion of the installation, west of Attu Street Extension, adjacent to Horse Stables Skeet Range #1. The range was built around 1942 and closed in 1946. The date of closure is not documented nor is any remedial effort for the closure of the range.

Property records indicate that the range, when active, had a magazine, shed, and skeet tower in addition to a firing arch and high and low houses, typical structures associated with a skeet range. The magazine, shed, and skeet tower were used also for the adjacent skeet range. Munitions use was limited to small arms ammunition, primarily 12- and 20-gauge shotgun shells. The current conditions of the former Horse Stables Skeet Range #2 are shown on Figure 10.6.

A visual inspection conducted during the PA found no evidence of the former Horse Stables Skeet Range #2, as the structures formerly associated with the range were believed to have been removed prior to construction of four houses in the area. A site visit was conducted by TtNUS and NAVFAC MW personnel on September 15, 2008, with no expended munitions fragments or skeet targets visually noted at the former Horse Stables Skeet Range #2 during the site visit.

### 10.2.2 Conceptual Site Model for Skeet Ranges

#### 10.2.2.1 Potential or Known Contaminants

The munitions fragments expected to be present at skeet ranges are lead shot from the ammunition used and pieces of broken targets. Lead is assumed to be the primary inorganic, MC of concern because it was the primary constituent in the spent munitions used during operation of each skeet range and because of its documented toxicity to both human and ecological receptors. The other inorganic MCs that comprised the munitions used at the skeet ranges (e.g., antimony, zinc, copper, arsenic and tin) were traditionally present in much smaller fractions than lead and are accepted to be historically co-located with the lead present. Therefore, the BCT decided that lead is the only inorganic MC that will be investigated under this SI (see Worksheet #9 meeting minutes). Other MC of concern associated with skeet ranges consists of NG and PAHs. NG in the form of smokeless powder, along with lead from lead-azide initiating compound, may be present at the firing points as the result of ejection of unburned powders from shotguns. PAHs may be present due to fragmentation of clay pigeon targets containing pitch tar, which was used as a binding agent.

Based on past site usage, lead, and clay target fragments may have accumulated in the soil across the SDZ during operation of the skeet ranges. Lead and unburned smokeless powders may have also accumulated at the skeet ranges firing points. As the targets used at each of the skeet ranges were projected vertically upward, any munitions fragments (lead shot or skeet targets) would have fallen back down to the ground below due to gravity; as a result, it is expected that any MC present would have only penetrated into the first foot (0-1 foot depth interval) of the surficial soil at each skeet range.

10.2.2.2 Migration Pathways

As a result of frost heave, MC may become exposed at the ground surface and become a potential risk to both human and ecological receptors. MC, if present in surface soil, could potentially migrate to subsurface soil and subsequently infiltrate to the shallow water-bearing zones in the loess (present across most of NSA Mid-South) or alluvium (restricted to stream valleys, such as Big Creek) where it could potentially migrate with the groundwater flow. The loess, comprised of silt, silty clay, clay, and minor amounts of sand, is present at the Horse Stables Skeet Ranges #1 and #2, while alluvium, which is alleviated or reworked loess, is present at the Trap/Skeet Ranges #1 and #2. The upper alluvium is comprised primarily of silt and silty clay overlying sand and gravel (EnSafe, 2005).

Groundwater in the shallow water-bearing zone at NSA Mid-South is generally found at a depth of approximately five to 15 feet bgs. Groundwater in the loess is typically perched and discontinuous. When present, it moves slowly downward to recharge the underlying fluvial deposits and laterally to discharge locally to nearby streams, drainage ditches and surface water bodies. In addition, the loess, due to the low permeability of the silt, generally retards the downward movement to the underlying fluvial deposits (Carmichael, Parks, Kingsbury, and Ladd, 1997).

While the upper portions of the alluvium contain water, the chemical and physical properties of the silt/silty clay soils affect the permeability and downward migration of chemical constituents. Any potential contaminants entering the shallow water-bearing zone would be expected to primarily migrate laterally towards Big Creek Drainage Canal, the lowest hydraulic point in the area (Carmichael, Parks, Kingsbury, and Ladd, 1997). The leaching process for transferring metals from projectiles to soils is slow because of the relatively low surface area exposed to the elements and the virtual insolubility of the metals in the environment. While precipitation infiltration could mobilize lead into subsurface soil and shallow groundwater, given the near immobility of lead in soil and the depth to groundwater, significant migration of MCs is not expected. Therefore, the scope of this SI is limited to surface soil characterization.

10.2.2.3 Receptors, Exposure Pathways, and Future Land Use

Current human receptors at these sites include Navy personnel, Navy-escorted visitors (e.g., contractors conducting environmental or ecological surveys), and trespassers (e.g., authorized personnel who may wander outside of designated areas). In addition, future contractors hired to maintain grounds and perform other maintenance tasks such as sewer repair may be exposed to MC from former range activities. Current and future ecological receptors include the current flora (predominantly grassland species) and fauna (large mammals such as deer, small mammals such as rabbits, reptiles/amphibians, and bird species) present at the site.

Unexpended munitions that may be present at the site may become exposed and present an explosive risk to fauna and human receptors (e.g., groundskeepers or other personnel crossing the site). Fragments from small arms munitions and broken skeet targets present at the site may also become exposed and present a risk to both fauna and human receptors.

Future land use for the Skeet Ranges is undetermined; however, the current land use for each site is as follows:

- The northern portion of Trap/Skeet Ranges #1 and #2 is the location of a water tower, commissary, military housing, and child care facility, and the southern portion is currently undeveloped.
- The Horse Stables Skeet Range #1 is currently being redeveloped as residential housing.
- The Horse Stables Skeet Range #2 is currently used as a grazing area for nearby Horse Stables.

### 10.2.3 Problem Statement:

As a result of historical range activities, MC consisting of NG, lead, and PAHs may be present in surface soil at the Skeet Range site. Surface soil, which is the environmental medium most likely to be contaminated from past site operations, needs to be investigated during the SI to determine whether MC are present at concentrations that could present unacceptable human health risk and if an RI of the site is required.

## SAP Worksheet #11 -- Project Quality Objectives/Systematic Planning Process

### IDENTIFY THE GOALS OF THE STUDY

The primary goals of the SIs for these seven sites are as follows:

1. Determine whether surface soil MC concentrations exceed human health risk-based screening values or background concentrations. If screening values and background concentrations are exceeded proceed to an RI, otherwise, recommend No Further Action (NFA).

2. Collect adequate data to determine the presence and approximate horizontal and vertical extent of soil contamination. If the data collected during the initial round of sampling are adequate, stop delineation, otherwise, complete delineation during an RI.

### IDENTIFY INFORMATION INPUTS

Data required for making the decisions include the following:

1. Field screening data: Lead concentrations in surface soil will be determined by field-portable X-ray fluorescence spectroscopy (XRF). These measurements will be used as a field screening tool for potential contaminant delineation purposes and for locating hot spots of lead. A correlation study of lead XRF data to fixed-base laboratory data will be conducted. The correlation coefficient must be greater than or equal to 0.65 and less than or equal to 1.00 to convert field XRF values into equivalent laboratory lead concentrations. The XRF Standard Operating Procedure (SOP)-01 is included in Appendix A.

2. Laboratory target analyte data: Laboratory data will be used to determine concentrations of MC of concern in surface soil. MC at the Firing Ranges includes lead and NG, while MC at the Skeet Ranges includes lead, NG and PAHs. The data will also be used to determine the potential for contaminated soil to impact groundwater. Surface soil samples will be analyzed as prescribed in Worksheet #18.

3. Miscellaneous laboratory soil parameters: Soil total organic carbon (TOC), cation exchange capacity (CEC), and soil pH will be determined by Empirical Laboratory for select samples. These data are necessary for the evaluation of MC environmental fate and transport if contamination is detected.

4. Field identification/classification of soil types (i.e., lithology and Unified Soil Classification System for grain size, color, plasticity, etc.). Worksheet #21 provides relevant SOPs.

5. Screening values: The SIs require field screening and laboratory data that can be used to determine if further investigation is necessary. To conduct comparisons of site data to screening values, laboratory quantitation limits must be low enough to measure MC concentrations down to the screening values. For the SIs, the screening values are also the project action limits (PALs), as follows:

   - USEPA Regional Screening Levels (RSLs) for Chemical Contaminants at Superfund Sites (ORNL, 2009).

   - The XRF field screening value is 280 mg/kg, which value represents 70 percent of the 400 mg/kg USEPA residential soil action level to provide a conservative margin of error. The margin of error was selected based on recent experiences with field XRF analyses at other small arms ranges.

   - The background concentrations for NSA Mid-South presented in the Reference Concentrations Technical Memorandum (E/A&H, 1996) will be used as screening values if they are greater than the USEPA human health screening values for any particular analyte.

The applicable screening values for the MC of concern can be found in Worksheet #15.

**DEFINE THE BOUNDARIES OF THE STUDY**

The current SIs are limited to the evaluation of soil. Descriptions of the ranges, when operational, are provided in WS #10, with visual depictions presented on Figures 10.1 through 10.6. The horizontal and vertical boundaries as well as the temporal boundaries for the study are as follows:

1. The population of interest includes surface soil that may have been contaminated directly by site operations or by subsequent migration of contaminants. The 0 to 1 foot bgs vertical interval will be investigated to achieve study goals, because the CSM indicates that penetration of soil by MC is not generally expected to be deeper than 1 foot bgs.

2. The initial horizontal study boundary will encompass the areas most likely to have been impacted by site activities as described in the CSM.

3. Temporal boundaries are not a significant consideration in this study because MC metals concentrations are anticipated to be relatively unchanged (stable) over the course of time needed to conduct the environmental investigations and into the foreseeable future.

## DEVELOP THE ANALYTIC APPROACH

Decision rules for this investigation include the following:

1. A value of 280 mg/kg will be used as the discriminator between contaminated and uncontaminated locations. If the XRF-measured concentrations of lead in any soil sample on the perimeter (vertical or horizontal) of the sampled area exceed 280 mg/kg, "step out" for XRF lead analysis of additional soil samples until the results of the XRF analysis are less than 280 mg/kg. No more than two step-out samples will be collected in either the horizontal or vertical direction.

2. If MC (lead and NG at the Firing Ranges and lead, NG and PAHs at the Skeet Ranges) concentrations in all soil samples in the initial round of sampling are less than screening and background levels, recommend NFA; otherwise, proceed to an RI to conduct risk assessment and to fully delineate the extent of contamination.

## SPECIFY PERFORMANCE OR ACCEPTANCE CRITERIA

Surface soil samples will be collected from areas most likely to be contaminated to determine the presence of contamination in surface soil and to determine whether further investigation is necessary based on comparison of individual sample results to numerical action levels. A secondary goal is to begin to delineate the contamination, if present, within a limited number of step-out rounds. This selection of sample locations is biased, and data use does not support the use of statistics to estimate decision performance as specified in the DQO guidance (USEPA, 2006). Instead, the project team selected locations and numbers of samples which, based on their experience, will support the attainment of the stated project objectives. The project team will use the results of the investigation to determine whether the amount and type of data collected are sufficient to support the attainment of project objectives. This will involve an evaluation of contaminant concentrations and concentration patterns to ensure that contaminants are likely to have been detected, if present, and that enough data have been collected to support the SIs. The project team will review the field screening and analytical results to ensure that all viewpoints are included in decision making.

## DEVELOP THE PLAN FOR OBTAINING DATA

The proposed SI field data collection program for these sites is presented on Figures 17.1 through 17.6. The information developed in previous sections of this worksheet was used to develop the program, which assumes that the majority of the contamination is present in the 0 to 1-foot bgs soil interval. Areas most likely to be contaminated by site activities are described in the CSMs presented in Sections 10.1.2

and 10.2.2. The horizontal and vertical boundaries of the soil areas to be sampled will encompass all areas significantly impacted by site activities.

The sampling design consists of samples spaced along transect lines extending from the shooters' positions to the maximum shot fall line (for the skeet ranges) or within grids (for the firing ranges) to ensure that the sites are well represented spatially. The MC sampling and analytical program for soil samples is as follows:

- XRF Field Screening

Up to 75 surface soil samples (0- to 1-foot bgs) will initially be collected and analyzed with the XRF from the grids and transect lines established in the areas to be sampled as shown on Figures 17.1 through 17.6. Additionally, a site "walk over" will be conducted by the FOL to identify any obvious areas of fragment/lead shot accumulation. Up to 10 additional "discretionary" surface soil samples may be collected at each site based on visual observation of accumulated fragments or shot in the area to be sampled. Additional step-out samples will also be collected at the boundaries of the area to be sampled if XRF lead concentrations greater than 280 mg/kg are detected in initial surface soil samples. No more than two horizontal step-out samples will be collected per exceedance of 280 mg/kg by XRF analysis.

Vertical step out subsurface soil samples (starting at 1 foot bgs) will also be collected for field XRF analysis if their respective surface soil sample lead concentrations exceed 280 mg/kg. If the subsurface sample XRF lead concentration also exceeds 280 mg/kg, up to two additional subsurface soil samples will then be collected vertically in 2-foot intervals, if possible depending on soil conditions, until lead concentrations in subsurface soil do not exceed 280 mg/kg.

- Laboratory Analytical Program

Initially, all soil samples will be screened using XRF analysis. A minimum of 20 samples (or 20 percent of the total, whichever is greater) for each site will also be analyzed for lead at a fixed-base laboratory. The samples sent to the fixed-base laboratory will be selected to reflect the complete range of lead concentrations detected in soil at the site using XRF field screening. A correlation analysis (field screening versus fixed-base laboratory concentrations) will be conducted to determine the linear correlation between field and laboratory data if the data appear to be linearly correlated

For each skeet range, a minimum of 20 samples will be sent to the laboratory for PAH analysis. Samples will be collected at locations where PAH contamination is anticipated based upon knowledge of historical operations and/or locations where skeet fragments are observed during sample collection. Samples selected for PAH analysis will not necessarily be from locations selected for laboratory lead analysis.

Composite soil samples will be collected at the firing line(s) at each site for fixed-base laboratory NG and lead analyses. For all seven sites, GPL Laboratories (per WS 16) will prepare the firing line NG samples according to Appendix A SW-846 Method 8330B, as follows:

- Two composite surface soil samples (comprised of 10 grab samples each) will be collected from the firing line at the Horse Stables Skeet Ranges #1 and #2.

- Three composite surface soil samples (comprised of 10 grab samples each) will be collected, one from each firing line, at the Rifle Range.

- One composite surface soil sample (comprised of 10 grab samples) will be collected from the firing line at the Pistol Range.

- Ten composite surface soil samples (comprised of 10 grab samples each) will be collected along the firing lines at Trap Skeet Ranges #1 and #2.

- Two composite surface soil samples (comprised of 10 grab samples each) will be collected from the western firing line at the Aircraft Firing Range. Due to excessive disturbance of the eastern firing line and the fact that it is currently paved with asphalt, the samples from the western firing line will serve as the representative for both firing lines.

During the February 2009 planning meeting, it was discussed that existing soil geochemical data (e.g., TOC, CEC, and pH) would be used, if possible. Following this meeting, a review of historic environmental investigations conducted at NSA Mid-South yielded an incomplete set of geochemical data for soils. As a result, three grab soil samples from each site will be collected during the SIs for TOC, CEC, and pH analysis at the fixed-base laboratory.

## SAP Worksheet #12 -- Measurement Performance Criteria Table - Field Quality Control Samples

| QC Sample | Analytical Group | Frequency | Data Quality Indicators (DQIs) | Measurement Performance Criteria (MPCs) | QC Sample Assesses Error for Sampling (S), Analytical (A) or Both (S&A) |
|---|---|---|---|---|---|
| Equipment Rinsate Blank | All Fractions | One per 20 field samples per matrix per type of sampling equipment | Bias/Contamination | Detections ≤ Quantitation Limit (QL) | S&A |
| Field Duplicate | All Fractions | One per 10 field samples collected | Precision | Soil samples Relative Percent Difference (RPD) less than 50 | S |
| Cooler Temperature Blank | All Fractions | One per cooler | Accuracy/Representativeness | Between 2 and 6 °C | S&A |

**SAP Worksheet #13 – Secondary Data Criteria and Limitations Table**

(UFP-QAPP Manual Section 2.7)

| Secondary Data | Data Source (originating organization, report title and date) | Data Generator(s) (originating organization, data types, data generation / collection dates) | How Data Will Be Used | Limitations on Data Use |
|---|---|---|---|---|
| Site Histories, Initial CSMs | Naval Facilities Engineering Command Midwest, Preliminary Assessment, Naval Support Activity Mid-South, Millington, Tennessee, August 2005 | Malcolm Pirnie, Inc. | Basis for UFP-SAP, site histories, and CSMs | The information is qualitative and no quantitative (site-specific nature and extent of contamination) information is available. The information was used to establish the field work program and identify areas most likely to be contaminated. |
| Background Reference Concentrations | Table 1, Summary of Reference Concentrations, Technical Memorandum, Naval Support Activity Memphis, August 1996 | Ensafe/Allen and Hoshall (E/A&H) | Background lead concentration | These background reference concentrations are limited to metals. There are no background reference concentration s for PAHs. |

This is the initial phase of field investigation at the seven ranges, and no known secondary data exist.

## SAP Worksheet #14 -- Summary of Project Tasks

(UFP-QAPP Manual Section 2.8.1)

Small Arms Range SI project activities will consist of the following tasks:

- Field Tasks including:
  - Mobilization/demobilization and utility clearance
  - Drilling
  - Sample collection tasks
  - Sample custody
  - Quality control (QC) sample collection
  - Field instrument calibration
  - Decontamination
  - Investigation-Derived Waste (IDW) removal and disposal
  - Sample location with Global positioning system (GPS)
  - XRF lead analysis of soil
- Analytical tasks
- Data management
- Assessment and oversight
- Data review
- Project report

These tasks are summarized below. The SOPs and field forms referenced below and in other worksheets are included in Appendices A and C, respectively.

### Field Tasks

- **Mobilization/Demobilization:** Mobilization/demobilization activities include field equipment procurement and transport to the work site, subcontractor procurement and coordination, utility awareness and clearance, location and setup of areas for decontamination and waste storage, acquisition of vehicles, and establishment of an on-site staging area for work at all sites.

  Equipment requirements will be finalized by the FOL following the acceptance of the UFP-SAP. The FOL will review the scope of work and assemble a list of equipment (e.g., vehicles and sampling, personal protection, and decontamination equipment) required to implement and the field investigations.

This list will be reviewed by the Project Team and the PM. The FOL will be responsible for receiving and unpacking the equipment and ensuring that all equipment is operable and calibrated. The FOL will also be responsible for tracking equipment used in the field and coordinating acquisition and delivery of sample containers to the site and shipment of samples to the laboratories.

During mobilization, the FOL will review the roles and responsibilities of each field team member and review the requirements of the various field activities. A series of meetings will be conducted to review the sampling and analytical requirements. Upon mobilization, an on-site meeting will be conducted to review health and safety requirements. The FOL will also serve as the Site Safety Officer (SSO) responsible for reviewing the HASP with field team members and subcontractors. The field team will also be required to attend a hazard control briefing administered by the Mid-South Environmental Division POC prior to commencing any field work at NSA Mid-South. Daily safety briefings by Tetra Tech will occur for all field personnel.

- **Utility Clearance Tasks:** Prior to commencing any work at NSA Mid-South, the Comprehensive Work Approval Process (CWAP) will be followed. The CWAP will identify constraints in the work area, such as the locations of eagle's nests, archeological sites, wetlands, etc. that may affect work at the site and other requirements that must be met prior to commencing work, such as locating underground utilities, etc. Upon completion of the CWAP, a Dig Permit will be issued to the utility contractor. The FOL will coordinate with facility personnel and with a Tetra Tech subcontractor for utility clearance of all soil boring locations. Utilities that are not shown or are incorrectly located will be marked on the permit and the marked-up permit will be returned to the Activity POC for inclusion in the Activity geographic information system (GIS).

- **Drilling Tasks:** All soil borings at Pistol Range, Rifle Range, Aircraft Firing Range, Trap/Skeet Ranges #1 and #2, Horse Stables Skeet Range #1, and Horse Stables Skeet Range #2 will be advanced using hand augering techniques in accordance with SOP-05 (Soil Coring and Sampling Using Hand Augering Techniques). If necessary, some samples will be collected utilizing direct-push technology (DPT) in accordance with SOP-06 (Borehole Advancement and Soil Coring and Sampling Using Direct-Push Technology). All borings will be logged in accordance with SOP-10 (Borehole and Soil Sample Logging). Methods for recording data are included in each SOP. Field SOPs are included in Appendix A.

- **Sample Collection Tasks:** Surface and subsurface soil samples will be collected in accordance with SOP-05 (Soil Coring and Sampling Using Hand Augering Techniques) and, if necessary, SOP-06 (Borehole Advancement and Soil Coring and Sampling Using Direct-Push Technology). Soil

samples will be collected in accordance with SOP-08 (Soil Sampling). The sample numbering scheme will be in accordance with SOP-02 (Sample Identification and Nomenclature). Methods for recording data are included in each SOP and SOP-03 (Sample Custody and Documentation of Field Activities). Sample labeling will be in accordance with SOP-01 (Labeling), and sample containers, preservation, packaging, and shipping will be in accordance with SOP-11 (Sample Packaging). Field SOPs are included in Appendix A of this document.

The numbers and types of samples to be collected at each site and the analyses to be conducted are presented in Worksheets 18.1 through 18.6.

- **Sample Custody:** As described in SOP-03, chain-of-custody procedures and forms will be used for analytical samples collected. Chain-of-custody forms will be provided in the sample kit provided by the laboratory. When transferring the possession of samples, the individuals relinquishing and receiving the samples will sign, date, and note the time on the record. This record documents transfer of custody of samples from the sampler to the permanent laboratory. Until this transfer takes place, field personnel will ensure the samples are in their custody or in a secure storage area (e.g. locked vehicle or field trailer).

- **Quality Control Tasks:** Equipment blanks, field duplicates, matrix spikes, and matrix spike duplicates will be collected as presented in Worksheet #20.

  Initial and continuing calibration, tuning, and reagent blanks, surrogates, duplicates, laboratory control samples, and all other applicable QC sample for all analytical methods are presented in Worksheet #22.

- **Field Instrument Calibration:** These procedures are described in Worksheet #22.

- **Equipment Decontamination**: All reusable sampling equipment (e.g., stainless steel trowels, etc.) will be decontaminated prior to sampling and between samples, according to the sequence established in SOP-04 (Appendix A).

- **Investigation-Derived Waste (IDW) Tasks:** It is anticipated that waste materials (soil cuttings, decontamination fluids, and personal protective equipment (PPE)) will be generated during the field investigation. These wastes must be disposed in such a manner that does not contribute to further environmental contamination or pose a threat to public health or safety. SOP-09 in Appendix B provides information on the handling of IDW.

- **Global Positioning System:** A GPS survey will be used to locate all sampling points.

- **XRF Analysis:** On-site field XRF analysis for lead will be performed according to SOP-13. A minimum of 20 samples from each site will be sent to the fixed-base laboratory for confirmation analysis.

**Analytical Tasks** - Chemical analysis for lead and PAHs will be performed by Empirical Laboratories in Nashville, Tennessee and chemical analysis for NG will be performed by the subcontracted laboratory, GPL Laboratories. Empirical Laboratories and GPL Laboratories are NFESC approved and National Environmental Laboratory Accreditation Program (NELAP) accredited. Analyses will be performed in accordance with the analytical methods identified in Worksheet #30. Empirical Laboratories and GPL Laboratories will meet the Project Quantitation Limits (PQLs) specified in Worksheet #15. Empirical Laboratories and GPL Laboratories will perform the chemical analyses following laboratory-specific SOPs (Worksheets #19 and #23) developed based on the methods listed in Worksheets #19 and #30. Copies of the SOPs are included in Appendix B on the attached CD.

## SAP Worksheet #15 -- Reference Limits and Evaluation Table

**Matrix: Soil**

**Analytical Group: Inorganics – Lead (Fixed-Base Laboratory)**

| Analyte | CAS Number | Project Action Limit[1] (mg/kg) | Screening Value Reference | Project Quantitation Limit Goal (mg/kg) | Laboratory-Specific Limits[2] | |
|---|---|---|---|---|---|---|
| | | | | | Quantitation Limit (mg/kg) | Method Detection Limit (mg/kg) |
| Lead | 7439-92-1 | 400 | R-RSL | 130 | 1 | 0.3 |

**Matrix: Soil**

**Analytical Group: Inorganics – Lead (Field Data Collected via X-Ray Fluorescence Analyzer)**

| Analyte | CAS Number | Project Action Limit[3] (mg/kg) | Screening Value Reference | Project Quantitation Limit Goal (mg/kg) | Laboratory-Specific Limits[2] | |
|---|---|---|---|---|---|---|
| | | | | | Quantitation Limit (mg/kg) | Method Detection Limit (mg/kg) |
| Lead | 7439-92-1 | 280 | R-RSL | 130 | Not Calculated | 20 |

**Matrix: Soil**

**Analytical Group: Organics – PAHs**

| Analyte | CAS Number | Project Action Limit [1] (mg/kg) | Screening Value Reference | Project Quantitation Limit Goal (mg/kg) | Laboratory-Specific Limits[2] | |
|---|---|---|---|---|---|---|
| | | | | | Quantitation Limit (mg/kg) | Method Detection Limit (mg/kg) |
| 1-Methylnaphthalene | 90-12-0 | 22 | R-RSL | 7 | 0.010 | 0.003 |
| 2-Methylnaphthalene | 91-57-6 | 310 | R-RSL | 100 | 0.010 | 0.003 |
| Acenaphthene | 83-32-9 | 3400 | R-RSL | 1100 | 0.010 | 0.003 |
| Acenaphthylene [4] | 208-96-8 | 3400 | R-RSL | 1100 | 0.010 | 0.003 |
| Anthracene | 120-12-7 | 17000 | R-RSL | 5700 | 0.010 | 0.003 |
| Benzo(a)anthracene | 56-55-3 | 0.15 | R-RSL | 0.05 | 0.010 | 0.003 |
| Benzo(a)pyrene | 50-32-8 | 0.015 | R-RSL | **0.005** | 0.010 | 0.003 |
| Benzo(b)fluoranthene | 205-99-2 | 0.15 | R-RSL | 0.05 | 0.010 | 0.003 |
| Benzo(g,h,i)perylene [5] | 191-24-2 | 1700 | R-RSL | 570 | 0.010 | 0.003 |
| Benzo(k)fluoranthene | 207-08-9 | 0.15 | R-RSL | 0.5 | 0.010 | 0.003 |
| Chrysene | 218-01-9 | 15 | R-RSL | 5 | 0.010 | 0.003 |
| Dibenzo(a,h)anthracene | 53-70-3 | 0.015 | R-RSL | **0.005** | 0.010 | 0.003 |
| Fluoranthene | 206-44-0 | 2300 | R-RSL | 770 | 0.010 | 0.003 |
| Fluorene | 86-73-7 | 2300 | R-RSL | 770 | 0.010 | 0.003 |
| Indeno(1,2,3-cd)pyrene | 193-39-5 | 0.15 | R-RSL | 0.05 | 0.010 | 0.003 |
| Naphthalene | 91-20-3 | 3.9 | R-RSL | 1 | 0.010 | 0.003 |
| Phenanthrene[5] | 85-01-8 | 1700 | R-RSL | 570 | 0.010 | 0.003 |
| Pyrene | 129-00-0 | 1700 | R-RSL | 570 | 0.010 | 0.003 |

**Matrix: Soil**

**Analytical Group: Nitroglycerin**

| Analyte | CAS Number | Project Action Limit[1] (mg/kg) | Project Action Limit Reference | Project Quantitation Limit Goal (mg/kg) | Laboratory-Specific[6] | |
|---|---|---|---|---|---|---|
| | | | | | Quantitation Limit (mg/kg) | Method Detection Limit (mg/kg) |
| Nitroglycerin | 55-63-0 | 6.1 | R-RSL | **2.0** | 4.0 | 0.65 |

A bolded cell indicates that the project quantitation limit goal is less than the laboratory quantitation limit.

Site background concentrations were not included in Worksheet #15 tables because in all cases project action limits were greater than corresponding site background concentrations.

Acyonms

CAS = Chemical Abstracts Service

mg/kg = Milligrams per kilogram.

N/A = Not applicable

R-RSL = Residential – Regional Screening Level

Footnotes

1 USEPA Regional Screening Levels for Chemical Contaminants at Superfund Sites (ORNL, April 2009).

2 Empirical Laboratories will be performing lead and PAH analyses for this project.

3 PALs were determined by dividing the R-RSL for lead by 2 to provide a conservative margin of error.

4 The value for acenaphthene has been used as a surrogate for acenaphthylene.

5 The value for pyrene has been used as a surrogate for benzo(g,h,i)perylene and phenanthrene.

6 GPL Laboratories will be performing nitroglycerin analyses for this project.

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

(UFP-QAPP Manual Section 2.8.2)

| Activity | Organization | Dates (MM/DD/YY) | | Deliverable | Delivery Due Date |
|---|---|---|---|---|---|
| | | Anticipated Date(s) of Initiation | Anticipated Date of Completion | | |
| Prepare Rough Draft SIs SAP | Tetra Tech | 06/16/08 | 08/17/09 | NA | NA |
| Submit Rough Draft SIs SAP | Tetra Tech | 08/17/09 | 08/17/09 | Rough Draft SIs Work Plan | 08/17/09 |
| Navy Review | Navy | 08/18/09 | 11/19/09 | NA | NA |
| Prepare Draft SIs SAP | Tetra Tech | 11/20/09 | 12/01/09 | NA | NA |
| Submit Draft SIs SAP | Tetra Tech | 12/01/09 | 12/01/09 | Draft SIs Work Plan | 12/01/09 |
| Regulator Review | USEPA and TDEC | 12/01/09 | 01/29/10 | NA | NA |
| Receive Comments/Comment Resolution | Tetra Tech | 01/29/10 | 02/05/10 | Comments | NA |
| Prepare Final SIs SAP | Tetra Tech | 01/29/10 | 02/05/10 | NA | NA |
| Submit Final SIs SAP | Tetra Tech | 02/05/10 | 02/05/10 | Final SIs Work Plan | 02/05/10 |
| Mobilization and Field Investigation | Tetra Tech | 02/15/10 | 02/24/10 | NA | NA |
| Complete Field Investigation and Demobilization | Tetra Tech | 02/24/10 | 02/24/10 | NA | NA |
| Laboratory Analysis | Empirical Laboratories | 02/15/10 | 03/16/10 | Data Package | NA |
| Laboratory Analysis (NG only) | GPL Laboratories | 02/15/10 | 03/16/10 | Data Package | NA |
| Data Validation | Tetra Tech | 03/1710 | 03/30/10 | Data Validation Letter | NA |

| Activity | Organization | Dates (MM/DD/YY) | | Deliverable | Delivery Due Date |
|---|---|---|---|---|---|
| | | Anticipated Date(s) of Initiation | Anticipated Date of Completion | | |
| Database Entry | Tetra Tech | 03/31/10 | 04/06/10 | NA | NA |
| Prepare Rough Draft SIs Report | Tetra Tech | 03/31/10 | 05/07/10 | NA | NA |
| Submit Rough Draft SIs Report | Tetra Tech | 05/07/10 | 05/07/10 | Rough Draft SIs Report | 05/07/10 |
| Navy Review | Navy | 05/08/10 | 05/21/10 | NA | NA |
| Prepare Draft SIs Report | Tetra Tech | 05/24/10 | 06/04/10 | NA | NA |
| Submit Draft SIs Report | Tetra Tech | 06/04/10 | 06/04/10 | Draft SIs Report | 06/04/10 |
| Regulator Review | USEPA and TDEC | 06/05/10 | 07/19/10 | NA | NA |
| Receive Comments/Comment Resolution | Tetra Tech | 07/20/10 | 08/02/10 | Comments | Comments |
| Prepare Final SIs Report | Tetra Tech | 08/03/10 | 08/23/10 | NA | NA |
| Submit Final SIs Report | Tetra Tech | 08/23/10 | 08/23/10 | Final SIs Report | 08/23/10 |

## SAP Worksheet #17 -- Sampling Design and Rationale

(UFP-QAPP Manual Section 3.1.1)

### SAMPLING LOCATIONS, ANALYSES, AND RATIONALES

This section identifies sampling locations, QA/QC samples to be collected, analyses to be performed, and rationales for the sampling and analytical programs. All soil samples selected for laboratory analyses will be prepared and analyzed according to laboratory protocol as identified on Worksheet #30. Proposed sampling locations are shown on the following figures for each of the seven sites:

- Figure 17.1, Pistol Range
- Figure 17.2, Rifle Range
- Figure 17.3, Aircraft Firing Range
- Figure 17.4, Trap/Skeet Ranges #1 and #2
- Figure 17.5, Horse Stables Skeet Range #1
- Figure 17.6, Horse Stables Skeet Range #2

The total numbers of soil analyses for each analyte group are tabulated in Worksheet #18 and #20. Soil QA/QC samples will be collected at frequencies listed in Worksheet #20. Worksheet #19 presents a summary of the sample analyses, container types and volumes, preservation requirements, and holding times for the samples to be collected.

### Soil Sampling Strategies

The chosen sampling strategies employ a judgmental design based on a sampling grid or linear sampling transects to target those areas that are most likely to be contaminated according to the CSMs presented in Appendix A and summarized in Worksheet #10. This is a conservative strategy. The data collected under this strategy will probably not represent the concentrations to which human or ecological receptors would actually be exposed. Instead, the measured concentrations are likely to be greater than actual exposure concentrations. The strategy therefore attempts to ensure that a potential environmental problem is not overlooked. Samples will also be collected from areas outside of those expected to be contaminated to provide the spatial coverage needed to validate the sampling design and CSMs. If the CSMs are correct, the areas outside of those expected to be contaminated will exhibit significantly lower, and perhaps even non-detectable, concentrations of MC than in the targeted contamination areas. It is anticipated that surface soil sampling will be performed using hand auger soil sampling techniques. If field conditions necessitate, DPT sampling may be performed. All soil borings will be logged to document

lithology. Details regarding soil sampling equipment and procedures are included in Worksheet #14 and SOP-05.

All soil borings will initially be advanced to a maximum depth of 1 foot bgs, and one soil sample will be collected from each boring and analyzed for lead in the field using XRF equipment in accordance with SOP-13. The sample will consist of soil from the entire 0- to 1-foot depth interval.

Actual sample locations may vary from the proposed locations based on field conditions as observed during an initial site walkover by the FOL. The FOL may bias the actual sampling locations toward areas most likely to be contaminated based on professional judgment and the initial site walkover. Professional judgment will take into account the topography of the site and the FOL's understanding of site operations. GPS measurements will be taken at each individual sample location, which will allow for future repeatable investigations or as a guide in any remedial action. During the processing of samples for XRF field analysis, any observed lead bullets, bullet fragments, or lead shot will be removed from the sample so as not to bias the sample with a high lead result. The sampling objective is to determine metals concentrations in soil as a result of potential leaching of contaminants from the bullets. However, the presence of bullets, bullet fragments, or lead will be described in the individual field sample log sheets (see SOP-03) and will also be used as the basis for collection of "step-out" samples (horizontal or vertical) even if XRF results from the surface samples are less than the 280 mg/kg threshold. All visual observations will be noted on the soil sample log. All bullets, bullet fragments, or lead collected from any of the seven sites will be containerized for later off-site disposal.

The field XRF data for lead and laboratory data for NG, lead, and PAHs are intended to be used to establish the nature and extent of contamination and the potential environmental mobility of contaminants. Lead is the major contaminant of concern at all sites and will be used as an indicator of the potential presence of additional inorganic contaminants. Fixed-base laboratory data will be used to establish a correlation between field XRF results and laboratory data for lead. Soil properties (pH, TOC, and CEC) are principal determinants of environmental mobility, therefore, select soil samples at each site will be analyzed for pH, TOC, and CEC.

Samples submitted to the fixed-base laboratory for lead analysis will be selected to represent the full range of lead concentrations, with emphasis on samples with field XRF lead concentrations between 280 and 500 mg/kg. Samples for PAH analysis by the fixed-base laboratory will be collected at locations where PAH contamination is anticipated based upon knowledge of historical operations (e.g., expected fallout zones) and/or locations where skeet fragments are observed during sample collection. The samples selected for PAH analysis will not necessarily be from the same locations as samples selected for laboratory led analysis. Samples for NG analysis will be collected at the firing lines because NG in the

form of smokeless powder may be present at the firing points as a result of ejection of unburned propellants. Firing line samples will also be analyzed for lead associated with lead-azide initiating compound.

**Pistol Range**

Up to 75 surface soil samples are proposed to be collected at the Pistol Range, as shown on Figure 17.1. Surface soil samples will be collected at the 0- to 1-foot level over a grid as shown on Figure 17.1 (grid node spacing is approximately 20 feet). This grid was designed to cover the former berm area, firing area, and range floor directly in front of the firing line.

All samples collected will be analyzed in the field for lead utilizing portable XRF equipment. A minimum of 20 samples will then be selected for fixed-base laboratory analysis for lead. One composite surface soil sample (comprised of 10 grab samples) will be collected from the firing line for fixed-base laboratory NG and lead analyses.

**Rifle Range**

Up to 75 surface soil samples are proposed to be collected at the Rifle Range, as shown on Figure 17.2. Surface soil samples will be collected from the 0- to 1-foot level over a grid as shown on Figure 17.2 (grid node spacing is approximately 20 feet) and from the three former firing lines.

All samples collected will be analyzed in the field for lead utilizing the portable XRF equipment. A minimum of 20 samples will then be selected for fixed-base laboratory analysis for lead. Three composite surface soil samples (comprised of 10 grab samples each) will be collected, one from each firing line, for fixed-base laboratory NG and lead analyses.

**Aircraft Firing Range**

Up to 75 surface soil samples are proposed to be collected at the Aircraft Firing Range, as shown on Figure 17.3. Surface soil samples will be collected at the 0- to 1-foot level over a grid as shown on Figure 17.3 (grid node spacing is approximately 20 feet). This grid was designed to cover the former berm area, firing area, and range floor directly in front of the firing line.

All samples collected will be analyzed in the field for lead utilizing portable XRF equipment. A minimum of 20 samples will then be selected for fixed-base laboratory analysis for lead. Two composite surface soil samples will be collected from the western firing line for fixed-base laboratory NG and lead analyses.

Due to excessive disturbance of the eastern firing line and the fact that it is currently paved with asphalt, the samples from the western firing line will serve as the representative for both firing lines.

**Trap/Skeet Ranges #1 and # 2**

Up to 75 surface soil samples are proposed to be collected at the Trap/Skeet Ranges #1 and #2 as shown on Figure 17.4. Surface soil samples from the 0- to 1- foot level will be collected along transect lines focused on the expected fall-out area. This sampling distribution covers the expected location of maximum shot fall-out.

All samples collected will be analyzed in the field for lead utilizing portable XRF equipment. A minimum of 20 samples will then be selected for fixed-base laboratory analysis for lead. A minimum of 20 of the 75 surface soil samples will be selected for fixed-based laboratory PAH analysis. These samples will not necessarily be from the same locations as the samples submitted for lead analysis. Ten composite surface soil samples (comprised of 10 grab samples each) will be collected along the firing lines for fixed-base laboratory NG and lead analyses.

**Horse Stables Skeet Range #1**

Up to 75 surface soil samples are proposed to be collected at the Horse Stables Skeet Range #1, as shown on Figure 17.6. Surface soil samples form the 0- to 1- foot level will be collected over a weighted grid interval focused on the expected fall-out area. This sampling distribution covers the expected location of maximum shot fall-out.

All samples collected will be analyzed in the field for lead utilizing portable XRF equipment. A minimum of 20 samples will then be selected for fixed-base laboratory analysis for lead. A minimum of 20 of the 75 surface soil samples will be selected for fixed-based laboratory PAH analysis. These samples will not necessarily be from the same locations as the samples submitted for lead analysis. Two composite surface soil samples (comprised of 10 grab samples each) will be collected from the firing line for fixed-base laboratory NG and lead analyses.

**Horse Stables Skeet Range #2**

Up to 75 surface soil samples are proposed to be collected at the Horse Stables Skeet Range #2, as shown on Figure 17.6. Surface soil samples from the 0- to 1- foot level will be collected along transect lines focused on the expected fall-out area. This sampling distribution covers the expected location of maximum shot fall-out.

All samples collected will be analyzed in the field for lead utilizing portable XRF equipment. A minimum of 20 samples will then be selected for fixed-base laboratory analysis for lead. A minimum of 20 of the 75 surface soil samples will be selected for fixed-based laboratory PAH analysis. These samples will not necessarily be from the same locations as the samples submitted for lead analysis. Two composite surface soil samples (comprised of 10 grab samples each) will be collected from the firing line for fixed-base laboratory NG and lead analyses.

**SAP Worksheet #18.1 -- Sampling Locations and Methods/SOP Requirements Table**

(UFP-QAPP Manual Section 3.1.1)

**PISTOL RANGE**

| Sample Location[1] | Sample ID[2] | Lead[3] | | Nitroglycerin[3,4] | pH[5] |
|---|---|---|---|---|---|
| | | Field XRF (Lead Only) | SW-846 3050B/6010B (Lead) | SW-846 8330B | SW-846 9045C |
| PRSS 001 | PRSS 0010001 | 1 | TBD | TBD | TBD |
| PRSS 002 | PRSS 0020001 | 1 | TBD | TBD | TBD |
| PRSS 003 | PRSS 0030001 | 1 | TBD | TBD | TBD |
| PRSS 004 | PRSS 0040001 | 1 | TBD | TBD | TBD |
| PRSS 005 | PRSS 0050001 | 1 | TBD | TBD | TBD |
| PRSS 006 | PRSS 0060001 | 1 | TBD | TBD | TBD |
| PRSS 007 | PRSS 0070001 | 1 | TBD | TBD | TBD |
| PRSS 008 | PRSS 0080001 | 1 | TBD | TBD | TBD |
| PRSS 009 | PRSS 0090001 | 1 | TBD | TBD | TBD |
| PRSS 010 | PRSS 0100001 | 1 | TBD | TBD | TBD |
| PRSS 011 | PRSS 0110001 | 1 | TBD | TBD | TBD |
| PRSS 012 | PRSS 0120001 | 1 | TBD | TBD | TBD |
| PRSS 013 | PRSS 0130001 | 1 | TBD | TBD | TBD |
| PRSS 014 | PRSS 0140001 | 1 | TBD | TBD | TBD |
| PRSS 015 | PRSS 0150001 | 1 | TBD | TBD | TBD |
| PRSS 016 | PRSS 0160001 | 1 | TBD | TBD | TBD |
| PRSS 017 | PRSS 0170001 | 1 | TBD | TBD | TBD |
| PRSS 018 | PRSS 0180001 | 1 | TBD | TBD | TBD |
| PRSS 019 | PRSS 0190001 | 1 | TBD | TBD | TBD |
| PRSS 020 | PRSS 0200001 | 1 | TBD | TBD | TBD |
| Field Duplicate | | -- | TBD | TBD | TBD |
| PRSS 021 | PRSS 0210001 | 1 | TBD | TBD | TBD |
| PRSS 022 | PRSS 0220001 | 1 | TBD | TBD | TBD |

| Sample Location[1] | Sample ID[2] | Lead[3] | | Nitroglycerin[3,4] | pH[5] |
|---|---|---|---|---|---|
| | | Field XRF (Lead Only) | SW-846 3050B/6010B (Lead) | SW-846 8330B | SW-846 9045C |
| PRSS 023 | PRSS 0230001 | 1 | TBD | TBD | TBD |
| PRSS 024 | PRSS 0240001 | 1 | TBD | TBD | TBD |
| PRSS 025 | PRSS 0250001 | 1 | TBD | TBD | TBD |
| PRSS 026 | PRSS 0260001 | 1 | TBD | TBD | TBD |
| PRSS 027 | PRSS 0270001 | 1 | TBD | TBD | TBD |
| PRSS 028 | PRSS 0280001 | 1 | TBD | TBD | TBD |
| PRSS 029 | PRSS 0290001 | 1 | TBD | TBD | TBD |
| PRSS 030 | PRSS 0300001 | 1 | TBD | TBD | TBD |
| PRSS 031 | PRSS 0310001 | 1 | TBD | TBD | TBD |
| PRSS 032 | PRSS 0320001 | 1 | TBD | TBD | TBD |
| PRSS 033 | PRSS 0330001 | 1 | TBD | TBD | TBD |
| PRSS 034 | PRSS 0340001 | 1 | TBD | TBD | TBD |
| PRSS 035 | PRSS 0350001 | 1 | TBD | TBD | TBD |
| PRSS 036 | PRSS 0360001 | 1 | TBD | TBD | TBD |
| PRSS 037 | PRSS 0370001 | 1 | TBD | TBD | TBD |
| PRSS 038 | PRSS 0380001 | 1 | TBD | TBD | TBD |
| PRSS 039 | PRSS 0390001 | 1 | TBD | TBD | TBD |
| PRSS 040 | PRSS 0400001 | 1 | TBD | TBD | TBD |
| Field Duplicate | | -- | TBD | TBD | TBD |
| PRSS 041 | PRSS 0410001 | 1 | TBD | TBD | TBD |
| PRSS 042 | PRSS 0420001 | 1 | TBD | TBD | TBD |
| PRSS 043 | PRSS 0430001 | 1 | TBD | TBD | TBD |
| PRSS 044 | PRSS 0440001 | 1 | TBD | TBD | TBD |
| PRSS 045 | PRSS 0450001 | 1 | TBD | TBD | TBD |
| PRSS 046 | PRSS 0460001 | 1 | TBD | TBD | TBD |
| PRSS 047 | PRSS 0470001 | 1 | TBD | TBD | TBD |
| PRSS 048 | PRSS 0480001 | 1 | TBD | TBD | TBD |

| Sample Location[1] | Sample ID[2] | Lead[3] | | Nitroglycerin[3,4] | pH[5] |
|---|---|---|---|---|---|
| | | Field XRF (Lead Only) | SW-846 3050B/6010B (Lead) | SW-846 8330B | SW-846 9045C |
| PRSS 049 | PRSS 0490001 | 1 | TBD | TBD | TBD |
| PRSS 050 | PRSS 0500001 | 1 | TBD | TBD | TBD |
| PRSS 051 | PRSS 0510001 | 1 | TBD | TBD | TBD |
| PRSS 052 | PRSS 0520001 | 1 | TBD | TBD | TBD |
| PRSS 053 | PRSS 0530001 | 1 | TBD | TBD | TBD |
| PRSS 054 | PRSS 0540001 | 1 | TBD | TBD | TBD |
| PRSS 055 | PRSS 0550001 | 1 | TBD | TBD | TBD |
| PRSS 056 | PRSS 0560001 | 1 | TBD | TBD | TBD |
| PRSS 057 | PRSS 0570001 | 1 | TBD | TBD | TBD |
| PRSS 058 | PRSS 0580001 | 1 | TBD | TBD | TBD |
| PRSS 059 | PRSS 0590001 | 1 | TBD | TBD | TBD |
| PRSS 060 | PRSS 0600001 | 1 | TBD | TBD | TBD |
| Field Duplicate | | -- | TBD | TBD | TBD |
| PRSS 061 | PRSS 0610001 | 1 | TBD | TBD | TBD |
| PRSS 062 | PRSS 0620001 | 1 | TBD | TBD | TBD |
| PRSS 063 | PRSS 0630001 | 1 | TBD | TBD | TBD |
| PRSS 064 | PRSS 0640001 | 1 | TBD | TBD | TBD |
| PRSS 065 | PRSS 0650001 | 1 | TBD | TBD | TBD |
| PRSS 066 | PRSS 0660001 | 1 | TBD | TBD | TBD |
| PRSS 067 | PRSS 0670001 | 1 | TBD | TBD | TBD |
| PRSS 068 | PRSS 0680001 | 1 | TBD | TBD | TBD |
| PRSS 069 | PRSS 0690001 | 1 | TBD | TBD | TBD |
| PRSS 070 | PRSS 0700001 | 1 | TBD | TBD | TBD |
| Field Duplicate | | -- | TBD | TBD | TBD |
| PRSS 071 | PRSS 0710001 | 1 | TBD | TBD | TBD |
| PRSS 072 | PRSS 0720001 | 1 | TBD | TBD | TBD |
| PRSS 073 | PRSS 0730001 | 1 | TBD | TBD | TBD |

| Sample Location[1] | Sample ID[2] | Lead[3] | | Nitroglycerin[3,4] | pH[5] |
|---|---|---|---|---|---|
| | | Field XRF (Lead Only) | SW-846 3050B/6010B (Lead) | SW-846 8330B | SW-846 9045C |
| PRSS 074 | PRSS 0740001 | 1 | TBD | TBD | TBD |
| PRSS 075 | PRSS 0750001 | 1 | TBD | TBD | TBD |
| **Total Soil Samples** | | 75 | **TBD** | **TBD** | **TBD** |

PR=Pistol Range

SS=Surface Soil Sample

1 Surface soil samples will be will be collected over a grid focused on the expected fall-out area. All samples will be collected in accordance with SOP-05. Proposed sample locations are shown on Figure 17.1.

2 The sample ID number will consist of, in order, site acronym, sample matrix, sample location number, and sequential depth interval. Sample depth to a maximum of 1-foot bgs for surface samples.

3 Dependent upon field XRF screening results, a minimum of 20 samples will be selected for fixed-base laboratory lead analysis

4 One composite surface soil sample along the firing line will be submitted for fixed-base laboratory NG and lead analyses.

5 At each site, the field geologist will select up to three of the samples submitted to the fixed-base-laboratory for pH, TOC, and CEC analysis

## SAP Worksheet #18.2 -- Sampling Locations and Methods/SOP Requirements Table

(UFP-QAPP Manual Section 3.1.1)

### RIFLE RANGE

| Sample Location[1] | Sample ID[2] | Lead[3] | | Nitroglycerin[3,4] | pH[5] |
|---|---|---|---|---|---|
| | | Field XRF (Lead Only) | SW-846 3050B/6010B (Lead) | SW-846 8330B | SW-846 9045C |
| RRSS 001 | RRSS 0010001 | 1 | TBD | TBD | TBD |
| RRSS 002 | RRSS 0020001 | 1 | TBD | TBD | TBD |
| RRSS 003 | RRSS 0030001 | 1 | TBD | TBD | TBD |
| RRSS 004 | RRSS 0040001 | 1 | TBD | TBD | TBD |
| RRSS 005 | RRSS 0050001 | 1 | TBD | TBD | TBD |
| RRSS 006 | RRSS 0060001 | 1 | TBD | TBD | TBD |
| RRSS 007 | RRSS 0070001 | 1 | TBD | TBD | TBD |
| RRSS 008 | RRSS 0080001 | 1 | TBD | TBD | TBD |
| RRSS 009 | RRSS 0090001 | 1 | TBD | TBD | TBD |
| RRSS 010 | RRSS 0100001 | 1 | TBD | TBD | TBD |
| RRSS 011 | RRSS 0110001 | 1 | TBD | TBD | TBD |
| RRSS 012 | RRSS 0120001 | 1 | TBD | TBD | TBD |
| RRSS 013 | RRSS 0130001 | 1 | TBD | TBD | TBD |
| RRSS 014 | RRSS 0140001 | 1 | TBD | TBD | TBD |
| RRSS 015 | RRSS 0150001 | 1 | TBD | TBD | TBD |
| RRSS 016 | RRSS 0160001 | 1 | TBD | TBD | TBD |
| RRSS 017 | RRSS 0170001 | 1 | TBD | TBD | TBD |
| RRSS 018 | RRSS 0180001 | 1 | TBD | TBD | TBD |
| RRSS 019 | RRSS 0190001 | 1 | TBD | TBD | TBD |
| RRSS 020 | RRSS 0200001 | 1 | TBD | TBD | TBD |
| Field Duplicate | | -- | TBD | TBD | TBD |
| RRSS 021 | RRSS 0210001 | 1 | TBD | TBD | TBD |
| RRSS 022 | RRSS 0220001 | 1 | TBD | TBD | TBD |

| Sample Location[1] | Sample ID[2] | Lead[3] | | Nitroglycerin[3,4] | pH[5] |
|---|---|---|---|---|---|
| | | Field XRF (Lead Only) | SW-846 3050B/6010B (Lead) | SW-846 8330B | SW-846 9045C |
| RRSS 023 | RRSS 0230001 | 1 | TBD | TBD | TBD |
| RRSS 024 | RRSS 0240001 | 1 | TBD | TBD | TBD |
| RRSS 025 | RRSS 0250001 | 1 | TBD | TBD | TBD |
| RRSS 026 | RRSS 0260001 | 1 | TBD | TBD | TBD |
| RRSS 027 | RRSS 0270001 | 1 | TBD | TBD | TBD |
| RRSS 028 | RRSS 0280001 | 1 | TBD | TBD | TBD |
| RRSS 029 | RRSS 0290001 | 1 | TBD | TBD | TBD |
| RRSS 030 | RRSS 0300001 | 1 | TBD | TBD | TBD |
| RRSS 031 | RRSS 0310001 | 1 | TBD | TBD | TBD |
| RRSS 032 | RRSS 0320001 | 1 | TBD | TBD | TBD |
| RRSS 033 | RRSS 0330001 | 1 | TBD | TBD | TBD |
| RRSS 034 | RRSS 0340001 | 1 | TBD | TBD | TBD |
| RRSS 035 | RRSS 0350001 | 1 | TBD | TBD | TBD |
| RRSS 036 | RRSS 0360001 | 1 | TBD | TBD | TBD |
| RRSS 037 | RRSS 0370001 | 1 | TBD | TBD | TBD |
| RRSS 038 | RRSS 0380001 | 1 | TBD | TBD | TBD |
| RRSS 039 | RRSS 0390001 | 1 | TBD | TBD | TBD |
| RRSS 040 | RRSS 0400001 | 1 | TBD | TBD | TBD |
| Field Duplicate | | 1 | TBD | TBD | TBD |
| RRSS 041 | RRSS 0410001 | 1 | TBD | TBD | TBD |
| RRSS 042 | RRSS 0420001 | 1 | TBD | TBD | TBD |
| RRSS 043 | RRSS 0430001 | 1 | TBD | TBD | TBD |
| RRSS 044 | RRSS 0440001 | 1 | TBD | TBD | TBD |
| RRSS 045 | RRSS 0450001 | 1 | TBD | TBD | TBD |
| RRSS 046 | RRSS 0460001 | 1 | TBD | TBD | TBD |
| RRSS 047 | RRSS 0470001 | 1 | TBD | TBD | TBD |
| RRSS 048 | RRSS 0480001 | 1 | TBD | TBD | TBD |

| Sample Location[1] | Sample ID[2] | Lead[3] | | Nitroglycerin[3,4] | pH[5] |
|---|---|---|---|---|---|
| | | Field XRF (Lead Only) | SW-846 3050B/6010B (Lead) | SW-846 8330B | SW-846 9045C |
| RRSS 049 | RRSS 0490001 | 1 | TBD | TBD | TBD |
| RRSS 050 | RRSS 0500001 | 1 | TBD | TBD | TBD |
| RRSS 051 | RRSS 0510001 | 1 | TBD | TBD | TBD |
| RRSS 052 | RRSS 0520001 | 1 | TBD | TBD | TBD |
| RRSS 053 | RRSS 0530001 | 1 | TBD | TBD | TBD |
| RRSS 054 | RRSS 0540001 | 1 | TBD | TBD | TBD |
| RRSS 055 | RRSS 055001 | 1 | TBD | TBD | TBD |
| RRSS 056 | RRSS 0560001 | 1 | TBD | TBD | TBD |
| RRSS 057 | RRSS 0570001 | 1 | TBD | TBD | TBD |
| RRSS 058 | RRSS 0580001 | 1 | TBD | TBD | TBD |
| RRSS 059 | RRSS 0590001 | 1 | TBD | TBD | TBD |
| RRSS 060 | RRSS 0600001 | 1 | TBD | TBD | TBD |
| Field Duplicate | | -- | TBD | TBD | TBD |
| RRSS 061 | RRSS 0610001 | 1 | TBD | TBD | TBD |
| RRSS 062 | RRSS 0620001 | 1 | TBD | TBD | TBD |
| RRSS 063 | RRSS 0630001 | 1 | TBD | TBD | TBD |
| RRSS 064 | RRSS 0640001 | 1 | TBD | TBD | TBD |
| RRSS 065 | RRSS 0650001 | 1 | TBD | TBD | TBD |
| RRSS 066 | RRSS 0660001 | 1 | TBD | TBD | TBD |
| RRSS 067 | RRSS 0670001 | 1 | TBD | TBD | TBD |
| RRSS 068 | RRSS 0680001 | 1 | TBD | TBD | TBD |
| RRSS 069 | RRSS 0690001 | 1 | TBD | TBD | TBD |
| RRSS 070 | RRSS 0700001 | 1 | TBD | TBD | TBD |
| RRSS 071 | RRSS 0710001 | 1 | TBD | TBD | TBD |
| Field Duplicate | | -- | TBD | TBD | TBD |
| RRSS 072 | RRSS 0720001 | 1 | TBD | TBD | TBD |
| RRSS 073 | RRSS 0730001 | 1 | TBD | TBD | TBD |

| Sample Location[1] | Sample ID[2] | Lead[3] | | Nitroglycerin[3,4] | pH[5] |
|---|---|---|---|---|---|
| | | Field XRF (Lead Only) | SW-846 3050B/6010B (Lead) | SW-846 8330B | SW-846 9045C |
| RRSS 074 | RRSS 0740001 | 1 | TBD | TBD | TBD |
| RRSS 075 | RRSS 0750001 | 1 | TBD | TBD | TBD |
| **Total Soil Samples** | | **75** | **TBD** | **TBD** | **TBD** |

RR=Rifle Range

SS=Surface Soil Sample

1 Surface soil samples will be collected over a grid focused on the expected fall-out area. All samples will be collected in accordance with SOP-05. Proposed sample locations are shown on Figure 17.2.

2 The sample ID number will consist of, in order, site acronym, sample matrix, sample location number, and sequential depth interval. Sample depth to a maximum of 1-foot bgs for surface samples.

3 Dependent upon field XRF screening results, a minimum of 20 samples will be selected for fixed-base laboratory lead analysis.

4 Three composite surface soil samples along the firing lines will be submitted for fixed-base laboratory NG and lead analyses.

5 At each site the field geologist will select up to three of the samples submitted to the fixed-base-laboratory for pH, TOC, and CEC analyses.

**SAP Worksheet #18.3 -- Sampling Locations and Methods/SOP Requirements Table**

(UFP-QAPP Manual Section 3.1.1)

**Aircraft Firing Range**

| Sample Location[1] | Sample ID[2] | Lead[3] | | Nitroglycerin[3,4] | pH[5] |
|---|---|---|---|---|---|
| | | Field XRF (Lead Only) | SW-846 3050B/6010B (Lead) | SW-846 8330B | SW-846 9045C |
| AFRSS 001 | AFRSS 0010001 | TBD | TBD | TBD | TBD |
| AFRSS 002 | AFRSS 0020001 | TBD | TBD | TBD | TBD |
| AFRSS 003 | AFRSS 0030001 | TBD | TBD | TBD | TBD |
| AFRSS 004 | AFRSS 0040001 | TBD | TBD | TBD | TBD |
| AFRSS 005 | AFRSS 0050001 | TBD | TBD | TBD | TBD |
| AFRSS 006 | AFRSS 0060001 | TBD | TBD | TBD | TBD |
| AFRSS 007 | AFRSS 0070001 | TBD | TBD | TBD | TBD |
| AFRSS 008 | AFRSS 0080001 | TBD | TBD | TBD | TBD |
| AFRSS 009 | AFRSS 0090001 | TBD | TBD | TBD | TBD |
| AFRSS 010 | AFRSS 0100001 | TBD | TBD | TBD | TBD |
| AFRSS 011 | AFRSS 0110001 | TBD | TBD | TBD | TBD |
| AFRSS 012 | AFRSS 0120001 | TBD | TBD | TBD | TBD |
| AFRSS 013 | AFRSS 0130001 | TBD | TBD | TBD | TBD |
| AFRSS 014 | AFRSS 0140001 | TBD | TBD | TBD | TBD |
| AFRSS 015 | AFRSS 0150001 | TBD | TBD | TBD | TBD |
| AFRSS 016 | AFRSS 0160001 | TBD | TBD | TBD | TBD |
| AFRSS 017 | AFRSS 0170001 | TBD | TBD | TBD | TBD |
| AFRSS 018 | AFRSS 0180001 | TBD | TBD | TBD | TBD |
| AFRSS 019 | AFRSS 0190001 | TBD | TBD | TBD | TBD |
| AFRSS 020 | AFRSS 0200001 | TBD | TBD | TBD | TBD |
| Field Duplicate | | -- | TBD | TBD | TBD |
| AFRSS 021 | AFRSS 0210001 | TBD | TBD | TBD | TBD |
| AFRSS 022 | AFRSS 0220001 | TBD | TBD | TBD | TBD |

| Sample Location[1] | Sample ID[2] | Lead[3] | | Nitroglycerin[3,4] | pH[5] |
|---|---|---|---|---|---|
| | | Field XRF (Lead Only) | SW-846 3050B/6010B (Lead) | SW-846 8330B | SW-846 9045C |
| AFRSS 023 | AFRSS 0230001 | TBD | TBD | TBD | TBD |
| AFRSS 024 | AFRSS 0240001 | TBD | TBD | TBD | TBD |
| AFRSS 025 | AFRSS 0250001 | TBD | TBD | TBD | TBD |
| AFRSS 026 | AFRSS 0260001 | TBD | TBD | TBD | TBD |
| AFRSS 027 | AFRSS 0270001 | TBD | TBD | TBD | TBD |
| AFRSS 028 | AFRSS 0280001 | TBD | TBD | TBD | TBD |
| AFRSS 029 | AFRSS 0290001 | TBD | TBD | TBD | TBD |
| AFRSS 030 | AFRSS 0300001 | TBD | TBD | TBD | TBD |
| AFRSS 031 | AFRSS 0310001 | TBD | TBD | TBD | TBD |
| AFRSS 032 | AFRSS 0320001 | TBD | TBD | TBD | TBD |
| AFRSS 033 | AFRSS 0330001 | TBD | TBD | TBD | TBD |
| AFRSS 034 | AFRSS 0340001 | TBD | TBD | TBD | TBD |
| AFRSS 035 | AFRSS 0350001 | TBD | TBD | TBD | TBD |
| AFRSS 036 | AFRSS 0360001 | TBD | TBD | TBD | TBD |
| AFRSS 037 | AFRSS 0370001 | TBD | TBD | TBD | TBD |
| AFRSS 038 | AFRSS 0380001 | TBD | TBD | TBD | TBD |
| AFRSS 039 | AFRSS 0390001 | TBD | TBD | TBD | TBD |
| AFRSS 040 | AFRSS 0400001 | TBD | TBD | TBD | TBD |
| Field Duplicate | | -- | TBD | TBD | TBD |
| AFRSS 041 | AFRSS 0410001 | TBD | TBD | TBD | TBD |
| AFRSS 042 | AFRSS 0420001 | TBD | TBD | TBD | TBD |
| AFRSS 043 | AFRSS 0430001 | TBD | TBD | TBD | TBD |
| AFRSS 044 | AFRSS 0440001 | TBD | TBD | TBD | TBD |
| AFRSS 045 | AFRSS 0450001 | TBD | TBD | TBD | TBD |
| AFRSS 046 | AFRSS 0460001 | TBD | TBD | TBD | TBD |
| AFRSS 047 | AFRSS 0470001 | TBD | TBD | TBD | TBD |
| AFRSS 048 | AFRSS 0480001 | TBD | TBD | TBD | TBD |

| Sample Location[1] | Sample ID[2] | Lead[3] | | Nitroglycerin[3,4] | pH[5] |
|---|---|---|---|---|---|
| | | Field XRF (Lead Only) | SW-846 3050B/6010B (Lead) | SW-846 8330B | SW-846 9045C |
| AFRSS 049 | AFRSS 0490001 | TBD | TBD | TBD | TBD |
| AFRSS 050 | AFRSS 0500001 | TBD | TBD | TBD | TBD |
| AFRSS 051 | AFRSS 0510001 | TBD | TBD | TBD | TBD |
| AFRSS 052 | AFRSS 0520001 | TBD | TBD | TBD | TBD |
| AFRSS 053 | AFRSS 0530001 | TBD | TBD | TBD | TBD |
| AFRSS 054 | AFRSS 0540001 | TBD | TBD | TBD | TBD |
| AFRSS 055 | AFRSS 0550001 | TBD | TBD | TBD | TBD |
| AFRSS 056 | AFRSS 0560001 | TBD | TBD | TBD | TBD |
| AFRSS 057 | AFRSS 0570001 | TBD | TBD | TBD | TBD |
| AFRSS 058 | AFRSS 0580001 | TBD | TBD | TBD | TBD |
| AFRSS 059 | AFRSS 0590001 | TBD | TBD | TBD | TBD |
| AFRSS 060 | AFRSS 0600001 | TBD | TBD | TBD | TBD |
| Field Duplicate | | -- | TBD | TBD | TBD |
| AFRSS 061 | AFRSS 0610001 | TBD | TBD | TBD | TBD |
| AFRSS 062 | AFRSS 0620001 | TBD | TBD | TBD | TBD |
| AFRSS 063 | AFRSS 0630001 | TBD | TBD | TBD | TBD |
| AFRSS 064 | AFRSS 0640001 | TBD | TBD | TBD | TBD |
| AFRSS 065 | AFRSS 0650001 | TBD | TBD | TBD | TBD |
| AFRSS 066 | AFRSS 0660001 | TBD | TBD | TBD | TBD |
| AFRSS 067 | AFRSS 0670001 | TBD | TBD | TBD | TBD |
| AFRSS 068 | AFRSS 0680001 | TBD | TBD | TBD | TBD |
| AFRSS 069 | AFRSS 0690001 | TBD | TBD | TBD | TBD |
| AFRSS 070 | AFRSS 0700001 | TBD | TBD | TBD | TBD |
| Field Duplicate | | -- | TBD | TBD | TBD |
| AFRSS 071 | AFRSS 0710001 | TBD | TBD | TBD | TBD |
| AFRSS 072 | AFRSS 0720001 | TBD | TBD | TBD | TBD |
| AFRSS 073 | AFRSS 0730001 | TBD | TBD | TBD | TBD |

| Sample Location[1] | Sample ID[2] | Lead[3] | | Nitroglycerin[3,4] | pH[5] |
|---|---|---|---|---|---|
| | | Field XRF (Lead Only) | SW-846 3050B/6010B (Lead) | SW-846 8330B | SW-846 9045C |
| AFRSS 074 | AFRSS 0740001 | TBD | TBD | TBD | TBD |
| AFRSS 075 | AFRSS 0750001 | TBD | TBD | TBD | TBD |
| **Total Soil Samples** | | **75** | **TBD** | **TBD** | **TBD** |

AFR=Aircraft Firing Range
SS=Surface Soil Sample

1 Surface soil samples will be will be collected over a grid focused on the expected fall-out area. All samples will be collected in accordance with SOP-05. Proposed sample locations are shown on Figure 17.3.

2 The sample ID number will consist of, in order, site acronym, sample matrix, sample location number, and sequential depth interval. Sample depth to a maximum of 1-foot bgs for surface samples.

3 Dependent upon field XRF screening results, a minimum of 20 samples will be selected for fixed-base laboratory lead analysis.

4 Two composite surface soil samples along the western firing line will be submitted for fixed-base laboratory NG and lead analyses.

5 At each site the field geologist will select up to three of the samples submitted to the fixed-base-laboratory for pH, TOC, and CEC analyses.

**SAP Worksheet #18.4 -- Sampling Locations and Methods/SOP Requirements Table**

(UFP-QAPP Manual Section 3.1.1)

**Trap Skeet Ranges #1 and #2**

| Sample Location[1] | Sample ID[2] | Lead[3] | | PAHs[4] | Nitroglycerin[3,5] | pH[6] |
|---|---|---|---|---|---|---|
| | | Field XRF (Lead Only) | SW-846 3050B/6010B (Lead) | SW-846 8270C SIM | SW-846 8330B | SW-846 9045C |
| TSRSS 001 | TSRSS 0010001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 002 | TSRSS 0020001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 003 | TSRSS 0030001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 004 | TSRSS 0040001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 005 | TSRSS 0050001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 006 | TSRSS 0060001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 007 | TSRSS 0070001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 008 | TSRSS 0080001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 009 | TSRSS 0090001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 010 | TSRSS 0100001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 011 | TSRSS 0110001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 012 | TSRSS 0120001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 013 | TSRSS 0130001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 014 | TSRSS 0140001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 015 | TSRSS 0150001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 016 | TSRSS 0160001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 017 | TSRSS 0170001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 018 | TSRSS 0180001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 019 | TSRSS 0190001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 020 | TSRSS 0200001 | 1 | TBD | TBD | TBD | TBD |
| Field Duplicate | | -- | TBD | TBD | TBD | TBD |
| TSRSS 021 | TSRSS 0210001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 022 | TSRSS 0220001 | 1 | TBD | TBD | TBD | TBD |

| Sample Location[1] | Sample ID[2] | Lead[3] | | PAHs[4] | Nitroglycerin[3,5] | pH[6] |
|---|---|---|---|---|---|---|
| | | Field XRF (Lead Only) | SW-846 3050B/6010B (Lead) | SW-846 8270C SIM | SW-846 8330B | SW-846 9045C |
| TSRSS 023 | TSRSS 0230001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 024 | TSRSS 0240001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 025 | TSRSS 0250001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 026 | TSRSS 0260001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 027 | TSRSS 0270001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 028 | TSRSS 0280001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 029 | TSRSS 0290001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 030 | TSRSS 0300001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 031 | TSRSS 0310001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 032 | TSRSS 0320001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 033 | TSRSS 0330001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 034 | TSRSS 0340001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 035 | TSRSS 0350001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 036 | TSRSS 0360001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 037 | TSRSS 0370001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 038 | TSRSS 0380001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 039 | TSRSS 0390001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 040 | TSRSS 0400001 | 1 | TBD | TBD | TBD | TBD |
| Field Duplicate | | -- | TBD | TBD | TBD | TBD |
| TSRSS 041 | TSRSS 0410001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 042 | TSRSS 0420001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 043 | TSRSS 0430001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 044 | TSRSS 0440001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 045 | TSRSS 0450001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 046 | TSRSS 0460001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 047 | TSRSS 0470001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 048 | TSRSS 0480001 | 1 | TBD | TBD | TBD | TBD |

| Sample Location[1] | Sample ID[2] | Lead[3] | | PAHs[4] | Nitroglycerin[3,5] | pH[6] |
|---|---|---|---|---|---|---|
| | | Field XRF (Lead Only) | SW-846 3050B/6010B (Lead) | SW-846 8270C SIM | SW-846 8330B | SW-846 9045C |
| TSRSS 049 | TSRSS 0490001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 050 | TSRSS 0490001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 051 | TSRSS 0510001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 052 | TSRSS 0520001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 053 | TSRSS 0530001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 054 | TSRSS 0540001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 055 | TSRSS 0550001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 056 | TSRSS 0560001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 057 | TSRSS 0570001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 058 | TSRSS 0580001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 059 | TSRSS 0590001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 060 | TSRSS 0600001 | 1 | TBD | TBD | TBD | TBD |
| Field Duplicate | | -- | TBD | TBD | TBD | TBD |
| TSRSS 061 | TSRSS 0610001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 062 | TSRSS 0620001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 063 | TSRSS 0630001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 064 | TSRSS 0640001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 065 | TSRSS 0650001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 066 | TSRSS 0660001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 067 | TSRSS 0670001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 068 | TSRSS 0680001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 069 | TSRSS 0690001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 070 | TSRSS 0700001 | 1 | TBD | TBD | TBD | TBD |
| Field Duplicate | | -- | TBD | TBD | TBD | TBD |
| TSRSS 071 | TSRSS 0710001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 072 | TSRSS 0720001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 073 | TSRSS 0730001 | 1 | TBD | TBD | TBD | TBD |

| Sample Location[1] | Sample ID[2] | Lead[3] | | PAHs[4] | Nitroglycerin[3,5] | pH[6] |
|---|---|---|---|---|---|---|
| | | Field XRF (Lead Only) | SW-846 3050B/6010B (Lead) | SW-846 8270C SIM | SW-846 8330B | SW-846 9045C |
| TSRSS 074 | TSRSS 0740001 | 1 | TBD | TBD | TBD | TBD |
| TSRSS 075 | TSRSS 0750001 | 1 | TBD | TBD | TBD | TBD |
| **Total Soil Samples** | | **75** | **TBD** | **TBD** | **TBD** | **TBD** |

TSR=Trap Skeet Ranges #1 and #2

PAHs=Polynuclear aromatic hydrocarbons

SS=Surface Soil Sample

1 Surface soil samples will be collected along transect lines focused on the expected fall-out area. All samples will be collected in accordance with SOP-05. Proposed sample locations are shown on Figure 17.4.

2 The sample ID number will consist of, in order, site acronym, sample matrix, sample location number, and sequential depth interval. Sample depth to a maximum of 1-foot bgs for surface samples.

3 Dependent upon field XRF screening results, a minimum of 20 samples will be selected for fixed-base laboratory lead analysis.

4 A minimum of 20 samples will be selected for fixed-base laboratory PAH analysis.

5 Ten composite surface soil samples along the firing lines will be submitted for fixed-base laboratory NG and lead analyses.

6 At each site the field geologist will select up to three of the samples submitted to the fixed-base laboratory for pH, TOC, and CEC analyses.

**SAP Worksheet #18.5-- Sampling Locations and Methods/SOP Requirements Table**

(UFP-QAPP Manual Section 3.1.1)

**HORSE STABLES SKEET RANGE #1**

| Sample Location[1] | Sample ID[2] | Lead[3] | | PAHs[4] | Nitroglycerin[3,5] | pH[6] |
|---|---|---|---|---|---|---|
| | | Field XRF (Lead Only) | SW-846 3050B/6010B (Lead) | SW-846 8270C SIM | SW-846 8330B | SW-846 9045C |
| HSR1SS 001 | HSR1SS 0010001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 002 | HSR1SS 0020001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 003 | HSR1SS 0030001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 004 | HSR1SS 0040001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 005 | HSR1SS 0050001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 006 | HSR1SS 0060001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 007 | HSR1SS 0070001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 008 | HSR1SS 0080001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 009 | HSR1SS 0090001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 010 | HSR1SS 0100001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 011 | HSR1SS 0110001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 012 | HSR1SS 0120001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 013 | HSR1SS 0130001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 014 | HSR1SS 0140001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 015 | HSR1SS 0150001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 016 | HSR1SS 0160001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 017 | HSR1SS 0170001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 018 | HSR1SS 0180001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 019 | HSR1SS 0190001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 020 | HSR1SS 0200001 | 1 | TBD | TBD | TBD | TBD |
| Field Duplicate | | -- | TBD | TBD | TBD | TBD |
| HSR1SS 021 | HSR1SS 0210001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 022 | HSR1SS 0220001 | 1 | TBD | TBD | TBD | TBD |

| Sample Location[1] | Sample ID[2] | Lead[3] | | PAHs[4] | Nitroglycerin[3,5] | pH[6] |
|---|---|---|---|---|---|---|
| | | Field XRF (Lead Only) | SW-846 3050B/6010B (Lead) | SW-846 8270C SIM | SW-846 8330B | SW-846 9045C |
| HSR1SS 023 | HSR1SS 0230001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 024 | HSR1SS 0240001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 025 | HSR1SS 0250001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 026 | HSR1SS 0260001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 027 | HSR1SS 0270001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 028 | HSR1SS 0280001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 029 | HSR1SS 0290001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 030 | HSR1SS 0300001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 031 | HSR1SS 0310001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 032 | HSR1SS 0320001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 033 | HSR1SS 0330001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 034 | HSR1SS 0340001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 035 | HSR1SS 0350001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 036 | HSR1SS 0360001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 037 | HSR1SS 0370001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 038 | HSR1SS 0380001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 039 | HSR1SS 0390001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 040 | HSR1SS 0400001 | 1 | TBD | TBD | TBD | TBD |
| Field Duplicate | | -- | TBD | TBD | TBD | TBD |
| HSR1SS 041 | HSR1SS 0410001 | TBD | TBD | TBD | TBD | TBD |
| HSR1SS 042 | HSR1SS 0420001 | TBD | TBD | TBD | TBD | TBD |
| HSR1SS 043 | HSR1SS 0430001 | TBD | TBD | TBD | TBD | TBD |
| HSR1SS 044 | HSR1SS 0440001 | TBD | TBD | TBD | TBD | TBD |
| HSR1SS 045 | HSR1SS 0450001 | TBD | TBD | TBD | TBD | TBD |
| HSR1SS 046 | HSR1SS 0460001 | TBD | TBD | TBD | TBD | TBD |
| HSR1SS 047 | HSR1SS 0470001 | TBD | TBD | TBD | TBD | TBD |
| HSR1SS 048 | HSR1SS 0480001 | TBD | TBD | TBD | TBD | TBD |

| Sample Location[1] | Sample ID[2] | Lead[3] | | PAHs[4] | Nitroglycerin[3,5] | pH[6] |
|---|---|---|---|---|---|---|
| | | Field XRF (Lead Only) | SW-846 3050B/6010B (Lead) | SW-846 8270C SIM | SW-846 8330B | SW-846 9045C |
| HSR1SS 049 | HSR1SS 0490001 | TBD | TBD | TBD | TBD | TBD |
| HSR1SS 050 | HSR1SS 0490001 | TBD | TBD | TBD | TBD | TBD |
| HSR1SS 051 | HSR1SS 0510001 | TBD | TBD | TBD | TBD | TBD |
| HSR1SS 052 | HSR1SS 0520001 | TBD | TBD | TBD | TBD | TBD |
| HSR1SS 053 | HSR1SS 0530001 | TBD | TBD | TBD | TBD | TBD |
| HSR1SS 054 | HSR1SS 0540001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 055 | HSR1SS 0550001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 056 | HSR1SS 0560001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 057 | HSR1SS 0570001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 058 | HSR1SS 0580001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 059 | HSR1SS 0590001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 060 | HSR1SS 0600001 | 1 | TBD | TBD | TBD | TBD |
| Field Duplicate | | -- | TBD | TBD | TBD | TBD |
| HSR1SS 061 | HSR1SS 0610001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 062 | HSR1SS 0620001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 063 | HSR1SS 0630001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 064 | HSR1SS 0640001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 065 | HSR1SS 0650001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 066 | HSR1SS 0660001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 067 | HSR1SS 0670001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 068 | HSR1SS 0680001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 069 | HSR1SS 0690001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 070 | HSR1SS 0700001 | 1 | TBD | TBD | TBD | TBD |
| Field Duplicate | | -- | TBD | TBD | TBD | TBD |
| HSR1SS 071 | HSR1SS 0710001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 072 | HSR1SS 0720001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 073 | HSR1SS 0730001 | 1 | TBD | TBD | TBD | TBD |

| Sample Location[1] | Sample ID[2] | Lead[3] | | PAHs[4] | Nitroglycerin[3,5] | pH[6] |
|---|---|---|---|---|---|---|
| | | Field XRF (Lead Only) | SW-846 3050B/6010B (Lead) | SW-846 8270C SIM | SW-846 8330B | SW-846 9045C |
| HSR1SS 074 | HSR1SS 0740001 | 1 | TBD | TBD | TBD | TBD |
| HSR1SS 075 | HSR1SS 0750001 | 1 | TBD | TBD | TBD | TBD |
| **Total Soil Samples** | | | **TBD** | **TBD** | **TBD** | **TBD** |

HSR1=Horse Stables Skeet Range #1
PAHs=Polynuclear aromatic hydrocarbons
SS=Surface Soil Sample

1 Surface soil samples will be collected along transect lines focused on the expected fall-out area. All samples will be collected in accordance SOP-05. Proposed sample locations are shown on Figure 17.5.
2 The sample ID number will consist of, in order, site acronym, sample matrix, sample location number, and sequential depth interval.
3 Dependent upon field XRF screening results, a minimum of 20 samples will be selected for fixed-base laboratory lead analysis.
4 A minimum of 20 samples will be selected for fixed-base laboratory PAH analysis.
5 Two composite surface soil samples along the firing lines will be submitted for fixed-base laboratory NG and lead analyses.
6 At each site the field geologist will select up to three of the samples submitted to the fixed-base laboratory for pH, TOC, and CEC analyses

**SAP Worksheet #18.6-- Sampling Locations and Methods/SOP Requirements Table**

(UFP-QAPP Manual Section 3.1.1)

**Horse Stables Skeet Range #2**

| Sample Location[1] | Sample ID[2] | Lead[3] | | PAHs[4] | Nitroglycerin[3,5] | pH[6] |
|---|---|---|---|---|---|---|
| | | Field XRF (Lead Only) | SW-846 3050B/6010B (Lead) | SW-846 8270C SIM | SW-846 8330B | SW-846 9045C |
| HSR2SS 001 | HSR2SS 0010001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 002 | HSR2SS 0020001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 003 | HSR2SS 0030001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 004 | HSR2SS 0040001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 005 | HSR2SS 0050001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 006 | HSR2SS 0060001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 007 | HSR2SS 0070001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 008 | HSR2SS 0080001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 009 | HSR2SS 0090001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 010 | HSR2SS 0100001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 011 | HSR2SS 0110001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 012 | HSR2SS 0120001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 013 | HSR2SS 0130001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 014 | HSR2SS 0140001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 015 | HSR2SS 0150001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 016 | HSR2SS 0160001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 017 | HSR2SS 0170001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 018 | HSR2SS 0180001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 019 | HSR2SS 0190001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 020 | HSR2SS 0200001 | 1 | TBD | TBD | TBD | TBD |
| Field Duplicate | | -- | TBD | TBD | TBD | TBD |
| HSR2SS 021 | HSR2SS 0210001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 022 | HSR2SS 0220001 | 1 | TBD | TBD | TBD | TBD |

| Sample Location[1] | Sample ID[2] | Lead[3] | | PAHs[4] | Nitroglycerin[3,5] | pH[6] |
|---|---|---|---|---|---|---|
| | | Field XRF (Lead Only) | SW-846 3050B/6010B (Lead) | SW-846 8270C SIM | SW-846 8330B | SW-846 9045C |
| HSR2SS 023 | HSR2SS 0230001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 024 | HSR2SS 0240001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 025 | HSR2SS 0250001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 026 | HSR2SS 0260001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 027 | HSR2SS 0270001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 028 | HSR2SS 0280001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 029 | HSR2SS 0290001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 030 | HSR2SS 0300001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 031 | HSR2SS 0310001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 032 | HSR2SS 0320001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 033 | HSR2SS 0330001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 034 | HSR2SS 0340001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 035 | HSR2SS 0350001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 036 | HSR2SS 0360001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 037 | HSR2SS 0370001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 038 | HSR2SS 0380001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 039 | HSR2SS 0390001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 040 | HSR2SS 0400001 | 1 | TBD | TBD | TBD | TBD |
| Field Duplicate | | -- | TBD[3] | TBD | TBD | TBD |
| HSR2SS 041 | HSR2SS 0410001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 042 | HSR2SS 0420001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 043 | HSR2SS 0430001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 044 | HSR2SS 0440001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 045 | HSR2SS 0450001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 046 | HSR2SS 0460001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 047 | HSR2SS 0470001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 048 | HSR2SS 0480001 | 1 | TBD | TBD | TBD | TBD |

| Sample Location[1] | Sample ID[2] | Lead[3] | | PAHs[4] | Nitroglycerin[3,5] | pH[6] |
|---|---|---|---|---|---|---|
| | | Field XRF (Lead Only) | SW-846 3050B/6010B (Lead) | SW-846 8270C SIM | SW-846 8330B | SW-846 9045C |
| HSR2SS 049 | HSR2SS 0490001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 050 | HSR2SS 0490001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 051 | HSR2SS 0510001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 052 | HSR2SS 0520001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 053 | HSR2SS 0530001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 054 | HSR2SS 0540001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 055 | HSR2SS 0550001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 056 | HSR2SS 0560001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 057 | HSR2SS 0570001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 058 | HSR2SS 0580001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 059 | HSR2SS 0590001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 060 | HSR2SS 0600001 | 1 | TBD | TBD | TBD | TBD |
| Field Duplicate | | -- | TBD | TBD | TBD | TBD |
| HSR2SS 061 | HSR2SS 0610001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 062 | HSR2SS 0620001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 063 | HSR2SS 0630001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 064 | HSR2SS 0640001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 065 | HSR2SS 0650001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 066 | HSR2SS 0660001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 067 | HSR2SS 0670001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 068 | HSR2SS 0680001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 069 | HSR2SS 0690001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 070 | HSR2SS 0700001 | 1 | TBD | TBD | TBD | TBD |
| Field Duplicate | | -- | TBD | TBD | TBD | TBD |
| HSR2SS 071 | HSR2SS 0710001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 072 | HSR2SS 0720001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 073 | HSR2SS 0730001 | 1 | TBD | TBD | TBD | TBD |

| Sample Location[1] | Sample ID[2] | Lead[3] | | PAHs[4] | Nitroglycerin[3,5] | pH[6] |
|---|---|---|---|---|---|---|
| | | Field XRF (Lead Only) | SW-846 3050B/6010B (Lead) | SW-846 8270C SIM | SW-846 8330B | SW-846 9045C |
| HSR2SS 074 | HSR2SS 0730001 | 1 | TBD | TBD | TBD | TBD |
| HSR2SS 075 | HSR2SS 0740001 | 1 | TBD | TBD | TBD | TBD |
| **Total Soil Samples** | | **75** | **TBD** | **TBD** | **TBD** | **TBD** |

HSR2=Horse Stables Skeet Range #2
PAHs=Polynuclear aromatic hydrocarbons
SS=Surface Soil Sample

1 Surface soil samples will be collected along transect lines focused on the expected fall-out area. All samples will be collected in accordance with SOP-05. Proposed sample locations are shown on Figure 17.6.

2 The sample ID number will consist of, in order, site acronym, sample matrix, sample location number, and sequential depth interval. Sample depth to a maximum of 1-foot bgs for surface samples.

3 Dependent upon field XRF screening results, a minimum of 20 samples will be selected for fixed-base laboratory lead analysis.

4 A minimum of 20 samples will be selected for fixed-base laboratory PAH analysis.

5 Two composite surface soil samples along the firing lines will be submitted for fixed-base laboratory NG and lead analyses.

6 At each site the field geologist will select up to three of the samples submitted to the fixed-base-laboratory for pH, TOC, and CEC analyses.

**SAP Worksheet #19 – Analytical SOP Requirements Table - Empirical Laboratories**

(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) |
|---|---|---|---|---|---|---|
| Aqueous field quality control blank[1] | PAHs | SW-846 3510C/8270C SIM SOP-300/231 | Two 1-Liter glass amber bottle | 1,000 milliliters | Cool to 4° C ± 2° C | 7 days until extraction/ 40 days to analysis |
| | Lead | SW-846 3005A/6010B SOP-100/105 | 500-Milliliter plastic bottle | 50 milliliters | Nitric acid to a pH<2; Cool to 4° C ± 2° C | 180 days to analysis |
| Soil | PAHs | SW-846 3541/8270C SIM SOP-329/231 | 4-Ounce glass bottle | 15 grams | Cool to 4° C ± 2° C | 14 days until extraction/ 40 days to analysis |
| | Lead | SW-846 3050B/6010B SOP-100/105 | 4-Ounce glass bottle | 1 to 2 grams | Cool to 4° C ± 2° C | 180 days to analysis |
| | pH | SW-846 9045C/SOP-187 | 4-Ounce glass bottle | 20 grams | Cool to 4° C ± 2° C | Analyze immediately |
| | CEC | SW-846 9081 | 4-Ounce glass bottle | 4 to 6 grams | Cool to 4° C ± 2° C | Twenty-eight days to analyze |
| | TOC | Lloyd Kahn/SOP-221 | 4-Ounce glass bottle | 5 grams | Cool to 4° C ± 2° C | Analyze immediately |

1 Includes equipment rinsate blanks.

## SAP Worksheet #19 – Analytical SOP Requirements Table (GPL Laboratories)

(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) |
|---|---|---|---|---|---|---|
| Aqueous field quality control blank[1] | Nitroglycerin | SW-846 8330B/GPL Laboratories SOP S.11 | 1-liter amber glass bottle | 770 mL | Cool to 4°C ± 2°C | 7 days to extraction/ 40 days to analysis |
| Soil | Nitroglycerin | SW-846 8330B/GPL Laboratories SOP S.11 | 8-ounce glass bottle | 30 g | Cool to 4°C ± 2°C | 14 days to extraction/ 40 days to analysis |

1 Includes equipment rinsate blanks.

**SAP Worksheet #20 – Field Quality Control Sample Summary Table**

(UFP-QAPP Manual Section 3.1.1)

**Pistol Range, Rifle Range, Aircraft Firing Ranges, Trap/Skeet Ranges #1 and #2, Horse Stables Skeet Range #1, and Horse Stables Skeet Range #2**

Field-Portable X-Ray Fluorescence (XRF) Analyses

| Matrix | Analytical Group | Concentration Level | No. of Samples | No. of Field Duplicates(1) | No. of MS/MSDs (2) | No. of Field Blanks | No. of Equip. Blanks | No. of PT Samples(3) | Total No. of Samples to Lab |
|---|---|---|---|---|---|---|---|---|---|
| Solid | Lead | Low to Moderate | 450 | 23 | NA | NA | NA | NA | TBD |

Fixed-Base Laboratory Analyses

| Matrix | Analytical Group | Concentration Level | No. of Samples | No. of Field Duplicates(1) | No. of MS/MSDs (2) | No. of Field Blanks | No. of Equip. Blanks | No. of PT Samples(3) | Total No. of Samples to Lab |
|---|---|---|---|---|---|---|---|---|---|
| Solid | Lead | Low to Moderate | 138 | 7 | 7 | NA | 7 | NA | TBD |
| Solid | Nitroglycerin | Low to Moderate | 18 | 2 | 2 | NA | 7 | NA | TBD |
| Solid | PAHs | Low to Moderate | 120 | 6 | 6 | NA | 7 | NA | TBD |
| Solid | pH | TBD | 18 | 2 | 2 | NA | 7 | NA | TBD |
| Solid | CEC | TBD | 18 | 2 | 2 | NA | 7 | NA | TBD |
| Solid | TOC | TBD | 18 | 2 | 2 | NA | 7 | NA | TBD |

TBD = To be determined based on results of field XRF screening results and total number of samples collected (minimum of 20 samples will be submitted for laboratory analysis).
RB=Rinsate Blank sample
IDW=Investigational-derived waste sample

1 Duplicates will be collected at a frequency of 1 per 20 samples.
2 MS/MSDs will be collected at a frequency of 1 per 20 samples. Although MS/MSDs are not typically considered field QC samples, they are included here because field personnel typically designate which samples will be utilized as MS/MSDs.
3 Batch or project-specific proficiency testing (PT) samples are highly recommended but will not be collected during these SIs.

As described in SOP-02, the QC sample code will consist of a two- to five-segment alphanumeric code that identifies the sample QC type, date the sample was collected, and number of this type of QC sample collected on that date. For example, the first rinsate blank collected on March 5, 2008, would be identified as RB03050801.

**SAP Worksheet #21 – Project Sampling SOP References Table**

(UFP-QAPP Manual Section 3.1.2)

| Reference Number | Title, Revision Date and/or Number | Originating Organization of Sampling SOP | Equipment Type | Modified for Project Work? (Y/N) | Comments |
|---|---|---|---|---|---|
| SOP-01 | Sample Labeling | Tetra Tech | NA | N | SOP Contained in Appendix A |
| SOP-02 | Sample Identification Nomenclature | Tetra Tech | NA | N | SOP Contained in Appendix A |
| SOP-03 | Sample Custody and Documentation of Field Activities | Tetra Tech | NA | N | SOP Contained in Appendix A |
| SOP-04 | Decontamination of Field Sampling Equipment | Tetra Tech | Brushes, buckets, and decontamination solution | N | SOP Contained in Appendix A |
| SOP-05 | Borehole and Soil Sample Logging | Tetra Tech | Hand auger, trowels, and bowls | N | SOP Contained in Appendix A |
| SOP-06 | Borehole Advancement and Soil Coring and Sampling Using Direct-Push Technology | Tetra Tech | Drilling rig and accessories | N | SOP Contained in Appendix A |
| SOP-09 | Management of Investigation-Derived Waste | Tetra Tech | NA | N | SOP Contained in Appendix A |
| SOP-11 | Sample Preservation, Packaging, and Shipping | Tetra Tech | NA | N | SOP Contained in Appendix A |
| SOP-13 | Use of Field Portable X-Ray Fluorescence Analysis of Soil and Sediment | Tetra Tech | Portable XRF analyzer and accessories | N | SOP Contained in Appendix A |
| MRP SOP-05 | GPS Data Collection and Transfer | Tetra Tech | Trimble GPS | N | SOP Contained in Appendix A |

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

(UFP-QAPP Manual Section 3.1.2.4)

| Field Equipment | Activity[1] | Frequency | Acceptance Criteria | Corrective Action | Responsible Person | SOP Reference[2] | Comments |
|---|---|---|---|---|---|---|---|
| XRF | Verification/ lead standardization check | Prior to daily use and after every 20 samples throughout the day | 80<%Recovery< 120 | Restandardize. If still unacceptable, contact manufacturer for possible replacement | FOL | SOP-13 | |
| GPS | Calibration/ comparison to known survey coordinates | Prior to daily use | Manufacturer's guidance | Operator correction or replacement | FOL | MRP SOP-05 | |

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

2 Referenced SOPs can be found in Appendix A.

**SAP Worksheet #23 – Analytical SOP References Table - Empirical Laboratories**

(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) |
|---|---|---|---|---|---|---|
| SOP-100 | Metals Digestion/Preparation Methods 3005A,3010A, 3020A, 3030, 3040A, 3050B, USEPA CLP ILMO 4.1 Aqueous and Soil/Sediment, USEPA Method 200.7 (Standard Methods) 3030C. Rev. 19 | Definitive | Soil and Water/Lead | NA/Metals | Empirical | N |
| SOP-105 | Metals Analysis by ICP Technique Methods 200.7, SW846 6010B, SM 19th Edition 2340B, USEPA ILMO 4.1, Rev. 14 | Definitive | Soil and Water/Lead | ICP | Empirical | N |
| SOP-187 | Electrometric Determination of pH, Methods 150.1, Standard Methods 4500H+B and 9040B for Waters, Liquids and Liquid Wastes, 9045C for Soils and Solid Wastes, Rev. 6 | Definitive | Soil/pH | pH Meter | Empirical | N |
| SOP-221 | Total Organic Carbon SM5310C, USEPA Method 415.1 and SW846 Method 9060 and Lloyd Kahn Method, Rev. 7 | Definitive | Soil/TOC | TOC Analyzer | Empirical | N |
| SOP-231 | GC/MS Low Level PAHs by SW-846 Method 8270C SIM, Rev. 3 | Definitive | Soil and Water/PAHs | Agilent/HP GC/MS | Empirical | N |
| SOP-300 | GC/MS- Semivolatile BNA-Aqueous Matrix Extraction Using SW-846 Method 3510C for 8270C/625 Analysis, Rev. 17 | Definitive | Water/PAH Extraction | NA/Extraction | Empirical | N |
| SOP-329 | Soxhlet Extraction - BNA and Pest/PCB Using SW-846 Method 3541, Rev. 16 | Definitive | Soil/PAH Extraction | NA/Extraction | Empirical | N |

| Lab SOP Number | Title, Revision Date, and/or Number | Definitive or Screening Data | Matrix and Analytical Group | Instrument | Organization Performing Analysis | Modified for Project Work? (Y/N) |
|---|---|---|---|---|---|---|
| SOP-404 | Laboratory Sample Receiving Log-in and Storage Standard Operating Procedures, Rev. 12 | Definitive | Log-In | NA/Log-In | Empirical | N |
| SOP-405 | Analytical Laboratory Waste Disposal, Rev. 4 | Definitive | Log-in | NA/Log-In | Empirical | N |
| SOP-410 | Standard Operating Procedures for Laboratory Sample Storage, Secure Areas, and Sample Custody, Rev. 6 | Definitive | Log-in | NA/Log-In | Empirical | N |

**SAP Worksheet #23 – Analytical SOP References Table - GPL Laboratories**

(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) |
|---|---|---|---|---|---|---|
| F.2 | Sample Receipt, Inspection, Preservation and Storage Condition Requirements | Definitive | All | NA | GPL Laboratories | N |
| G.22 | General Laboratory Multi-Incremental Sampling (MIS) Sub-sampling Procedure | Definitive | Soil/Subsampling | NA | GPL Laboratories | N |
| S.11 | High Performance Liquid Chromatography (HPLC) Analysis of Nitroaromatics and Nitroamine Explosive Residues in Water, Soil and Sediment Samples | Definitive | Soil/Nitroglycerin | HPLC | GPL Laboratories | N |
| D.1 | Laboratory Waste Handling and Storage Procedure | Definitive | All | NA | GPL Laboratories | N |
| G.10 | Instrument Maintenance | Definitive | All | NA | GPL Laboratories | N |
| G.12 | Standard Operating Procedures for Report Generation | Definitive | All | NA | GPL Laboratories | N |

## SAP Worksheet #24 – Analytical Instrument Calibration Table - Empirical Laboratories

(UFP-QAPP Manual Section 3.2.2)

| Instrument | Calibration Procedure | Frequency of Calibration | Acceptance Criteria | Corrective Action (CA) | Person Responsible for CA | SOP Reference |
|---|---|---|---|---|---|---|
| pH Meter | Calibration | Calibrate before use with two buffers in the area to be measured. Check with a third buffer. | 0.05 pH units. | Recalibrate and/or perform necessary equipment maintenance. | Analyst/Supervisor | SOP-187 |
| Gas Chromatograph Mass Spectrometer PAHs | Initial Calibration - A minimum five-point calibration is required. | Calibrate the instrument when it is received and after a major change or if the daily calibration fails. A minimum five-point calibration is required. | The Percent Relative Standard Deviation (%RSD) for each Calibration Check Compound (CCC) must be ≤ 30%. The minimum mean response factor (RF) for each System Performance Check Compound (SPCC) must meet that stated in 8270C. If the RSD for an analyte is >15% use a linear curve (≥ 0.995 correlation) or quadratic curve (≥0.99 correlation, minimum six points) for quantitation. | Recalibrate and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data. | Analyst/Supervisor | SOP-231 |
| | Initial Calibration Verification (ICV) | Once after each initial calibration. | ±25% recovery of individual compounds. | Identify source of problem, correct, repeat calibration, rerun samples. | Analyst/Supervisor | |
| | Continuing Calibration (CCV) | Analyze a standard at the beginning of each 12-hour shift after a decafluorotriphenyl-phosphine (DFTPP) tune. | The minimum RF for SPCCs must meet those stated in method. The CCCs must be < 20 percent difference. | Recalibrate and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data. | Analyst/Supervisor | |

| Instrument | Calibration Procedure | Frequency of Calibration | Acceptance Criteria | Corrective Action (CA) | Person Responsible for CA | SOP Reference |
|---|---|---|---|---|---|---|
| Inductively Coupled Plasma (ICP) Spectrometer | Initial Calibration | At the beginning of each day or if the QC is out of criteria. | The instrument is calibrated by a one-point calibration per manufacturer's guidelines. Analytes run at their calibration levels must fall within 90-110% of the true values. | Recalibrate and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data. | Analyst/Supervisor | Empirical SOP-105 |
| | Initial Calibration Blank (ICB) | Before beginning a sample sequence. | No analytes detected > 2x the method detection limit (MDL). | Correct the problem, then re-prepare and reanalyze. | Analyst/Supervisor | |
| | Continuing Calibration Standard (CCV) | Analyze a standard at the beginning and end of the sequence and after every 10 samples. | The acceptance criterion for the continuing calibration standard is 90-110% of true values. | Recalibrate and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data. | Analyst/Supervisor | |
| | Continuing Calibration Blank (CCB) | After every 10 samples and at the end of the sequence. | No analytes detected > 2x MDL. | Correct problem, then reprepare and reanalyze calibration blank and previous 10 samples. | Analyst/Supervisor | |

## SAP Worksheet #24 – Analytical Instrument Calibration Table - GPL Laboratories

(UFP-QAPP Manual Section 3.2.2)

| Instrument | Calibration Procedure | Frequency of Calibration | Acceptance Criteria | CA | Person Responsible for CA | SOP Reference |
|---|---|---|---|---|---|---|
| High-Pressure Liquid Chromatography (HPLC) - Nitroglycerin | Initial Calibration Curve, six points plus a calibration blank. | Annually or more often as needed due to changes in response or retention times or following major instrument maintenance. | Average RF </= 20 %; if a linear fit is used, coefficient of determination ($r^2$) >/=0.995. | Determine and correct reason for failure. Repeat calibration. | HPLC Analyst | S.11 |
| | Initial calibration verification – from a source different than that used for calibration curve. | Following initial calibration prior to analysis of samples. | ±15% Difference. | Investigate reasons for failure, reanalyze once. If still unacceptable, repeat calibration. | HPLC Analyst | S.11 |
| | CCV | Daily prior to analysis of samples, every 10 sample injections or 12 hours (whichever is more frequent), and at the end of run. | ±15% Difference. | Investigate reasons for failure, reanalyze once. If still unacceptable, repeat calibration. | HPLC Analyst | S.11 |

**SAP Worksheet #25 – Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table – Empirical Laboratories**

(UFP-QAPP Manual Section 3.2.3)

| Instrument/ Equipment | Maintenance Activity | Testing Activity | Inspection Activity | Frequency | Acceptance Criteria | Corrective Action | Responsible Person | SOP Reference |
|---|---|---|---|---|---|---|---|---|
| pH Meter | Clean, drain, and refill reference electrode as needed. | pH | Reference electrode for white crystals. Inspect electrode for damage. | Before use | ± 0.09 units | Recalibrate and/or perform necessary equipment maintenance. Check buffers. Reanalyze affected data. | Analyst/ Supervisor | SOP-187 |
| Gas Chromatograph/ Mass Spectrometer | Clean source and replace filaments. Replace seal, liner, and septum. Change column. | PAHs | Check gas supply. Check seal, liner, and septum. | Source cleaning is performed when instrument response deteriorates. Other instrument maintenance done as needed to keep instrument at peak performance. | The minimum response factor (RF) for System Performance Check Compounds (SPCCs) must meet those stated in method SW-846 8270C. The calibration check compounds (CCCs) must be <20 percent difference. | Recalibrate and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data. | Analyst/ Supervisor | SOP-231 |

| Instrument/ Equipment | Maintenance Activity | Testing Activity | Inspection Activity | Frequency | Acceptance Criteria | Corrective Action | Responsible Person | SOP Reference |
|---|---|---|---|---|---|---|---|---|
| Inductively Coupled Plasma Spectrometer | Clean torch assembly and spray chamber when they become discolored or when degradation in data quality is observed. Clean nebulizer and check argon supply. Replace peristaltic pump tubing as needed. | Lead | Inspect torch, nebulizer chamber, pump, and tubing. | Prior to initial calibration or as necessary. | The acceptance criterion for the continuing calibration standard is 90 to 110% of true value. | Recalibrate and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data. | Analyst/ Supervisor | SOP-105 |

**SAP Worksheet #25 – Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table – GPL Laboratories**

(UFP-QAPP Manual Section 3.2.3)

| Instrument/ Equipment | Maintenance Activity | Testing Activity | Inspection Activity | Frequency | Acceptance Criteria | Corrective Action | Responsible Person | SOP Reference |
|---|---|---|---|---|---|---|---|---|
| High Pressure Liquid Chromatography (HPLC) | Check column backpressure. | Run instrument without sample injection. | Observe baseline. | Daily. | Constant backpressure within instrument limits, and a steady baseline. | Rinse column, change column pre-filter, prepare fresh mobile phase. | HPLC Analyst | S.11, G.10 |
| | Clean column. | Run continuing calibration verification. | Observe retention times and response/ recovery. | As needed. | Acceptable peak resolution, no shifts in retention time >10%. Responses within 15% of primary standard. | Change column pre-filter, prepare fresh mobile phase, replace column. Recalibrate if above steps do not correct. | HPLC Analyst | S.11, G.10 |
| | Replace pump seals. | Run several continuing calibration verifications. | Observe retention times and baseline. | As needed. | Steady baseline, acceptable peak resolution, repeatable retention times between injections. Relative Percent Difference (RPD) <5%. | Check other pump components for wear, check lines for air bubbles, call for service if required. | HPLC Analyst | S.11, G.10 |

**SAP Worksheet #26 – Sample Handling System**

(UFP-QAPP Manual Appendix A)

# SAMPLE HANDLING SYSTEM

| |
|---|
| **SAMPLE COLLECTION, PACKAGING, AND SHIPMENT** |
| Sample Collection (Personnel/Organization): TBD/Tetra Tech |
| Sample Packaging (Personnel/Organization): TBD/Tetra Tech |
| Coordination of Shipment (Personnel/Organization): TBD/Tetra Tech |
| Type of Shipment/Carrier: TBD |
| **SAMPLE RECEIPT AND ANALYSIS** |
| Sample Receipt (Personnel/Organization): Sample Custodians/Empirical Laboratories and GPL Laboratories |
| Sample Custody and Storage (Personnel/Organization): Sample Custodians/Empirical Laboratories and GPL Laboratories |
| Sample Preparation (Personnel/Organization): Extraction Lab, Metals Preparation Lab/Empirical Laboratories; Extraction Lab /GPL Laboratories |
| Sample Determinative Analysis (Personnel/Organization): Gas Chromatography Lab, Gas Chromatography/Mass Spectrometry Lab, Metals Lab/Empirical Laboratories ; HPLC/GPL Laboratories |
| **SAMPLE ARCHIVING** |
| Field Sample Storage: 60 days from receipt of collection. |
| Sample Extract/Digestate Storage (No. of days from extraction/digestion): 3 months from sample digestion/extraction |
| Biological Sample Storage (No. of days from sample collection): NA |
| **SAMPLE DISPOSAL** |
| Personnel/Organization: Sample Custodians/Empirical Laboratories and GPL Laboratories |
| Number of Days from Analysis: 30 days from submittal of final report or 60 days from receipt, whichever is longer |

**SAP Worksheet #27 – Sample Custody Requirements Table**

(UFP-QAPP Manual Section 3.3.3)

**SAMPLE CUSTODY REQUIREMENTS**

**Field Chain of Custody**

To ensure the integrity of a sample from collection through analysis, an accurate written record that traces the possession and handling of the sample is necessary. This documentation is referred to as the chain-of-custody form. Chain-of-custody begins at the time of sample collection.
A sample is under custody if:

- It is in the FOL's or the FOL's designee's actual possession, or
- It is in the FOL's or the FOL's designee's view, after being in the FOL's or the FOL's designee's physical possession, or
- It was in the FOL's or the FOL's designee's possession and then the FOL's or the FOL's designee's locked or sealed it to prevent tampering, or
- It is in a secure area.

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

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

The FOL is responsible for the care and custody of the samples collected until they are delivered to the laboratories or are entrusted to a carrier. When transferring samples, the individuals relinquishing and receiving them will sign, date, and note the time on the chain-of-custody form. This form documents the sample custody transfer from the sampler to the laboratory, often through another person or agency (common carrier). Field chain-of-custody requirements are provided in SOP-03. Upon arrival at the

laboratories, internal sample custody procedures will be followed as defined in the laboratory SOPs included in Appendix B.

**Laboratory Chain of Custody - Empirical**

Laboratory sample custody procedures (receipt of samples, archiving, and disposal) will be according to Empirical SOPs 404, 405, and 410. Coolers will be received and checked for proper temperature. A sample cooler receipt form will be filled out to note conditions and any discrepancies between the chain-of-custody and the sample container labels. Samples will be logged into the laboratory information management system (LIMS) and given a unique log number that can be tracked through processing. The client will be notified of any problems.

**Laboratory Chain of Custody - GPL Laboratories**

Documentation tracks the historical possession of samples. A completed chain-of-custody form must accompany all samples entering the laboratory. This form provides essential information to the laboratory regarding sample collection and analyses required and includes the customer name, project name, sampler's name or initials, sample location, sampling date and time, number of containers, type of preservative used, sample type, special remarks concerning the sample or project, and analytical parameters requested.

GPL Laboratories SOP-F.2 provides instruction on reviewing and inspecting the chain-of-custody for discrepancies. This is accomplished by inspection and comparison of the samples received to the chain-of-custody form to identify any discrepancies. Sample receiving will immediately notify the customer service department of samples received without a chain-of-custody via Federal Express. These samples will be placed on hold until the chain-of-custody form is received from the customer. Complete chain-of-custody documentation, including memos, transmittal forms, etc., is filed in the reporting department and properly retained by the laboratory along with the completed data report in hardcopy and CD form.

Sample preparation logs and sample analysis logs document the progression of extracts from preparation to analysis. LIMS also tracks this progression and documents dates and times of sample preparation and analyzed on the final laboratory analysis report.

Upon arrival in the laboratory, samples are received by a sample custodian who ensures that a proper chain-of-custody form accompanies all samples. The chain-of-custody form will be signed by the person delivering the samples to relinquish the samples to the sample custodian. The sample custodian will then inspect and compare the samples to be received to the chain-of-custody form to identify any

discrepancies before signing the form and receiving the samples into the custody of the laboratory. The date and time relinquished/received will also be recorded on the chain-of-custody form. A Cooler Receipt Checklist is also filled out on the chain-of-custody form for each container received by a third party transporter (i.e., FedEx). Information relating to shipping, sample temperature, custody seals, chain-of-custody/label agreement, container condition, sample amount, and container size/type/preservation is recorded.

If discrepancies cannot be resolved at the time of sample receipt by sample receiving personnel, project management will address them during the chain-of-custody review following LIMS entry.

## SAP Worksheet #28 – Laboratory QC Samples Table - Empirical Laboratories

(UFP-QAPP Manual Section 3.4)

| | |
|---|---|
| Matrix | Aqueous field quality control and soil |
| Analytical Group | PAHs |
| Analytical Method / SOP Reference | SW-846 8270C SIM/Empirical SOP-231 |

| QC Sample | Frequency/ Number | Method/SOP QC Acceptance Limits | Corrective Action | Person(s) Responsible for Corrective Action | Data Quality Indicator | Measurement Performance Criteria |
|---|---|---|---|---|---|---|
| Method Blanks | One per batch of 20 or less. | No target compounds should be ≥1/2 the QL except common lab contaminants, which should be < QL | Reclean, retest, re-extract, reanalyze, and/or qualify the data. | Analyst, Laboratory Supervisor, and Data Validator | Bias/Contamination | No target compounds > QLs. |
| Surrogates | Two per sample. | Percent recoveries:<br>Aqueous:<br>2-Fluorobiphenyl 34-167<br>Terphenyl-d14 34-167<br><br>Soil:<br>2-Fluorobiphenyl 14-129<br>Terphenyl-d14 14-129 | Reprepare and reanalyze for confirmation of matrix interference when appropriate. | Analyst, Laboratory Supervisor and Data Validator | Accuracy/Bias | Percent recoveries:<br>Aqueous:<br>2-Fluorobiphenyl 34-167<br>Terphenyl-d14 34-167<br><br>Soil:<br>2-Fluorobiphenyl 14-129<br>Terphenyl-d14 14-129 |

| | |
|---|---|
| Matrix | Aqueous field quality control and soil |
| Analytical Group | PAHs |
| Analytical Method / SOP Reference | SW-846 8270C SIM/Empirical SOP-231 |

| QC Sample | Frequency/ Number | Method/SOP QC Acceptance Limits | Corrective Action | Person(s) Responsible for Corrective Action | Data Quality Indicator | Measurement Performance Criteria |
|---|---|---|---|---|---|---|
| Laboratory Control Sample (LCSs) | One per batch of 20 or less. | Compound specific as per SOP-231. | (1) Evaluate and reanalyze if possible. (2) If an MS/MSD was performed in the same 12-hour period and acceptable, narrate. (3) If LCS recoveries are high but sample results are <QL, then narrate; otherwise, reprepare and reanalyze the batch. | Analyst, Laboratory Supervisor, and Data Validator | Precision/Accuracy/Bias | Compound specific as per SOP-231. |

| | |
|---|---|
| Matrix | Aqueous field quality control and soil |
| Analytical Group | PAHs |
| Analytical Method / SOP Reference | SW-846 8270C SIM/Empirical SOP-231 |

| QC Sample | Frequency/ Number | Method/SOP QC Acceptance Limits | Corrective Action | Person(s) Responsible for Corrective Action | Data Quality Indicator | Measurement Performance Criteria |
|---|---|---|---|---|---|---|
| Internal Standards (ISs) | Two per sample<br>Perylene-d12<br>Phenanthrene-d10 | Retention times for ISs must be ± 30 seconds and the responses within -50% to +100% of last calibration verification (12 hours). | Inspect mass spectrometer or gas chromatograph for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning. | Analyst, Laboratory Supervisor, and Data Validator | Precision/Accuracy/Bias | Retention times for ISs must be ± 30 seconds and the responses within -50% to +100% of last calibration verification (12 hours). |
| Matrix Spike/Matrix Spike Duplicate (MS/MSD) Samples | One per Sample Delivery Group (SDG) every 20 samples. | Compound specific as per SOP-231. | (1) Corrective action will not be taken for samples when recoveries are outside limits and surrogate and LCS criteria are met.<br>(2) If both the LCS and MS/MSD are unacceptable, reprepare the samples and QC. | Analyst, Laboratory Supervisor, and Data Validator | Precision/Accuracy/Bias | Compound specific as per SOP-231. |

| | |
|---|---|
| Matrix | Aqueous field quality control |
| Analytical Group | Lead |
| Analytical Method / SOP Reference | SW-846 3005A, 6010B/SOP-100/105 |

| QC Sample | Frequency/ Number | Method/SOP QC Acceptance Limits | Corrective Action | Person(s) Responsible for Corrective Action | Data Quality Indicator | Measurement Performance Criteria |
|---|---|---|---|---|---|---|
| Method Blanks | One per digestion batch of 20 or fewer samples. | Contaminants in method blank must be less than ½ QLs. | Investigate source of contamination. Redigest and reanalyze all associated samples if sample concentration ≥ QL and <10x blank concentration. | Analyst, Laboratory Supervisor, and Data Validator | Bias/Contamination | Contaminants in method blank must be less than QLs. |
| LCSs | One per digestion batch of 20 or fewer samples. | Recovery must be within ± 20% of the true values, unless vendor-supplied or statistical limits have been established. | (1) Investigate source of problem. (2) Redigest and reanalyze all associated samples. | Analyst, Laboratory Supervisor, and Data Validator | Accuracy/Bias/Contamination | Recovery must be within ±20% of the true values, unless vendor-supplied or statistical limits have been established. |
| Duplicate Samples | One per digestion batch of 20 or fewer samples. | Relative percent difference should be within ≤20% for duplicate spikes. | Flag results. | Analyst, Laboratory Supervisor, and Data Validator | Precision | The relative percent difference should be within ≤20% for duplicate spikes. |
| MSs | One per digestion batch of 20 or fewer samples. | Recovery should be ±25% of the true value, if sample < 4x spike added. | Flag results. | Analyst, Laboratory Supervisor, and Data Validator | Accuracy/Bias | Recovery should be ±25% of the true value, if sample < 4x spike added. |
| ICP Serial Dilutions | One per digestion batch. | If original sample result is at least 50x instrument detection limit, five-fold dilution must agree within ± 10% of original result. | Flag result or dilute and reanalyze sample to eliminate interference. | Analyst, Laboratory Supervisor, and Data Validator | Accuracy/Bias | If original sample result is at least 50x instrument detection limit, five-fold dilution must agree within ± 10% of original result. |

| | |
|---|---|
| Matrix | Soil |
| Analytical Group | Lead |
| Analytical Method / SOP Reference | SW-846 3050B, 6010B/SOP-100/105 |

| QC Sample | Frequency / Number | Method/SOP QC Acceptance Limits | Corrective Action | Person(s) Responsible for Corrective Action | Data Quality Indicator | Measurement Performance Criteria |
|---|---|---|---|---|---|---|
| Method Blanks | One per digestion batch of 20 or fewer samples. | Contaminants in method blank must be less than ½ QLs. | Investigate source of contamination. Redigest and reanalyze all associated samples if sample concentration ≥ reporting limit and <10x blank concentration. | Analyst, Laboratory Supervisor, and Data Validator | Bias/Contamination | Contaminants in method blank must be less than QLs. |
| LCSs | One per digestion batch of 20 or fewer samples. | Recovery must be within ±20% of true value, unless vendor-supplied or statistical limits have been established. | Investigate source of problem. Redigest and reanalyze all associated samples. | Analyst, Laboratory Supervisor, and Data Validator | Accuracy/Bias/Contamination | Recovery must be within ±20% of the true values, unless vendor-supplied or statistical limits have been established. |
| Duplicate Samples | One per digestion batch of 20 or fewer samples. | Relative percent difference should be within ≤20% for duplicate spikes. | Flag results. | Analyst, Laboratory Supervisor, and Data Validator | Precision | Relative percent difference should be within ≤20% for duplicate spikes. |
| Matrix Spikes | One per digestion batch of 20 or fewer samples. | Recovery should be ±25% of the true values, if sample < 4x spike added. | Flag results. | Analyst, Laboratory Supervisor, and Data Validator | Accuracy/Bias | Recovery should be ±25% of true value, if sample <4x spike added. |

| Matrix | Soil |
|---|---|
| Analytical Group | Lead |
| Analytical Method / SOP Reference | SW-846 3050B, 6010B/SOP-100/105 |

| QC Sample | Frequency / Number | Method/SOP QC Acceptance Limits | Corrective Action | Person(s) Responsible for Corrective Action | Data Quality Indicator | Measurement Performance Criteria |
|---|---|---|---|---|---|---|
| ICP Serial Dilutions | One per digestion batch. | If original sample result is at least 50x the instrument detection limit, five-fold dilution must agree within ±10% of original result. | Flag result or dilute and reanalyze sample to eliminate interference. | Analyst, Laboratory Supervisor, and Data Validator | Accuracy/Bias | If original sample result is at least 50x the instrument detection limit, five-fold dilution must agree within ±10% of original result. |

## SAP Worksheet #28 -- Laboratory QC Samples Table - GPL Laboratories

(UFP-QAPP Manual Section 3.4)

| | |
|---|---|
| Matrix | Aqueous field quality control and soil |
| Analytical Group | Nitroglycerin |
| Analytical Method / SOP Reference | 8330B/GPL Laboratories SOP-S.11 |

| QC Sample | Frequency/ Number | Method/SOP QC Acceptance Limits | Corrective Action | Person(s) Responsible for Corrective Action | DQI | Measurement Performance Criteria |
|---|---|---|---|---|---|---|
| Method Blanks | One per batch of 20 or less samples of the same matrix. | No target compounds >QLs. | Re-extract batch if affected compound is detected in any samples. If no volume to re-extract, data will be "B" flagged where required. | HPLC Analyst and Supervisor | Bias/Contamination | No target compounds > QLs |
| Surrogates | One per sample. | 4-Nitroaniline 50% to130% for water 31% to129% for solids | If surrogate recovery is less than QC limits, re-extract affected samples if volume is available. High surrogate failures are evaluated on sample-by-sample basis. | HPLC Analyst and Supervisor | Accuracy/Bias | 4-Nitroaniline 50% to 130% for water 31% to 129% for solids |
| Laboratory Control Spikes (LCSs) | One per batch of 20 or less sample of the same matrix. | Water and soil: %Recovery = 50 to 150 | If LCS recovery is low and volume is available, re-extract batch. If LCS recovery is high but sample results are <QLs, narrate failure. | HPLC Analyst and Supervisor | Precision/Accuracy/Bias | Water and soil: %Recovery = 50 to 150 |

| | |
|---|---|
| Matrix | Aqueous field quality control and soil |
| Analytical Group | Nitroglycerin |
| Analytical Method / SOP Reference | 8330B/GPL Laboratories SOP-S.11 |

| QC Sample | Frequency/ Number | Method/SOP QC Acceptance Limits | Corrective Action | Person(s) Responsible for Corrective Action | DQI | Measurement Performance Criteria |
|---|---|---|---|---|---|---|
| MS/MSDs | One per batch of 20 or less sample of the same matrix. | Water and soil: %Recovery = 50 to 150 RPD <25 | Corrective action will not be taken for samples when recoveries are outside limits and surrogate and LCS criteria are met. If both LCS and MS/MSD are unacceptable, reprepare samples and QC. | HPLC Analyst and Supervisor | Precision/Accuracy/Bias | Water and soil: %Recovery = 50 to150 RPD <25 |
| Confirmation Samples | Any sample with positive results. | RPD<40 from primary peak concentration | Results will be "J" flagged on report and difference mentioned in case narrative. If difference exceeds 70%, results will be reported or deleted based on discussion with Supervisor or QAM. | HPLC Analyst and Supervisor | Presence/Absence | RPD <40% |

| Matrix | Soil |
|---|---|
| Analytical Group | pH |
| Analytical Method / SOP Reference | SW-846 9045C/ SOP-187 |

| QC Sample | Frequency / Number | Method / SOP QC Acceptance Limits | Corrective Action | Person(s) Responsible for Corrective Action | Data Quality Indicator (DQI) | Measurement Performance Criteria |
|---|---|---|---|---|---|---|
| Laboratory Duplicates | One per prep batch of 10 or fewer samples. | ± 0.09 units | Recalibrate and reanalyze samples. | Analyst, Supervisor, and QAMr | Precision | ± 0.09 units |
| LCSs | Once per 20 samples. | ± 0.25 units | Re-analyze associated samples. If sample is within holding time, re-analyze affected sample batch. | Analyst, Supervisor, and QAM | Accuracy/Bias | ± 0.25 units |

## SAP Worksheet #29 – Project Documents and Records Table

(UFP-QAPP Manual Section 3.5.1)

| Document | Where Maintained |
|---|---|
| Sample Collection Documents and Records<br>Field logbook (and sampling notes)<br>Field sample forms (e.g. boring logs, sample log sheets, etc.)<br>Chain-of-custody forms<br>Sample shipment airbills<br>Equipment calibration logs<br>Photographs<br>FTMRs<br>Sampling and Analysis Plan<br>Field Sampling SOPs | Tetra Tech Project File, results will be discussed in subject documents. |
| Laboratory Documents and Records<br>Sample receipt/log-in forms<br>Sample storage records<br>Sample preparation logs<br>Standard traceability logs<br>Equipment calibration logs<br>Sample analysis run logs<br>Equipment maintenance, testing, and inspection logs<br>Reported results for standards, quality control checks, and quality control samples<br>Data completeness checklists<br>Sample storage and disposal records<br>Telephone logs<br>Extraction/clean-up records<br>Raw data | Tetra Tech Project File, long-term data package storage at third-party professional document storage firm, results will be discussed in subject documents. |
| Data Assessment Documents and Records<br>Field Sampling Audit Checklist (if an audit is conducted)<br>Analytical Audit Checklist (if an audit is conducted)<br>Data Validation Memoranda<br>Site Investigation Reports | Tetra Tech Project File, results will be discussed in subject documents. |

## SAP Worksheet #30 – Analytical Services Table

| Matrix | Analytical Group | Sample Locations/ID Numbers | 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) |
|---|---|---|---|---|---|---|
| Soil and aqueous quality control samples | PAHs | See Worksheet #18 | SW-846 8270C SIM | 21 calendar days | Empirical Laboratories<br>227 French Landing Drive<br>Suite 550<br>Nashville, TN 37228 Kim Kostzer (615)-345-1115 | NA |
| | Lead | See Worksheet #18 | SW-846 6010B | 21 calendar days | | |
| | pH | See Worksheet #18 | SW-846 9045 | 21 calendar days | | |
| | CEC | See Worksheet #18 | SW-846 | 21 calendar days | | |
| | TOC | See Worksheet #18 | SW-846 | 21 calendar days | | |
| | Nitroglycerin | See Worksheet #18 | SW-846 8330B | 21 calendar days | GPL Laboratories<br>7210A Corporate Court<br>Frederick, MD 21703<br>Chino Ortiz (301)-694-5310 | NA |

## SAP Worksheet #31 -- Planned Project Assessments Table

(UFP-QAPP Manual Section 4.1.1)

| Assessment Type | Frequency | Internal or External | Organization Performing Assessment | Person(s) Responsible for Performing Assessment (title and organizational affiliation) | Person(s) Responsible for Responding to Assessment Findings (title and organizational affiliation) | Person(s) Responsible for Identifying and Implementing Corrective Actions (CA) (title and organizational affiliation) | Person(s) Responsible for Monitoring Effectiveness of CA (title and organizational affiliation) |
|---|---|---|---|---|---|---|---|
| Field Supervision | Daily during sampling events | Internal | Tetra Tech | Tetra Tech FOL | Tetra Tech FOL | Tetra Tech FOL and field crew | Tetra Tech PM, QAM, and FOL |
| Project Supervision | Every sampling event | Internal | Tetra Tech | Tetra Tech PM | Tetra Tech FOL | Tetra Tech PM and FOL | Tetra Tech PM and FOL |
| Field Sampling System Audit | One per contract year | Internal | Tetra Tech | TBD | Tetra Tech PM and FOL | Tetra Tech Auditor and PM | NAVFAC AH<br>NAVFAC SE |
| Laboratory System Audit | Every 18 months | External | NFESC | Naval Facilities Engineering Service Center (NFESC) | Empirical Laboratories and GPL Laboratories Laboratory QAM or Laboratory Manager | Empirical Laboratories and GPL Laboratories Laboratory QAM or Manager | NFESC |

## SAP Worksheet #32 – Assessment Findings and Corrective Action Responses

(UFP-QAPP Manual Section 4.1.2)

| Assessment Type | Nature of Deficiencies Documentation | Individual(s) Notified of Findings (name, title, organization) | Time Frame of Notification | Nature of Corrective Action Response Documentation | Individual(s) Receiving Corrective Action Response (name, title, organization) | Time Frame for Response |
|---|---|---|---|---|---|---|
| Field Supervision | Site logbook and sample collection logs | Lawson Anderson, PM, Tetra Tech<br>Fred Grosskopf, FOL, Tetra Tech | Immediately | Entry in site logbook | Lawson Anderson, PM, Tetra Tech<br>Fred Grosskopf, FOL, Tetra Tech | 24 hours |
| Project Supervision | Written report | John Trepanowski, Program Manager (ER,N sites), Tetra Tech and<br>Debbie Humbert, Program Manager (BRAC sites), Tetra Tech | Monthly | Written memo | John Trepanowski, Program Manager (ER,N sites), Tetra Tech and<br>Debbie Humbert, Program Manager (BRAC Sites), Tetra Tech | Within 1 week of notification |
| Field Sampling System Audit | Audit checklist and written audit finding summary | Lawson Anderson, PM, Tetra Tech<br>Fred Grosskopf, FOL, Tetra Tech<br>John Trepanowski, Program Manager (ER,N sites), Tetra Tech and<br>Debbie Humbert, Program Manager (BRAC Sites), 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 | Tom Johnston and Kelly Carper, CLEAN QAMs, Tetra Tech<br>Designee, Field Auditor, Tetra Tech<br>John Trepanowski, Program Manager (ER,N sites), Tetra Tech and<br>Debbie Humbert, Program Manager (BRAC Sites), Tetra Tech | Within 48 hours of notification |
| Laboratory System Audit | Written audit report | Randy Ward, QAM, Empirical Laboratories<br>Virginia Zusman, QAM, GPL Laboratories | Not specified by NFESC | Letter | NFESC | Specified by NFESC |

## SAP Worksheet #33 – QA Management Reports Table

(UFP QAPP Manual Section 4.2)

| Type of Report | Frequency (daily, weekly monthly, quarterly, annually, etc.) | Projected Delivery Date(s) | Person(s) Responsible for Report Preparation (title and organizational affiliation) | Report Recipient(s) (title and organizational affiliation) |
|---|---|---|---|---|
| Data validation report | Per SDG | Completion of data validation | DVM and staff chemists, Tetra Tech | PM and Project File, Tetra Tech |
| Major analysis problem identification (internal memo) | When persistent analysis problems are detected | Immediately | NAVFAC ATL<br>NAVFAC SE, Tetra Tech | PM, CLEAN QAMs, Program Manager, and Project File, Tetra Tech |
| Project monthly progress report | Monthly for duration of project | Monthly | PM, Tetra Tech | Project File, Navy |
| Field progress report | Daily, oral, during the course of sampling | Every day that field sampling occurs | FOL, Tetra Tech | PM, Tetra Tech |
| Laboratory QA report | When significant plan deviations result from unanticipated circumstances | Immediately | PM, Empirical Laboratories<br>PM, GPL Laboratories | Project File, Tetra Tech |
| Audit report | In conjunction with audits | After completion of audits (usually 3 weeks) | Auditor(s), Tetra Tech | PM, QAMs, Tetra Tech, and audited entity |

## SAP Worksheet #34 – Verification (Step I) Process Table

(UFP-QAPP Manual Section 5.2.1)

| Verification Input | Description | Internal / External | Responsible for Verification (name, organization) |
|---|---|---|---|
| Chain-of-custody forms | The Tetra Tech FOL or designee will review and sign the chain-of-custody form to verify that all samples listed are included in the shipment to the laboratories and that the sample information is accurate. The forms will be signed by the sampler and a copy will be retained for the project file, PM, and data validators. See SOP-03 | Internal | Sampler and FOL, Tetra Tech |
| SAP sample tables | Verify that all proposed samples listed in the SAP tables have been collected. | Internal | FOL or designee, Tetra Tech |
| Sample log sheets | Verify that information recorded on the log sheets is accurate and complete. | Internal | FOL or designee, Tetra Tech |
| Sample coordinates | Verify that sample locations are correct and in accordance with the SAP proposed locations. | Internal | FOL or designee, Tetra Tech |
| Field QC samples | Check that field QC samples listed in Worksheet #20 were collected as required. | Internal | FOL or designee, Tetra Tech |
| Chain-of-custody forms | The laboratory sample custodian will review the sample shipment for completeness, and integrity, and sign accepting the shipment. The data validators will check that the chain-of-custody form was signed/dated by the Tetra Tech FOL or designee relinquishing the samples and also by the laboratory sample custodian receiving the samples for analyses. | Internal/ External | 1 - Laboratory sample custodian<br>2 -Data Validators, Tetra Tech |
| Analytical data package | All analytical data packages will be verified internally for completeness by the laboratory performing the work. The laboratory QAM will sign the case narrative for each data package. | Internal | Randy Ward QAM, Empirical Laboratories<br>Virginia Zusman, QAM, GPL Laboratories, |
| Analytical data package | The data package will be verified for completeness by Tetra Tech data validators. Missing information will be requested from the laboratory, and validation will be suspended until missing data are received. | External | Data Validators, Tetra Tech |
| Analytical data package and electronic data deliverables | The electronic data will be verified against the chain-of-custody form and hard copy data package for accuracy and completeness. Laboratory analytical results will be verified and compared to the electronic analytical results for accuracy. Sample results will be evaluated for laboratory contamination and will be qualified for false positives using the laboratory method/preparation blank summaries. Positive results reported between the method detection limit and reporting limit will be qualified as estimated. Extraneous laboratory qualifiers will be removed from the validation qualifiers. | External | Data Validators, Tetra Tech |

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

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

(UFP-QAPP Manual Section 5.2.2) (Figure 37, page 110 UFP-QAPP Manual) (Table 9 UFP-QAPP Manual)

| Step IIa / IIb | Validation Input | Description | Responsible for Validation (name, organization) |
|---|---|---|---|
| IIa | Field SOPs/Field Logs/Sample Collection Logs | Ensure that all sampling SOPs were followed. Verify that deviations have been documented and MPCs have been achieved. Particular attention will be given to verifying that samples were correctly identified, that sampling location coordinates are accurate, and that documentation establishes an unbroken trail of chain-of-custody from sample collection to report generation. Verify that the correct sampling and analytical methods/SOPs were applied. Verify that the SAP was implemented and carried out as written and that any deviations are documented. | PM, FOL, or designee, Tetra Tech |
| IIa | Analytical SOPs | Ensure that all laboratory SOPs were followed. Verify that the correct analytical methods/SOPs were applied. | Laboratory QAM (Empirical Laboratories and GPL Laboratories) |
| 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. | Randy Ward, QAM, Empirical Laboratories<br>Virginia Zusman, QAM, GPL Laboratories |
| IIa | Chain-of-Custody forms | Ensure that the custody and integrity of the samples were maintained from collection to analysis and that custody records are complete and any deviations are recorded. | Project Chemist or Data Validators, Tetra Tech |
| IIa | Holding Times | Review that the samples were shipped and stored at the required temperature and that sample pH values 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. | Project Chemist or Data Validators, Tetra Tech |
| IIa/IIb | Laboratory Data Results for Accuracy | Ensure that the laboratory QC samples listed in Worksheet #28 were analyzed and that the MPCs 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. | Project Chemist or Data Validators, Tetra Tech |

| Step IIa / IIb | Validation Input | Description | Responsible for Validation (name, organization) |
|---|---|---|---|
| IIa/IIb | Field and Laboratory Duplicate Analyses for Precision | Check field sampling precision by calculating RPDs for field duplicate samples. Check laboratory precision by reviewing the RPD or percent difference values from laboratory duplicate analyses, MS/MSDs, and LCSs/LCSD. Ensure compliance with the methods and project MPCs accuracy goals listed in Worksheet #12. | Project Chemist or Data Validators, Tetra Tech |
| IIa/IIb | Sample Results for Representativeness | Check that the laboratory recorded the temperature at sample receipt and the pH of chemically preserved samples to ensure sample integrity from sample collection to analysis | Tetra Tech Project Chemist or Data Validators |
| IIa/IIb | PALs | Discuss the impact on matrix interferences or sample dilutions performed because of high concentrations of one or more contaminant on the other target compounds reported as not detected. Document this usability issue and inform the PM. | Tetra Tech Project Chemist or Data Validators |
| 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 database qualified data depicting data qualifiers, and data qualifiers codes that summarize the reason for data qualifications. Determine if the data met the MPCs and determine the impact of any deviations on the technical usability of the data. | Tetra Tech Project Chemist or Data Validators |
| 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 will contact the Tetra Tech PM. | Tetra Tech PM or designee |

| Step IIa / IIb | Validation Input | Description | Responsible for Validation (name, organization) |
|---|---|---|---|
| IIa, IIb | Documentation of Analytical Reports for Completeness | Ensure that the chain-of-custody form generated in the field agrees with analytical data received from the laboratories and 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 elements of the data package required 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. | Project Chemist or Data Validators, Tetra Tech |
| IIa/IIb | PALs | Review and add PALs to the laboratory electronic data deliverable. Flag samples and notify PM of samples that exceed PALs as listed in Worksheet #15. | PM or designee, Tetra Tech |
| IIb | Project QLs for Sensitivity | Ensure that the PQLs listed in Worksheet #15 were achieved. | Project Chemist or Data Validators, Tetra Tech |
| IIb | Analytical Data Deviations | Determine the impact of any deviation from sampling or analytical methods, SOPs requirements, and matrix interferences on the analytical results. | Project Chemist or Data Validators, Tetra Tech |

**SAP Worksheet #36 –Analytical Data Validation (Steps IIa and IIb) Summary Table**

(UFP-QAPP Manual Section 5.2.2.1) (Figure 37, page 110 UFP-QAPP Manual)

| Step IIa/IIb | Matrix | Analytical Group | Validation Criteria | Data Validator (title and organizational affiliation) |
|---|---|---|---|---|
| IIa and IIb | Soil and aqueous quality control samples | PAHs | 100% Full data validation will be performed. SW-846 8270C SIM method-specific criteria and those criteria listed in Worksheets #12, 15, 24, and 28. If not included in these worksheets, the logic outlined in USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review EPA-540/R-99-008, October 1999, will be used to apply qualifiers to data. | Data Validators, Tetra Tech |
| IIa and IIb | Soil and aqueous quality control samples | Lead | 100% Full data validation will be performed. SW-846 6010B method-specific criteria and those listed in Worksheets #12, 15, 24, and 28. If not included in these worksheets, the logic outlined in USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review EPA-540-R-04-004, October 2004, will be used to apply qualifiers to data. | Data Validators, Tetra Tech |
| IIa and IIb | Soil and aqueous quality control samples | Nitroglycerin | 100% Full data validation will be performed. SW-846 8330B method-specific criteria and those listed in Worksheets #12, 15, 24, and 28. If not included in these worksheets, the logic outlined in USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review EPA-540/R-99-008, October 1999, will be used to apply qualifiers to data. | Data Validators, Tetra Tech |

## SAP Worksheet #37 -- Usability Assessment

(UFP-QAPP Manual Section 5.2.3)

**Usability assessment process:**

Upon completion of data verification and validation, in which required data quality procedures are verified to have been followed and data completeness has been verified, a data usability assessment will be conducted. This assessment will be directed at reconciling data collected as part of this project with the DQOs established in this SAP. An evaluation of where samples were collected compared to the intended collection locations and depths will be part of this assessment. Project assumptions will be tested, if possible, to verify that significant deviations from assumptions did not occur. If significant deviations did occur, the effect on the deviations of decision making will be evaluated. The effect of deviations from SOPs or this SAP, if any, will also be evaluated for their effect on decision making.

The usability of the data directly affects whether project objectives can be achieved. The data characteristics discussed below will be evaluated at a minimum. The results of these evaluations will be included in the project reports. 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 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 compared to collected samples/analyses. If deviations from the scheduled sample collection or analyses are identified, the Tetra Tech PM and risk assessor 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 or BEC and other Project Team members, as necessary (determined by the Navy RPM or BEC), 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 reports.

**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, MS, and LSCs. If the goals are not met, limitations on the use of the data will be described in the project reports. Bias of the qualified results and a description of the impact of identified non-compliances on a specific data package or on overall project data will be described in the project reports.

**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 the 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 for 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 the 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 Worksheets #14 and 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.

**Field XRF/Laboratory Lead Data Correlation**

The project statistician will evaluate the correlation of field XRF data to laboratory data. Factors considered in this evaluation will include the magnitude of the slope and intercept of the correlation

equation, distribution of data points across the plotted concentration range, and values of the correlation coefficient. If the coefficient is less than 0.65 or the plotted data do not appear to be well correlated in accordance with standard statistical principles, limitations on the use of the data will be described in the project report.

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

After completion of data validation, the data and data quality will be reviewed to determine whether sufficient data of acceptable quality are available for decision making. In addition to the evaluations described above, a series of inspections and statistical analyses will be performed to estimate these characteristics. The statistical evaluations will include simple summary statistics for target analytes, such as maximum concentration, minimum concentration, number of samples with non-detected results, number of samples with detections, and proportion of samples with detected and non-detected results. The Project Team members identified by the PM will assess whether the data collectively support the attainment of project objectives. They will consider whether any missing or rejected data have compromised the ability to make decisions or to make the decisions with the desired level of confidence. The data will be evaluated to determine whether missing or rejected data can be compensated for 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.

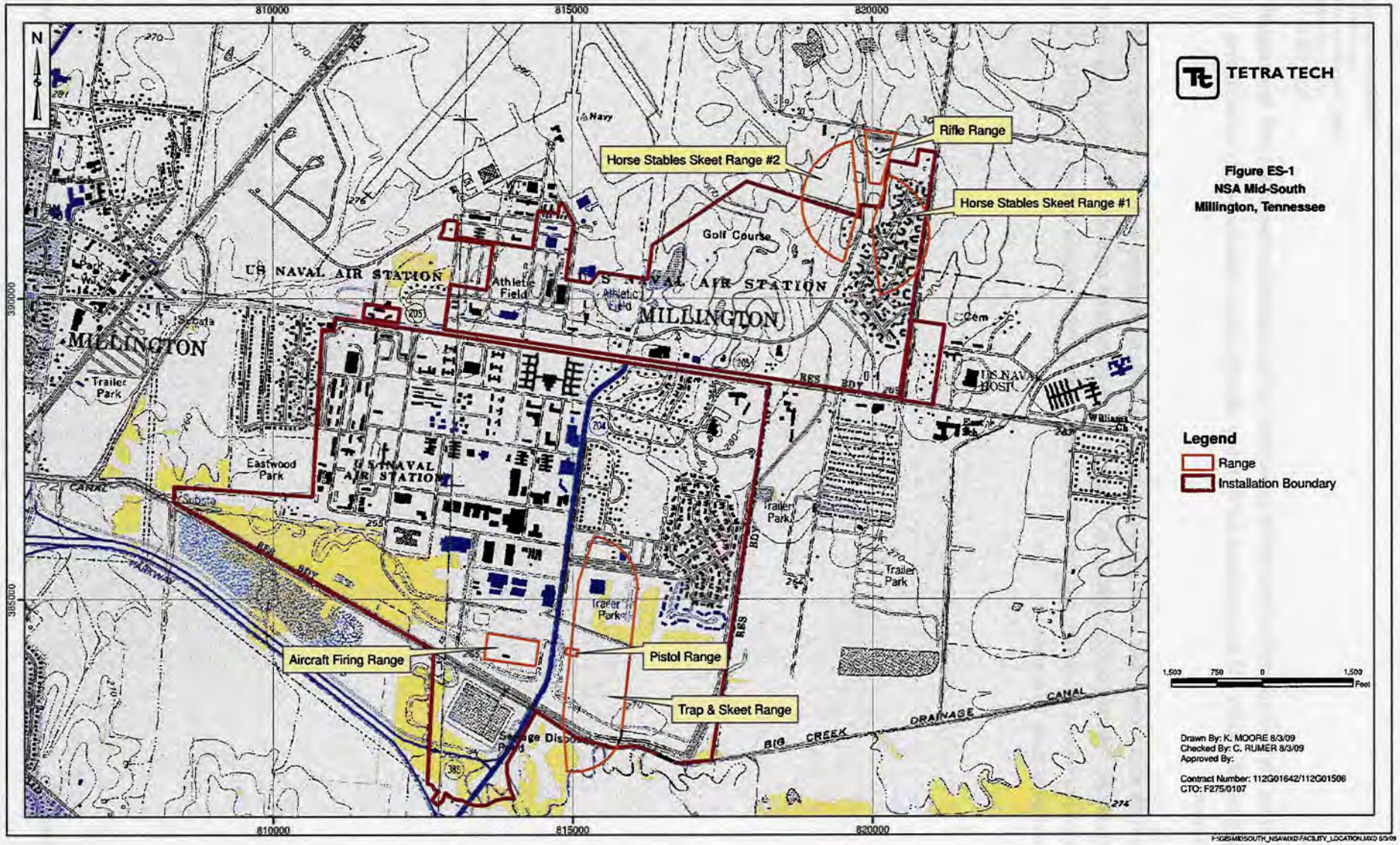
For statistical comparisons and mathematical manipulations, non-detected values will be represented by a concentration equal to one-half of the sample-specific reporting limit. Duplicate results (original and duplicate) will not be averaged for the purpose of representing ranges of concentrations; however,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

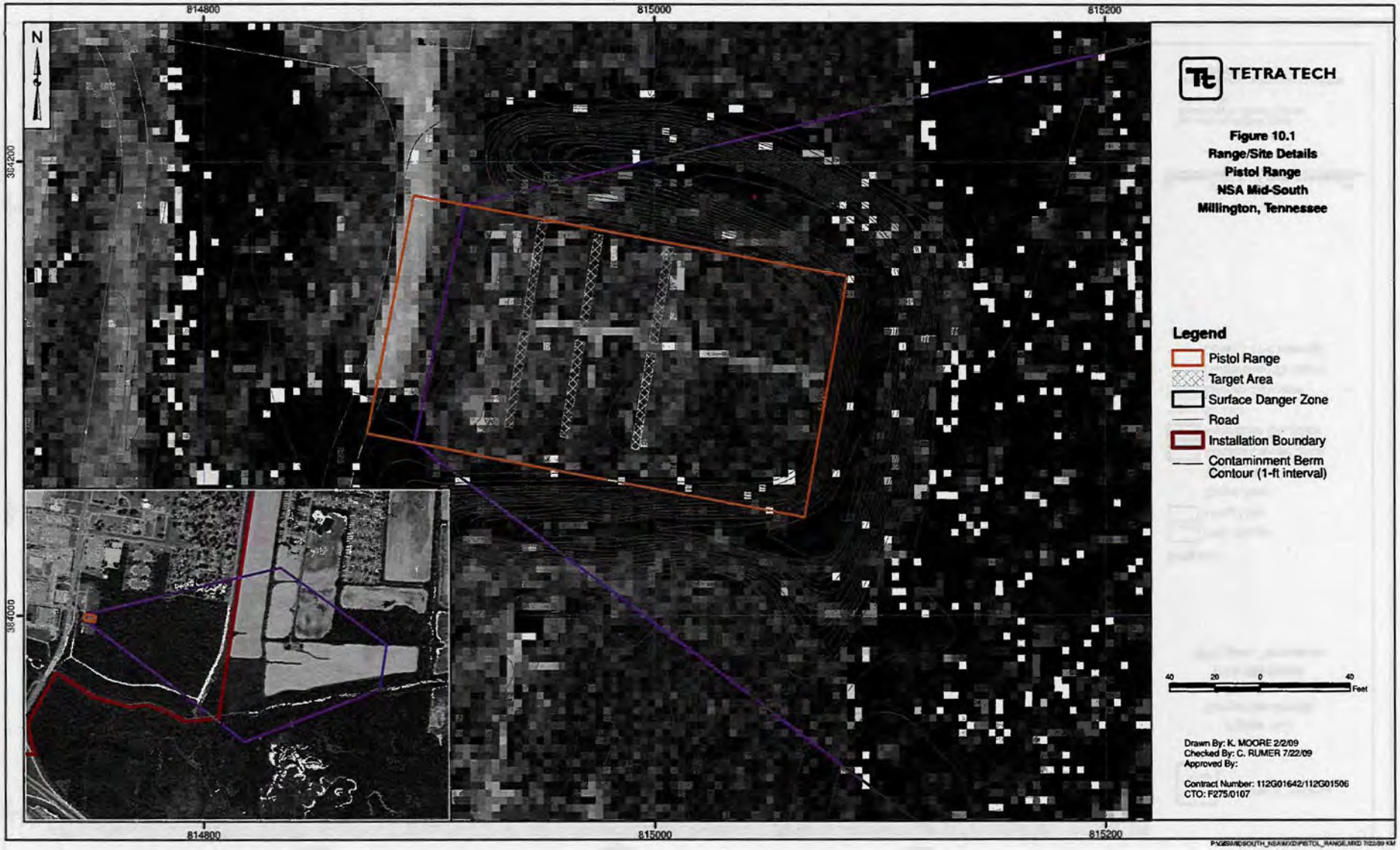
**Identify the personnel responsible for performing the usability assessment:**

The Project Team will participate in the data usability assessment. In general, large deviations from quality specifications will result in less useful data than small deviations. This assessment, however, will depend largely on which parameters and samples exhibited the deviations and to what degree.

**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 reports will identify and describe the data usability limitations and suggest resampling or other corrective actions, if necessary.


TETRA TECH
Figure ES-1
NSA Mid-South
Millington, Tennessee
Legend
Range
Installation Boundary
Rifle Range
Horse Stables Skeet Range #2
Horse Stables Skeet Range #1
Aircraft Firing Range
Pistol Range
Trap & Skeet Range
US NAVAL AIR STATION
MILLINGTON
Golf Course
Athletic Field
Trailer Park
Eastwood Park
Cem
Big Creek Drainage Canal
810000
815000
820000
390000
385000
1,500 750 0 1,500 Feet
Drawn By: K. MOORE 8/3/09
Checked By: C. RUMER 8/3/09
Approved By:
Contract Number: 112G01642/112G01506
CTO: F275/0107
P:\GIS\MIDSOUTH_NSA\MXD\FACILITY_LOCATION.MXD 8/3/09


814800
815000
815200
384200
384000
N
TETRA TECH
Figure 10.1
Range/Site Details
Pistol Range
NSA Mid-South
Millington, Tennessee
Legend
Pistol Range
Target Area
Surface Danger Zone
Road
Installation Boundary
Contaminment Berm Contour (1-ft interval)
40 20 0 40
Feet
Drawn By: K. MOORE 2/2/09
Checked By: C. RUMER 7/22/09
Approved By:
Contract Number: 112G01642/112G01506
CTO: F275/0107

819500
820000
820500
393000
392500
392000
N
Kerrville Rosemark Road
Alig Street Extension
Former Military Housing Complex
TETRA TECH
Figure 10.2
Range/Site Details
Rifle Range
NSA Mid-South
Millington, Tennessee
Legend
Rifle Range
Firing Line
Target Area
Target Area Berm
Surface Danger Zone
Installation Boundary
Stream
Existing Structure
Demolished Structure
Contour (1-ft interval)
150 75 0 150
Feet
Drawn By: K. MOORE 2/2/09
Checked By: C. RUMER 7/22/09
Approved By:
Contract Number: 112G01642/112G01506
CTO: F275/0107
P:\GIS\MIDSOUTH_NSA\MXD\RIFLE_RANGE.MXD 6/10/09 KM

813500
814000
814500
384000
N
TETRA TECH
Figure 10.3
Range/Site Details
Aircraft Firing Range
NSA Mid-South
Millington, Tennessee
Legend
Aircraft Firing Range
Firing Line
Location of Contaminent Berm
Surface Danger Zone
Installation Boundary
Road
Existing Structure
Contour (1-ft interval)
100 50 0 100
Feet
Drawn By: K. MOORE 2/2/09
Checked By: C. RUMER 7/22/09
Approved By:
Contract Number: 112G01642/112G01506
CTO: F275/0107

TETRA TECH

**Figure 10.4**
**Range/Site Details**
**Trap/Skeet Ranges 1&2**
**NSA Mid-South**
**Millington, Tennessee**

**Legend**

- Trap Skeet Range
- Pistol Range
- Firing Line
- SWMU 2 Boundary
- Target Area
- Installation Boundary
- Road
- Stream
- Existing Structure
- Contour (5-ft interval)

300 150 0 300 Feet

Drawn By: K. MOORE 2/2/09
Checked By: C. RUMER 7/22/09
Approved By:

Contract Number: 112G01642/112G01506
CTO: F2/5/0107

P:\GIS\MIDSOUTH_NSA\MXD\TRAP_SKEET_RANGE.MXD 7/22/09 KM

TETRA TECH

**Figure 10.5**
**Range/Skeet Details**
**Horse Stable Skeet Range #1**
**NSA Mid-South**
**Millington, Tennessee**

**Legend**

- Horse Stable Skeet Range #1
- Rifle Range
- Firing Line
- Surface Danger Zone
- Installation Boundary
- Road
- Stream
- Existing Structure
- Demolished Structure
- Contour (1-ft interval)

200 100 0 200 Feet

Drawn By: K. MOORE 2/2/09
Checked By: C. RUMER 7/22/09
Approved By:

Contract Number: 112G01642/112G01506
CTO: F275/0107

P:\GIS\MIDSOUTH_NSA\MXD\HORSE_STABLE_SKEET1.MXD 7/20/09 KM

TETRA TECH

**Figure 10.6**
**Range/Skeet Details**
**Horse Stable Skeet Range #2**
**NSA Mid-South**
**Millington, Tennessee**

**Legend**

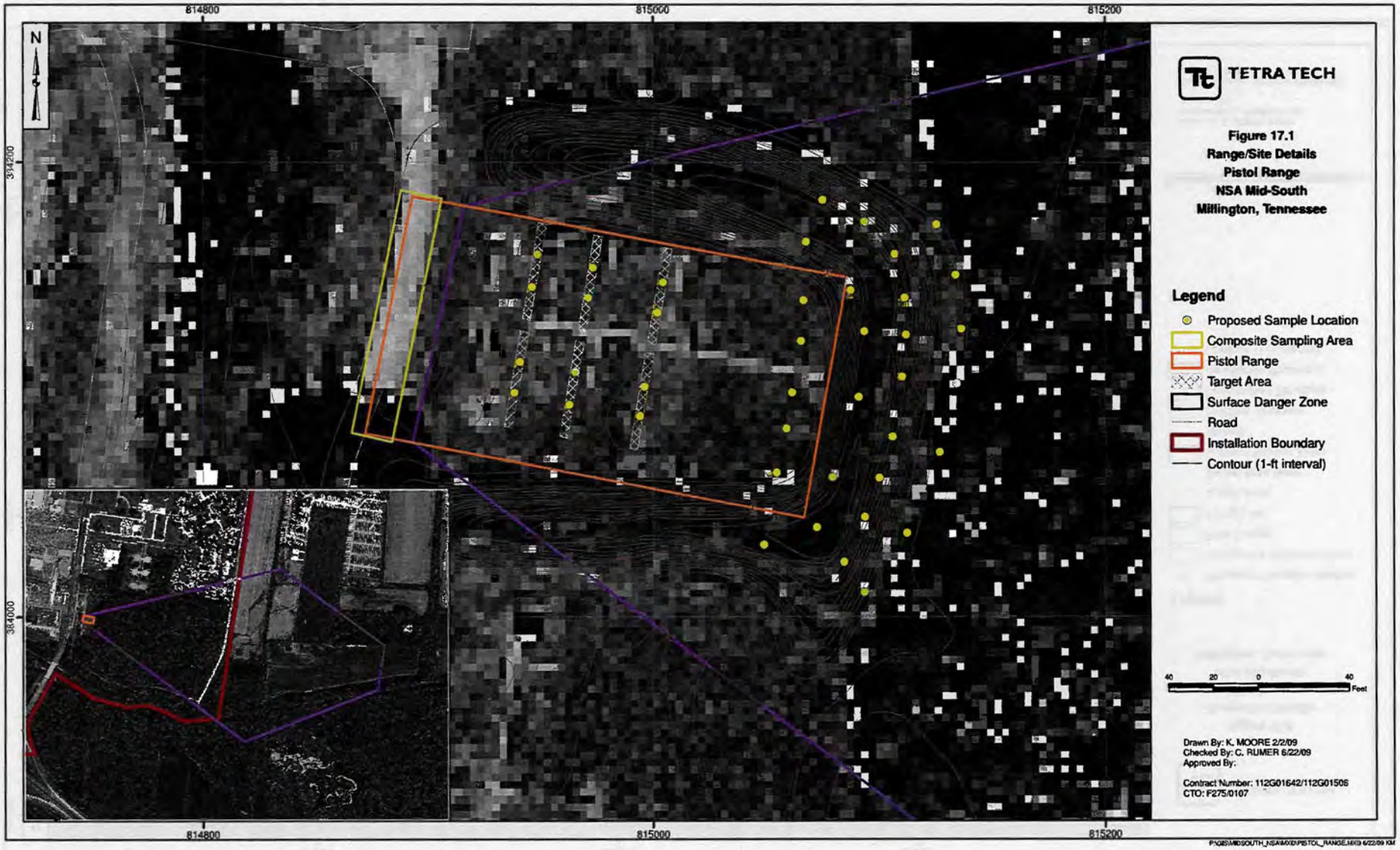
- Horse Stable Skeet Range #2
- Rifle Range
- Firing Line
- Surface Danger Zone
- Installation Boundary
- Road
- Stream
- Existing Structure
- Demolished Structure
- Water
- Contour (1-ft interval)

200 100 0 200 Feet

Drawn By: K. MOORE 2/2/09
Checked By: C. RUMER 7/22/09
Approved By:

Contract Number: 112G01642/112G01506
CTO: F275/0107

P:\GIS\MIDSOUTH_NSA\MXD\HORSE_STABLE_SHEET2.MXD 7/22/09 KM


814800
815000
815200
314200
314000
N
TETRA TECH
Figure 17.1
Range/Site Details
Pistol Range
NSA Mid-South
Millington, Tennessee
Legend
Proposed Sample Location
Composite Sampling Area
Pistol Range
Target Area
Surface Danger Zone
Road
Installation Boundary
Contour (1-ft interval)
40 20 0 40
Feet
Drawn By: K. MOORE 2/2/09
Checked By: C. RUMER 6/22/09
Approved By:
Contract Number: 112G01642/112G01506
CTO: F275/0107

819500
820000
820500
393000
392500
392000
N
Kerrville Rosemark Road
Airport Street Extension
Former Military Housing Complex
TETRA TECH
Figure 17.2
Range/Site Details
Rifle Range
NSA Mid-South
Millington, Tennessee
Legend
Proposed Sample Location
Composite Sampling Area
Rifle Range
Firing Line
Target Area
Target Area Berm
Surface Danger Zone
Stream
Existing Structure
Demolished Structure
Installation Boundary
Contour (1-ft interval)
150 75 0 150
Feet
Drawn By: K. MOORE 2/2/09
Checked By: C. RUMER 8/3/09
Approved By:
Contract Number: 112G01642/112G01506
CTO: F275/0107
P:\GIS\MIDSOUTH_NSA\MXD\RIFLE_RANGE_SAMPLES.MXD 8/3/09 KM

813500
814000
814500
384000
N
TETRA TECH
Figure 17.3
Range/Site Details
Aircraft Firing Range
NSA Mid-South
Millington, Tennessee
Legend
Proposed Sample Location
Composite Sampling Area
Aircraft Firing Range
Firing Line
Target Area
Surface Danger Zone
Road
Structure
Installation Boundary
Contour (1-ft interval)
100 50 0 100
Feet
Drawn By: K. MOORE 2/2/09
Checked By: C. RUMER 6/22/09
Approved By:
Contract Number: 112G01642/112G01506
CTO: F275/0107

TETRA TECH

**Figure 17.4**
**Range/Site Details**
**Trap/Skeet Ranges 1&2**
**NSA Mid-South**
**Millington, Tennessee**

**Legend**

- Proposed Sample Location
- Composite Sampling Area
- Trap Skeet Range
- Firing Line
- SWMU 2 Boundary
- Target Area
- Road
- Stream
- Installation Boundary

300 150 0 300 Feet

Drawn By: K. MOORE 2/2/09
Checked By: R. BAILEY 2/1/10
Revised By: K. MOORE 1/26/10

Contract Number: 112G01642/112G01506
CTO: F275/0107

P:\GIS\MIDSOUTH_NSA\MXD\TRAP_SKEET_RANGE_SAMPLES.MXD 2/1/10 KM

TETRA TECH

**Figure 17.5**
**Range/Skeet Details**
**Horse Stable Skeet Range #1**
**NSA Mid-South**
**Millington, Tennessee**

**Legend**

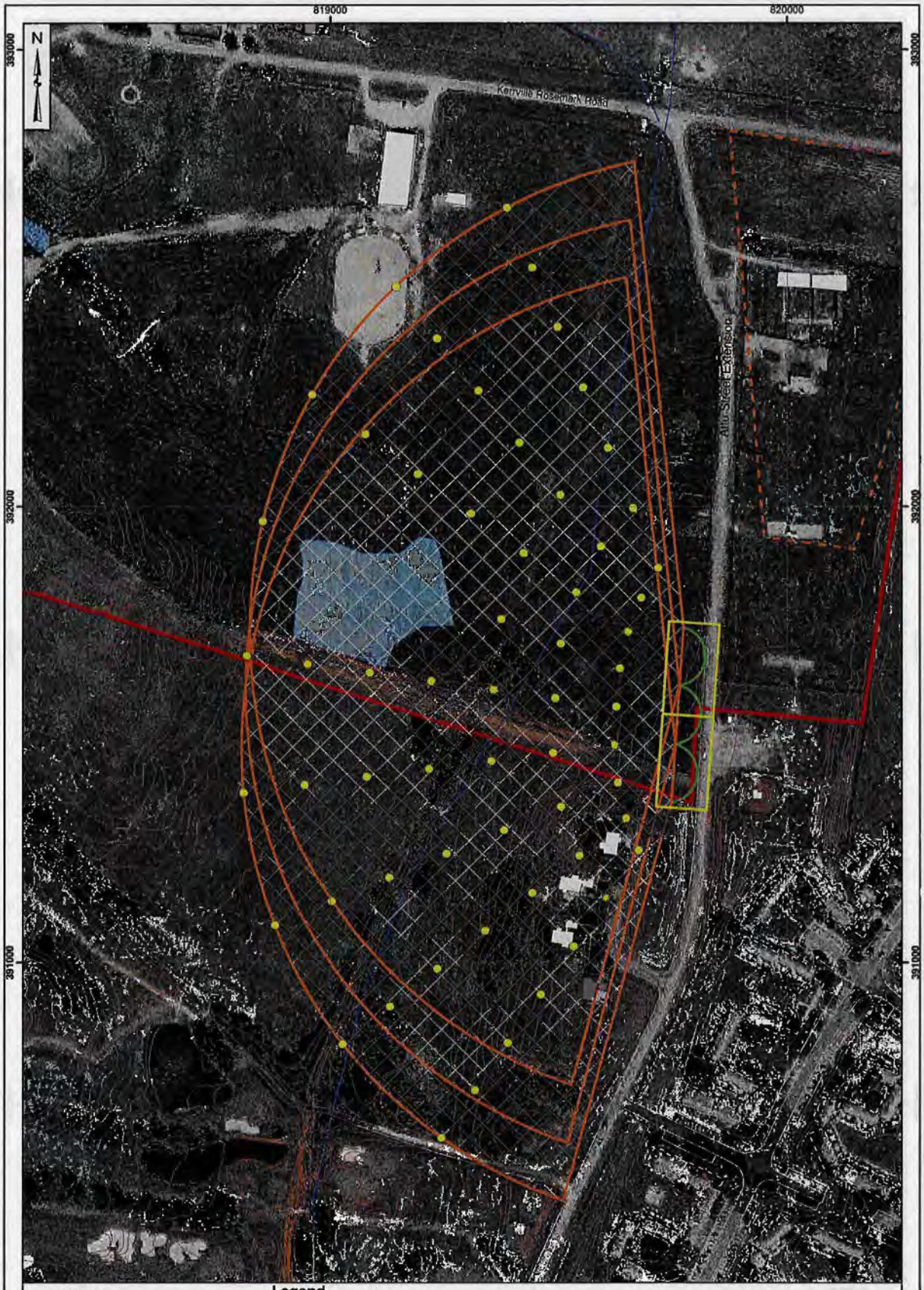
- Proposed Sample Location
- Composite Sampling Area
- Horse Stable Skeet Range #1
- Firing Line
- Target Area
- Road
- Stream
- Existing Structure
- Demolished Structure
- Installation Boundary
- Contour (1-ft interval)

200 100 0 200 Feet

Drawn By: K. MOORE 2/2/09
Checked By: R. BAILEY 2/1/10
Revised By: K. MOORE 1/26/10

Contract Number: 112G01642/112G01506
CTO: F275/0107

P:\GIS\MIDSOUTH_NSA\MXD\HORSE_STABLE_SKEET1_SAMPLES.MXD 2/1/10 KM




TETRA TECH

**Figure 17.6**
**Range/Skeet Details**
**Horse Stable Skeet Range #2**
**NSA Mid-South**
**Millington, Tennessee**

**Legend**

- Proposed Sample Location
- Composite Sampling Area
- Horse Stable Skeet Range #2
- Rifle Range
- Firing Line
- Target Area
- Road
- Stream
- Existing Structure
- Demolished Structure
- Water
- Installation Boundary
- Contour (1-ft interval)

200 100 0 200 Feet

Drawn By: K. MOORE 2/2/09
Checked By: C. RUMER 8/3/09
Approved By:

Contract Number: 112G01642/112G01506
CTO: F275/0107

P:\GIS\MIDSOUTH_NSA\MXD\HORSE_STABLE_SKEET2_SAMPLES.MXD 8/3/09 KM

# REFERENCES

Carmichael, Parks, Kingsbury, and Ladd, 1997. Hydrogeology and Ground-Water Quality at Naval Support Activity Memphis, Millington, Tennessee.

EnSafe, Inc., 2005. Groundwater Monitoring Well Management Plan, Revision 8.

EnSafe/Allen and Hoshall, 1996. Table 1, Summary of Reference Concentrations, Technical Memorandum, Naval Support Activity, Memphis, Tennessee.

Kingsbury and Carmichael, 1995. Hydrogeology of Post-Wilcox Group Stratigraphic Units in the Area of the Naval Air Station Memphis, Near Millington, Tennessee.

Malcolm Pirnie, 2005. Preliminary Assessment. August.

Oak Ridge National Laboratory (ORNL), 2009. Regional Screening Levels. April.

U.S. Environmental Protection Agency (USEPA), 2002. Guidance for Quality Assurance Project Plans, QA/G-5, EPA/240/R-02/009. December.

USEPA, 2005. Uniform Federal Policy for Quality Assurance Project Plans (UFP-QAPP), Evaluating, Assessing, and Documenting Environmental Data Collection and Use Programs (Final Version 1), EPA-505-B-04-900A. March.

USEPA, 2006. Guidance on Systematic Planning Using the Data Quality Objectives Process. February.

# APPENDIX A

# STANDARD OPERATING PROCEDURES

# APPENDIX A

## SOP TABLE OF CONTENTS


| | |
|---|---|
| SOP-01 | Sample Labeling |
| SOP-02 | Sample Identification Nomenclature |
| SOP-03 | Sample Custody and Documentation of Field Activities |
| SOP-04 | Decontamination of Field Sampling Equipment |
| SOP-05 | Soil Coring and Sampling Using Hand Auger Techniques |
| SOP-06 | Borehole Advancement and Soil Coring and Sampling Using Direct-Push Technology |
| SOP-07 | Surface Water Sampling |
| SOP-08 | Sediment Sampling |
| SOP-09 | Management of Investigation-Derived Waste |
| SOP-10 | Borehole and Soil Sample Logging |
| SOP-11 | Sample Preservation, Packaging, and Shipping |
| SOP-12 | Calibration and Care of Water Quality Meter |
| SOP-13 | Use of Field Portable X-Ray Fluorescence Analysis of Soil and Sediment |

# STANDARD OPERATING PROCEDURE

# SOP- 01

# SAMPLE LABELING

## 1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the procedures to be used for labeling sample containers. Sample labels are used to document the sample ID, date, time, analysis to be performed, preservative, matrix, sampler, and the analytical laboratory. A sample label will be attached to each sample container.

## 2.0 REQUIRED FIELD FORMS AND EQUIPMENT

**Writing utensil (preferably black pen with indelible ink)**

**Disposable medical-grade gloves (e.g. latex, nitrile)**

**Sample log sheets**

**Required sample containers**: All sample containers for analysis by fix-based laboratories will be supplied and deemed certified clean by the laboratory.

**Preprinted sample labels**

**Chain-of-custody records**

**Sealable polyethylene bags**

**Heavy-duty cooler**

**Ice**

## 3.0 PROCEDURES

3.1 The following information will be electronically printed on each sample label prior to mobilizing for field activities. Additional "generic" labels will also be printed prior to mobilization to be used for field QC and backups.

- Project number
- Sample location ID
- Contract Task Order number

- Sample ID
- Matrix
- Preservative
- Analysis to be performed
- Laboratory name

3.2 Select the container(s) that are appropriate for a given sample. Select the sample-specific ID label(s), complete date, time, and sampler name, and affix to the sample container(s).

3.3 Fill the appropriate containers with sample material. Securely close the container lids without overtightening.

3.4 Place the sample container in a sealable polyethylene bag and place in a cooler containing ice.

Example of a sample label is attached at the end of this SOP.

## 4.0 ATTACHMENTS

1. Sample Label

**ATTACHMENT 1**
**SAMPLE LABEL**

| | | |
|---|---|---|
| Tetra Tech NUS, Inc.<br>661 Andersen Drive<br>Pittsburgh, 15220<br>(412)921-7090 | **Project:**<br>**Location:**<br>**CTO:** | |
| **Sample No:** | | **Matrix:** |
| **Date:** | **Time:** | **Preserve:** |
| **Analysis:** | | |
| **Sampled by:** | **Laboratory** | |

# STANDARD OPERATING PROCEDURE

# SOP-02

# SAMPLE IDENTIFICATION NOMENCLATURE

## 1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to establish a consistent sample nomenclature system that will facilitate subsequent data management at NSA Mid-South. The sample nomenclature system has been devised such that the following objectives can be attained.

- Sorting of data by site, location, or matrix
- Maintenance of consistency (field, laboratory, and database sample numbers)
- Accommodation of all project-specific requirements
- Accommodation of laboratory sample number length constraints
- Ease of sample identification

The Maryland Department of Environmental Protection must approve any deviations from this procedure.

## 2.0 REQUIRED FIELD FORMS AND EQUIPMENT

**Writing utensil (preferably black pen with indelible ink)**

**Sample container labels**

## 3.0 SAMPLE IDENTIFICATION NOMENCLATURE

### 3.1 Samples

All samples will be properly labeled with a sample label affixed to the sample container. Each sample will be assigned a unique sample tracking number.

#### 3.1.1 Sample Numbering Scheme

The sample tracking number will consist of a four- or five-segment alpha-numeric code that identifies the sample's associated NSA Mid-South site, sample type, location, and for aqueous samples, where applicable, whether a sample is filtered, and/or the sample round number. For soil samples, the final four tracking numbers will identify the depth in units of feet below ground surface (bgs) at which the sample

was collected. For sediment samples, the final four tracking numbers will identify the depth in units of inches bgs at which the sample was collected.

The alphanumeric coding to be used is explained in the following diagram and subsequent definitions:

| AAA | AA | NNN | NNNN (Soils and Sediment only) |
|---|---|---|---|
| Site Acronym | Matrix | Sample Location Number | Sequential depth interval from freshly exposed surface |

**Character Type:**

A = Alpha
N = Numeric

**Site Name (NNN):**

HSSR2 = Horse Stable Skeet Range #2
RR = Rifle Range
PR = Pistol Range
TSR = Trap/Skeet Ranges #1 and #2
AFR = Aircraft Firing Range
HSSR1 = Horse Stable Skeet Range #1

**Matrix Code (AA):**

SS = Surface Soil Sample
SB = Subsurface Soil Sample
SD = Sediment Sample
SW = Surface Water Sample

**Location Number (NNN):**

Sequential number beginning with "001" for each matrix.

**Depth Interval:**

This code section will be used for soil and sediment samples only.

The depth code is used to note the depth 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 (soil) or inches (sediment) bgs of the sample. 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.

### 3.1.2 Examples of Confirmation Sample Nomenclature

A surface soil sample collected from the Pistol Range, sampling location 003, to a depth of 1 foot bgs would be labeled as "PRSS0030001". A sediment sample collected from the Horse Stable Skeet Range #1, sampling location 010, to a depth of 6 inches bgs would be labeled as "HSSR1SD0100006".

## 3.2 Field Quality Assurance/Quality Control (QA/QC) Sample Nomenclature

Field QA/QC samples are described in this UFP SAP. They will be designated using a different coding system than the one used for regular field samples.

### 3.2.1 QC Sample Numbering

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 |
|---|---|---|
| QC Type | Date | Sequence Number (per day) |

**Character Type:**

A = Alpha
N = Numeric

**QC Types:**

FD = Field Duplicate
RB = Rinsate Blank
SB = Source Blank

The sampling time recorded on the Chain-of-Custody Form, labels, and tags for field 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 sample log sheets and will document the location of the duplicate sample (sample log sheets are not provided to the laboratory).

### 3.2.2 Examples of Field QA/QC Sample Nomenclature

The first duplicate of the day at the Pistol Range site for a surface soil sample collected on March 24, 2008 would be designated as FD03240801.

The third duplicate of the day taken at the Pistol Range site of a surface soil sample collected on April 12, 2008 would be designated as FD04120803.

The first trip blank associated with samples collected on March 18, 2008 would be designated as TB03180801.

# STANDARD OPERATING PROCEDURE
# SOP-03

# SAMPLE CUSTODY AND DOCUMENTATION OF FIELD ACTIVITIES

## 1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedures for sample custody and documentation of field sampling and field analyses activities.

## 2.0 REQUIRED FIELD FORMS AND EQUIPMENT

The following logbooks, forms, labels, and equipment are required.

**Writing utensil (preferably black pen with indelible ink)**
**Site logbook**
**Field logbook**
**Sample label**
**Chain-of-Custody Form**
**Custody seals**
**Equipment calibration log**
**Soil and Sediment Sample Log Sheet**
**Surface Water Sample Log Sheet**

## 3.0 PROCEDURES

This section describes custody and documentation procedures. All entries made into the logbooks, custody documents, logs, and log sheets described in this SOP must be made in indelible ink (black is preferred). No erasures are permitted. If an incorrect entry is made, the entry will be crossed out with a single strike mark, initialed, and dated.

## 3.1 Site Logbook

The site logbook is a hard-bound, paginated, controlled-distribution record book in which all major on-site activities are documented. At a minimum, the following activities and events will be recorded (daily) in the site logbook:

- All field personnel present
- Arrival/departure of site visitors
- Arrival/departure of equipment
- Start or completion of sampling activities
- Daily on-site activities performed each day
- Sample pickup information
- Health and safety issues
- Weather conditions

The site logbook is initiated at the start of the first on-site activity (e.g., site visit or initial reconnaissance survey). Entries are to be made for every day that on-site activities take place.

The following information must be recorded on the cover of each site logbook:

- Project name
- Project number
- Book number
- Start date
- End date

Information recorded daily in the site logbook need not be duplicated in other field notebooks but must summarize the contents of these other notebooks and refer to specific page locations in these notebooks for detailed information (where applicable). At the completion of each day's entries, the site logbook must be signed and dated by the field operations leader (FOL).

## 3.2 Field Logbooks

The field logbook is a separate dedicated notebook used by field personnel to document his or her activities in the field. This notebook is hardbound and paginated.

## 3.3 Sample Labels

Adhesive sample container labels must be completed and applied to every sample container. Information on the label includes the project name, location, sample number, date, time, preservative, analysis, matrix, sampler's initials, and the name of the laboratory performing the analysis.

## 3.4 Chain-of-Custody Form

The Chain-of-Custody Form (COC) is a multi-part form that is initiated as samples are acquired and accompanies a sample (or group of samples) as it is transferred from person to person. Each COC is numbered. This form must accompany any samples collected for laboratory chemical analysis. A copy of a blank COC form is attached at the end of this SOP.

The FOL must include the name of the laboratory in the "Remarks" section to ensure that the samples are forwarded to the correct location. If more than one COC is necessary for any cooler, the FOL will indicate "Page __ of __" on each COC. The original (top) signed copy of the COC will be placed inside a sealable polyethylene bag and taped inside the lid of the shipping cooler. Once the samples are received at the laboratory, the sample custodian checks the contents of the cooler(s) against the enclosed COC(s). Any problems are noted on the enclosed COC Form (bottle breakage, discrepancies between the sample labels, COC form, etc.) and will be resolved through communication between the laboratory point-of-contact and the Task Order Manager (TOM). The COC form is signed and retained by the laboratory and becomes part of the sample's corresponding analytical data package.

## 3.5 Custody Seal

The custody seal is an adhesive-backed label, and it is part of the 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 transit to the laboratory. The custody seals are signed and dated by the samplers and affixed across the opening edges of each cooler (two seals per cooler) containing environmental samples. The laboratory sample custodian will examine the custody seal for evidence of tampering and will notify the Tetra Tech TOM if evidence of tampering is observed.

3.6 Equipment Calibration Log

The Equipment Calibration Log is used to document calibration of measuring equipment used in the field. The Equipment Calibration Log 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 requiring calibration. Entries must be made for each day the equipment is used.

3.7 Sample Log Sheets

The Soil and Sediment Sample Log Sheets are used to document the sampling of soils and sediments (see SOPs-05, 06, and -08). The surface water sample log sheets are used to document the sampling of surface waters (see SOP-07).

## 4.0 ATTACHMENTS

1. Chain-of-Custody Record
2. Equipment Calibration Log
3. Soil and Sediment Sample Log
4. Surface water Sample Log

# ATTACHMENT 1
## CHAIN-OF-CUSTODY RECORD

CHAIN OF CUSTODY | NUMBER | PAGE ____ OF _____

| PROJECT NO: | SITE NAME: | PROJECT MANAGER AND PHONE NUMBER | LABORATORY NAME AND CONTACT: |
|---|---|---|---|
| SAMPLERS (SIGNATURE) | | FIELD OPERATIONS LEADER AND PHONE NUMBER | ADDRESS |
| | | CARRIER/WAYBILL NUMBER | CITY, STATE |

STANDARD TAT ☐
RUSH TAT ☐
☐ 24 hr. ☐ 48 hr. ☐ 72 hr. ☐ 7 day ☐ 14 day

CONTAINER TYPE
PLASTIC (P) or GLASS (G)

PRESERVATIVE
USED

| DATE YEAR ____ | TIME | SAMPLE ID | MATRIX | GRAB (G) COMP (C) | No. OF CONTAINERS | TYPE OF ANALYSIS | | | | | | | | COMMENTS |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | |

| | DATE | TIME | | DATE | TIME |
|---|---|---|---|---|---|
| 1. RELINQUISHED BY | | | 1. RECEIVED BY | | |
| 2. RELINQUISHED BY | | | 2. RECEIVED BY | | |
| 3. RELINQUISHED BY | | | 3. RECEIVED BY | | |

COMMENTS

DISTRIBUTION: WHITE (ACCOMPANIES SAMPLE) YELLOW (FIELD COPY) PINK (FILE COPY)

3/99
FORM NO. TtNUS-001

Tetra Tech NUS, Inc.

# EQUIPMENT CALIBRATION LOG

PROJECT NAME : ____________________ INSTRUMENT NAME/MODEL: ____________________

SITE NAME: ____________________ MANUFACTURER: ____________________

PROJECT No.: ____________________ SERIAL NUMBER: ____________________

| Date of Calibration | Instrument I.D. Number | Person Performing Calibration | Instrument Settings | | Instrument Readings | | Calibration Standard (Lot No.) | Remarks and Comments |
|---|---|---|---|---|---|---|---|---|
| | | | Pre-calibration | Post-calibration | Pre-calibration | Post-calibration | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |

# ATTACHMENT 3
## SOIL AND SEDIMENT SAMPLE LOG SHEET

### SOIL & SEDIMENT SAMPLE LOG SHEET

Page___ of ___

Project Site Name: _____
Project No.: _____

- ▯ Surface Soil
- ▯ Subsurface Soil
- ▯ Sediment
- ▯ Other: _____
- ▯ QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____

Type of Sample:
- ▯ Low Concentration
- ▯ High Concentration

**GRAB SAMPLE DATA:**

| Date: | Depth Interval | Color | Description (Sand, Silt, Clay, Moisture, etc.) |
|---|---|---|---|
| Time: | | | |
| Method: | | | |
| Monitor Reading (ppm): | | | |

**COMPOSITE SAMPLE DATA:**

| Date: | Time | Depth Interval | Color | Description (Sand, Silt, Clay, Moisture, etc.) |
|---|---|---|---|---|
| Method: | | | | |
| Monitor Readings (Range in ppm): | | | | |

**SAMPLE COLLECTION INFORMATION:**

| Analysis | Container Requirements | Collected | Other |
|---|---|---|---|
| | | | |

**OBSERVATIONS / NOTES:**

**MAP:**

**Circle if Applicable:**

| MS/MSD | Duplicate ID No.: |
|---|---|

**Signature(s):**

# ATTACHMENT 4

# SURFACE WATER SAMPLE LOG SHEET

Tetra Tech NUS, Inc.

## SURFACE WATER SAMPLE LOG SHEET

Page___ of ___

Project Site Name: _____
Project No.: _____

[] Stream
[] Spring
[] Pond
[] Lake
[] Other: _____
[] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____

Type of Sample:
[] Low Concentration
[] High Concentration

**SAMPLING DATA:**

| | Color | pH | S.C. | Temp. | Turbidity | DO | Salinity | ORP |
|---|---|---|---|---|---|---|---|---|
| Date: | Visual | Standard | mS/cm | Degrees C | NTU | mg/l | % | mV |
| Time: | | | | | | | | |
| Depth: | | | | | | | | |
| Method: | | | | | | | | |

**SAMPLE COLLECTION INFORMATION:**

| Analysis | Preservative | Container Requirements | Collected |
|---|---|---|---|
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |

**OBSERVATIONS / NOTES:**

**MAP:**

**Circle if Applicable:**

| MS/MSD | Duplicate ID No.: |
|---|---|

**Signature(s):**

# STANDARD OPERATING PROCEDURE

# SOP-04

## DECONTAMINATION OF FIELD SAMPLING EQUIPMENT

### 1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedures to be followed when decontaminating non-dedicated field sampling equipment during the field investigations.

### 2.0 REQUIRED FIELD FORMS AND EQUIPMENT

**Writing utensil (preferably black pen with indelible ink)**
**Non-latex rubber or plastic gloves**
**Cotton gloves**
**Field logbook**
**Potable water**
**Deionized water**
**LiquiNox detergent**
**Brushes, spray bottles, paper towels, etc.**
**Container to collect and transport decontamination fluids**

### 3.0 DECONTAMINATION PROCEDURES

3.1 Don nitrile and/or cotton gloves and decontaminate sampling equipment (in accordance with the following steps) prior to field sampling and between samples.

3.2 Rinse the equipment with potable water. Rinsing may be conducted by spraying with water from a spray bottle or by dipping. Collect the potable water rinsate into a container.

3.3 Wash the equipment with a solution of LiquiNox detergent. Prepare the LiquiNox wash solution in accordance with the instructions on the LiquiNox container. Collect the LiquiNox wash solution into a container. Use brushes or sprays as appropriate for the equipment. If oily residue has accumulated on the sampling equipment, remove the residue with an isopropanol wash and repeat the Liquinox wash.

3.4 Rinse the equipment with potable water. Rinsing may be conducted by spraying with water from a spray bottle or by dipping. Collect the potable water rinsate into a container.

3.5 Rinse the equipment with deionized water. Rinsing may be conducted by spraying with water from a spray bottle or by dipping. Collect the deionized water rinsate into a container.

3.6 Remove excess water by air drying, shaking, or by wiping with paper towels as necessary.

3.7 Document decontamination by recording it in the field logbook.

3.8 Containerized decontamination solutions will be managed in accordance with the procedures described in SOP-09 and this UFP SAP.

# STANDARD OPERATING PROCEDURE

# SOP-05

# SOIL CORING AND SAMPLING USING HAND AUGER TECHNIQUES

## 1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the procedures for collecting surface and subsurface soil cores from unconsolidated overburden materials using hand augering techniques.

## 2.0 REQUIRED FIELD FORMS AND EQUIPMENT

**Disposable medical-grade gloves (e.g., latex, nitrile)**

**Writing utensil (preferably black pen with indelible ink)**

**Indelible marker**

**Stainless Steel Auger Buckets**

**Stainless Steel Extension Rods**

**Cross Handle**

**Required decontamination materials**

**Bentonite pellets**

**Sealable polyethylene bags**

**Sample labels**

**Shipping containers** (containing ice)

**Disposable plastic trowels or stainless steel trowels**

**Stainless steel mixing bowls**

**Sample containers:** Sample containers are certified clean by the laboratory supplying the containers.

**Soil Sample Log Forms**

**Daily Activity Logs**

**Chain-of-Custody Form**

**Soil Boring Log**

## 3.0 BOREHOLE ADVANCEMENT AND SOIL SAMPLING USING A HAND AUGER

Hand Augers may be employed to collect the soil cores. A hand augering system generally consists of a variety of all stainless steel bucket bits (i.e. cylinders 6-1/2" long and 2-3/4", 3-1/4", and 4" in diameter), a series of extension rods (available in 2', 3', 4' and 5' lengths), a cross handle.

3.1 The hand auger can be used in a wide variety of soil conditions. It can be used to sample soil, both from the surface, or to depths in excess of 12 feet. However, the presence or rock layers and the collapse of the borehole normally contribute to its limiting factors.

Attach a properly decontaminated bucket bit into a clean extension rod and further attach the cross handle to the extension rod.

3.2 Clear the area to be sampled of any surface debris (vegetation, twigs, rocks, letter, etc.)

3.3. Turn the hand auger sampler into the ground to a depth of 1 foot. The 0- to 1-foot depth soil interval is considered to be the surface soil. Subsurface soil samples will be collected at depths greater than 1 foot below ground surface.

3.4 After reaching the desired depth, slowly and carefully withdraw the apparatus from the borehole.

3.4 Utilizing a properly decontaminated stainless steel trowel or disposable trowel, remove the sample material from the bucket bit and place into a sealable polyethylene bag. Note in a field notebook or on a standardized data sheet any changes in the color, texture or odor of the soil.

3.5 Thoroughly homogenize the sample material and write sample ID, date, and time on the bag with an indelible marker.

3.6 Complete required information on the Soil Sample Log Sheet (copy attached at the end of this SOP). Update the Chain-of-Custody (COC) Form.

3.7 Excess soil core materials will be returned to the hole and tamped. If insufficient soil is available to fill the hole to the ground surface, then bentonite pellets mixed with the soil will be used to backfill the hole.

3.8 Decontaminate all soil sampling equipment in accordance with SOP-04 before collecting the next sample.

3.9 Soil samples will be transported to the field office where a portion of the sample will undergo XRF analysis for lead (See SOP-14).

3.10 For soil samples selected for fixed-base laboratory analysis, a portion of the sample will be used to fill the required sample containers as supplied by the laboratory. The sample labels will be completed and affixed to the sample container. The samples will then be packaged and shipped to the fixed-base laboratory in accordance with SOP-11.

## 4.0 ATTACHMENTS

1. Soil and Sediment Sample Log Sheet

# ATTACHMENT 1
# SOIL AND SEDIMENT SAMPLE LOG SHEET

## SOIL & SEDIMENT SAMPLE LOG SHEET

Page___ of ___

Project Site Name: _____
Project No.: _____

[] Surface Soil
[] Subsurface Soil
[] Sediment
[] Other: _____
[] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____

Type of Sample:
[] Low Concentration
[] High Concentration

**GRAB SAMPLE DATA:**

| Date: | Depth Interval | Color | Description (Sand, Silt, Clay, Moisture, etc.) |
|---|---|---|---|
| Time: | | | |
| Method: | | | |
| Monitor Reading (ppm): | | | |

**COMPOSITE SAMPLE DATA:**

| Date: | Time | Depth Interval | Color | Description (Sand, Silt, Clay, Moisture, etc.) |
|---|---|---|---|---|
| | | | | |
| Method: | | | | |
| | | | | |
| Monitor Readings (Range in ppm): | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |

**SAMPLE COLLECTION INFORMATION:**

| Analysis | Container Requirements | Collected | Other |
|---|---|---|---|
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |

**OBSERVATIONS / NOTES:**

**MAP:**

**Circle if Applicable:**

| MS/MSD | Duplicate ID No.: |
|---|---|

**Signature(s):**

# STANDARD OPERATING PROCEDURE
# SOP-06

## BOREHOLE ADVANCEMENT AND SOIL CORING FOR SOIL SAMPLING USING DIRECT-PUSH TECHNOLOGY

### 1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the procedures for collecting surface and subsurface soil cores from unconsolidated overburden materials using direct-push technology (DPT). For these investigations, a Geoprobe® rig with a Macrocore Sampler will be the type of DPT used.

### 2.0 REQUIRED FIELD FORMS AND EQUIPMENT

**Cut-resistant non-latex impermeable gloves**
**Cotton gloves**
**Disposable medical-grade gloves (e.g., latex, nitrile)**
**Writing utensil (preferably black pen with indelible ink)**
**Indelible marker**
**Boring log sheets:** A copy of this form is included in SOP-10
**Photoionization detector (PID)**
**Geoprobe® or equivalent DPT equipment**
**Geoprobe® Macrocore Sampler or equivalent**
**Geoprobe® Sampling Kit or equivalent**
**Clear acetate liners:** one new liner for each soil core
**Required decontamination materials** (see SOP-04)
**Bentonite pellets**

### 3.0 BOREHOLE ADVANCEMENT AND SOIL SAMPLING USING A GEOPROBE®

Direct-push technology (DPT) will be employed to collect soil cores. DPT refers to sampling tools and sensors that are driven directly into the ground without the use of conventional rotary drilling equipment. DPT typically utilizes hydraulic pressure and/or percussion hammers to advance the sampling tools. Geoprobe® is a manufacturer of a hydraulically powered, percussion/probing machine utilizing DPT to

collect subsurface environmental samples. This type of rig with a Macrocore Sampler will be used at the Naval Support Indian Head Stump Neck Annex to collect soil cores.

3.1 Clear the area to be sampled of any surface debris (herbaceous vegetation, twigs, rocks, litter, etc.).

3.2 Place a new clear acetate liner in the detachable Macrocore core barrel and attach coring device to the Geoprobe® rig.

3.3 Drive macrocore sampler (lined with acetate) into the ground to a specified depth using hydraulic pressure. The 0- to 1-foot depth soil interval is considered to be the surface soil. Subsurface soil samples will be collected at depths greater than 1 foot below ground surface.

3.4 Retract the sampler from the borehole and remove the acetate liner and the soil core from the Macrocore barrel.

3.5 Attach the metal trough from the Geoprobe® Sampling Kit firmly to a suitable surface.

3.6 Place the acetate liner containing the soil core in the trough.

3.7 While wearing cut-resistant gloves, cut the acetate liner through its entire length using the double-bladed knife that accompanies the Geoprobe® Sampling Kit. Then remove the strip of acetate from the trough to gain access to the collected soils. **CAUTION**: Do not attempt to cut the acetate liner while holding it in your hand.

3.8 Scan the entire length of the soil core for volatile organic compounds (VOCs) using the PID. Record the specific depth interval and the associated PID reading on the Boring Log Sheet.

3.9 Log the soil core on the Boring Log Sheet (see SOP-10).

3.10 Place the soil core in a sealable polyethylene bag, thoroughly homogenize the sample material and write sample ID, date, and time on the bag with an indelible marker.

3.11 Complete required information on the Soil Sample Log Sheet (copy attached at the end of this SOP). Update the Chain-of-Custody (COC) Form.

3.12 Repeat steps 3.2 through 3.11 for the next depth intervals, if required.

3.13 The depth to bedrock should be recorded on the Boring Log, and the estimated moisture content of the soil and the presence or absence of water in the boring should be noted.

3.14 If readings from the PID are all at background levels below field screening criteria, then excess soil core materials will be returned to the hole and tamped. If insufficient soil is available to fill the hole to the ground surface, then bentonite pellets mixed with the soil will be used to backfill the hole.

3.15 If screening instruments indicate that contaminants may be present in the soil materials, then all excess soil core materials will be placed in a plastic bag (or drum if larger quantities). The bag will be tagged identifying the location and depths from where the soils came and the date. The bag will then be placed in a 55-gallon drum and stored on-site until laboratory analyses of the soil are completed and classification of the soil waste materials can be determined (see SOP-09).

3.16 If soil materials from the boring are suspected of being contaminated (see 3.15 above), the soil boring will be backfilled with bentonite pellets up to the ground surface.

3.17 Decontaminate all soil sampling equipment in accordance with SOP-04 prior to collecting the next sample.

3.18 Soil samples will be transported to the field office where a portion of the sample will undergo XRF analysis for lead (See SOP-14).

3.19 For soil samples selected for fixed-base laboratory analysis, a portion of the sample will be used to fill the required sample containers as supplied by the laboratory. The sample labels will be completed and affixed to the sample container. The samples will then be packaged and shipped to the fixed-base laboratory in accordance with SOP-11.

# STANDARD OPERATING PROCEDURE

# SOP-07

# SURFACE WATER SAMPLING

## 1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedure for collecting surface water samples.

## 2.0 REQUIRED FIELD FORMS AND EQUIPMENT

**Surface Water Sample Log Sheet**: A copy of this form is attached at the end of this SOP.

**Field logbook**

**Writing utensil (preferably black pen with indelible ink)**

**Indelible marker**

**Multi-parameter water-quality meter**: The water-quality meter is used for the measurement of dissolved oxygen, pH, specific conductance, temperature, oxidation-reduction potential, and turbidity (see SOP CTO331-14).

**LaMotte Turbidity Meter**: Used to measure turbidity in the field.

**Disposable sample containers**: Disposable sample containers are used to fill sample containers and transport sample(s) to a pump for filtering.

**Sample containers**: Certified-clean sample containers will be provided by the laboratory that performs the analyses.

**0.45-micron filter assembly**: These are single-use filter cartridges used to filter samples scheduled for dissolved metals or dissolved thorium isotope analyses. The filters become investigation-derived waste (IDW) after one use.

**Peristaltic pump**

**Silicon tubing**

**Sealable polyethylene bags**

**Shipping containers (coolers)**

## 3.0 SURFACE WATER SAMPLING PROCEDURES

3.1 Sampling will start at the downstream end of a stream and proceed to the farthest upstream location.

3.2 While standing downstream or from the bank, gently remove any floating leaves or twigs that may be present in a sample pool area in a manner that will not disturb the bottom sediment.

3.3 While standing downstream or from the bank, place the sample container in the water at the sampling location at a 45-degree angle and lower it to approximately half the sample pool depth. With the mouth of the container facing upstream, fill the container with water, being careful not to disturb the sediment.

3.4 All samples will be collected into certified-clean, pre-preserved bottles (if preservation is required for the analysis to be performed) supplied by the laboratory performing the analyses. Sample containers should <u>not</u> be filled completely; a small amount of air should be left at the top.

3.5 Record the date and time that the sample containers are filled on the Surface Water Sample Log Sheet, the sample labels, and the Chain-of-Custody Form.

3.6 After the sample label is completed and checked, place the sample container into a sealable polyethylene bag and place the plastic storage bag holding the sample container into a cooler containing ice.

3.7 Repeat steps 3.3 through 3.6 until all the sample bottles containing unfiltered samples have been filled.

3.8 Fill two 1-liter unpreserved polyethylene bottles. Use these bottles to transfer the sample for field filtering. Set up a peristaltic pump for filtering of the dissolved metals samples. Using new, clean, disposable silicone tubing and a 0.45-micron filter, place the intake tubing from the pump into the transfer bottle with the filter attached to the discharge end and start the pump. Pre-rinse the filter with approximately 50-mL of sample water prior to filling the sample containers.

3.9 Using the discharge from the filter cartridge, fill one 1-liter polyethylene sample bottle for dissolved metals. Repeat steps 3.8 and 3.9 for these sample containers.

3.10 Obtain measurements of dissolved oxygen, pH, specific conductance, temperature, turbidity, and oxidation-reduction potential using the multi-parameter water-quality meter and LaMotte Turbidity Meter (see SOP-12). Record the readings in the appropriate fields on the Surface Water Sample Log Sheet.

3.11 Estimate the flow rate of the stream or spring in accordance with SOP-15. This is an estimate only. Round the flow rate to the nearest 5 gallons and record this number on the Surface Water Sample Log Sheet.

3.12 Decontaminate all equipment and load the equipment and the sample cooler in the sample vehicle for transport.

## 4.0 ATTACHMENTS

1. Surface Water Sample Log Sheet

# ATTACHMENT 1

## SURFACE WATER SAMPLE LOG SHEET

Tetra Tech NUS, Inc. **SURFACE WATER SAMPLE LOG SHEET**

Page ___ of ___

Project Site Name: _____
Project No.: _____

[] Stream
[] Spring
[] Pond
[] Lake
[] Other: _____
[] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____

Type of Sample:
[] Low Concentration
[] High Concentration

**SAMPLING DATA:**

| Date: / Time: | Color Visual | pH Standard | S.C. mS/cm | Temp. Degrees C | Turbidity NTU | DO mg/l | Salinity % | ORP mV |
|---|---|---|---|---|---|---|---|---|
| Depth: / Method: | | | | | | | | |

**SAMPLE COLLECTION INFORMATION:**

| Analysis | Preservative | Container Requirements | Collected |
|---|---|---|---|
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |

**OBSERVATIONS / NOTES:**

**MAP:**

**Circle if Applicable:**

| MS/MSD | Duplicate ID No.: |
|---|---|

**Signature(s):**

# STANDARD OPERATING PROCEDURE

# SOP-08

# SEDIMENT SAMPLING

## 1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedure for sediment sampling in streams and other waterways.

## 2.0 REQUIRED FIELD FORMS AND EQUIPMENT

The following field forms and equipment are required for sediment sampling.

**Sediment Sample Log Forms**: A copy of this form is attached at the end of this SOP.
**Writing utensil (preferably black pen with indelible ink)**
**Indelible marker**
**Bound field logbook**
**Disposable plastic trowels**
**Survey stakes and flagging:** Used to mark sampling locations after completion of sampling.
**Labeled sample containers**: See SOP-01 for sample identification procedures. Sample containers are certified clean by the laboratory supplying the containers.
**Sealable polyethylene bags**
**Shipping containers** (containing ice)
**Disposable medical-grade gloves (e.g., latex, nitrile)**
**Chain-of-Custody Form**

## 3.0 SEDIMENT SAMPLE LOCATION SELECTION

In general, sediments composed of fine-grained materials with greater surface area available for adsorption are more desirable for sample selection. The fined-grained materials may act as a sink or reservoir for adsorbing heavy metals and organic contaminants even if surface runoff concentrations are below detection limits. Therefore, it is important to locate the specific sampling points where the sediment has the greatest percentage of fine particles. The sampling personnel will determine specific sampling locations with these goals in mind.

## 4.0 SEDIMENT SAMPLING PROCEDURES

4.1 The sampler will wear clean, disposable medical-grade gloves. Clear vegetative matter or debris, if present, from the sample location using a disposable sampling trowel or spoon. Use the trowel to dig up and homogenize the sediment in an 18-inch-diameter circular area that is 6 inches deep. Stir the sediment within the circular area; do not move the sediment outside the circle. Also, do not dig or stir sediment that is deeper than 6 inches below the ground surface, until the next depth interval is sampled.

- Use the same trowel to scoop the homogenized sediment into the requisite labeled sample container(s).

4.2 Record the sample time (using military time) on the Sediment Sample Log Form and sample container labels. Record all other information required on the labels as specified by SOP-01.

4.3 Place the labeled sample container into a sealable polyethylene bag and then place the bag holding the sample container into a cooler containing ice.

4.4 Record date, sampling site, site conditions, location map, and other information (e.g., presence and flow rate of water in channel) on the Sediment Collection Log Sheet. Enter the sample information onto the Chain-of-Custody Form in accordance with SOP-03.

4.5 Using an indelible marker, write the sample identification on a survey stake, and drive the stake into the ground at the sample location. Tack a piece of brightly colored flagging to the stake. In addition, tie a piece of flagging to an overhead tree branch or other eye-level object to improve the ability to relocate the sampling site in the future.

## 5.0 ATTACHMENTS

1. Soil and Sediment Sample Log Sheet

# ATTACHMENT 1
## SOIL AND SEDIMENT SAMPLE LOG SHEET

Tetra Tech NUS, Inc. **SOIL & SEDIMENT SAMPLE LOG SHEET**

Page___ of ___

Project Site Name: _____
Project No.: _____

[] Surface Soil
[] Subsurface Soil
[] Sediment
[] Other: _____
[] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____

Type of Sample:
[] Low Concentration
[] High Concentration

**GRAB SAMPLE DATA:**

| Date: | Depth Interval | Color | Description (Sand, Silt, Clay, Moisture, etc.) |
|---|---|---|---|
| Time: | | | |
| Method: | | | |
| Monitor Reading (ppm): | | | |

**COMPOSITE SAMPLE DATA:**

| Date: | Time | Depth Interval | Color | Description (Sand, Silt, Clay, Moisture, etc.) |
|---|---|---|---|---|
| | | | | |
| Method: | | | | |
| | | | | |
| Monitor Readings | | | | |
| (Range in ppm): | | | | |
| | | | | |
| | | | | |
| | | | | |

**SAMPLE COLLECTION INFORMATION:**

| Analysis | Container Requirements | Collected | Other |
|---|---|---|---|
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |

**OBSERVATIONS / NOTES:**

**MAP:**

**Circle If Applicable:**

| MS/MSD | Duplicate ID No.: |
|---|---|
| | |

**Signature(s):**

# STANDARD OPERATING PROCEDURE
# SOP-09

## MANAGEMENT OF INVESTIGATION-DERIVED WASTE

### 1.0 PURPOSE

This Standard Operating Procedure (SOP) describes how investigation-derived waste (IDW) will be collected, segregated, classified, and managed during the field investigations at the NAS Corpus Christi, Texas facility. The following types of IDW will be generated during this investigation:

- Decontamination solutions
- Personal protective equipment and clothing (PPE)
- Miscellaneous trash and incidental items

### 2.0 REQUIRED FIELD FORMS AND EQUIPMENT

**Health and safety equipment (with PPE)**
**Decontamination equipment**
**Field logbook**
**Writing utensil (preferably black pen with indelible ink)**
**Plastic sheeting and/or tarps**
**55-gallon drums with sealable lids**
**IDW labels for drums**
**Wastewater container tanks**
**Plastic garbage bags**

### 3.0 PROCEDURES

Management of IDW includes the collection, segregation, temporary storage, classification, final disposal, and documentation of the waste-handling activities if necessary.

### 3.1 Liquid Wastes

Liquid wastes that will be generated during the site activities include decontamination solutions from sampling equipment. These wastes will be collected and transported to a central location at NAS Mid-South.

### 3.2 Solid Wastes

Solid wastes that may be generated during the site activities include collection of lead shot from surface soil samples. This waste will be containerized and handed over to the NAS Mid-South Environmental Department Manager at the conclusion of field activities.

### 3.3 PPE and Incidental Trash

All PPE wastes and incidental trash materials (e.g., wrapping or packing materials from supply cartons, waste paper) will be decontaminated (if contaminated), double bagged, securely tied shut, and placed in a designated waste receptacle at NAS Mid-South.

# STANDARD OPERATING PROCEDURE

# SOP-10

# BOREHOLE AND SOIL SAMPLE LOGGING

## 1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the standard procedures and technical guidance on the logging of soil cores.

## 2.0 FIELD FORMS AND EQUIPMENT

**Knife**
**Ruler** (marked in tenths and hundredths of feet)
**Boring Log:** An example of this form is attached.
**Photoionization detector (PID)**
**Writing utensil (preferably black pen with indelible ink)**

## 3.0 RESPONSIBILITIES

A field geologist or engineer is responsible for supervising all boring activities and assuring that each borehole is properly and completely logged.

## 4.0 PROCEDURES FOR BOREHOLE AND SAMPLE LOGGING

To maintain a consistent classification of soil, it is imperative that the field geologist understands and accurately uses the field classification system described in this SOP. This identification is based on visual examination and manual tests.

### 4.1 <u>USCS Classification</u>

Soils are to be classified according to the Unified Soil Classification System (USCS). This method of classification is detailed in Figure 1 (attached to this SOP).

This method of classification identifies soil types on the basis of grain size and cohesiveness.

Fine-grained soils, or fines, are smaller than the No. 200 sieve and are of two types: silt (M) and clay (C). Some classification systems define size ranges for these soil particles, but for field classification purposes, they are identified by their respective behaviors. Organic material (O) is a common component of soil but has no distinguishable size range; it is recognized by its composition. The careful study of the USCS will aid in developing the competence and consistency necessary for the classification of soils.

Coarse-grained soils will be divided into categories: rock fragments, sand, or gravel. The terms "sand" and "gravel" not only refer to the size of the soil particles but also to their depositional history. To insure accuracy in description, the term "rock fragments" will be used to indicate angular granular materials resulting from the breakup of rock. The sharp edges that are typically observed indicate little or no transport from their source area; and therefore, the term provides additional information in reconstructing the depositional environment of the soils encountered. When the term "rock fragments" is used, it will be followed by a size designation such as "(1/4 inchΦ-1/2 inchΦ)" or "coarse-sand size" either immediately after the entry or in the remarks column. The USCS classification would not be affected by this variation in terms.

## 4.2 Color

Soil colors will be described utilizing a single color descriptor preceded, when necessary, by a modifier to denote variations in shade or color mixtures. A soil could therefore be referred to as "gray" or "light gray" or "blue-gray." Because color can be utilized in correlating units between sampling locations, it is important for color descriptions to be consistent from one boring to another.

Colors must be described while the sample is still moist. Soil samples will be broken or split vertically to describe colors. Samplers tend to smear the sample surface, creating color variations between the sample interior and exterior.

The term "mottled" will be used to indicate soils irregularly marked with spots of different colors. Mottling in soils usually indicates poor aeration and lack of good drainage.

## 4.3 Relative Density and Consistency

To classify the relative density and/or consistency of a soil, the geologist is to first identify the soil type. Granular soils contain predominantly sands and gravels. They are non-cohesive (particles do not adhere well when compressed). Finer-grained soils (silts and clays) are cohesive (particles will adhere together when compressed).

Granular soils are given the USCS classifications GW, GP, GM, SW, SP, SM, GC, or SC (see Figure 1).

The consistency of cohesive soils is determined by performing field tests and identifying the consistency as shown in the following table.

**CONSISTENCY FOR COHESIVE SOILS**

| Consistency | Standard Penetration Resistance (Blows per Foot) | Unconfined Compressive Strength (Tons/Sq. Foot by pocket penetration) | Field Identification |
|---|---|---|---|
| Very soft | 0 to 2 | Less than 0.25 | Easily penetrated several inches by fist. |
| Soft | 2 to 4 | 0.25 to 0.50 | Easily penetrated several inches by thumb. |
| Medium stiff | 4 to 8 | 0.50 to 1.0 | Can be penetrated several inches by thumb with moderate effort. |
| Stiff | 8 to 15 | 1.0 to 2.0 | Readily indented by thumb but penetrated only with great effort. |
| Very stiff | 15 to 30 | 2.0 to 4.0 | Readily indented by thumbnail. |
| Hard | Over 30 | More than 4.0 | Indented with difficulty by thumbnail. |

Cohesive soils are given the USCS classifications ML, MH, CL, CH, OL, or OH (see Figure 1).

The consistency of cohesive soils is determined by hand by determining the resistance to penetration by the thumb. The thumb determination methods are conducted on a selected sample of the soil, preferably the lowest 0.5 foot of the sample. The sample will be broken in half and the thumb pushed into the end of the sample to determine the consistency. Do not determine consistency by attempting to penetrate a rock fragment. If the sample is decomposed rock, it is classified as a soft decomposed rock rather than a hard soil. One of the other methods will be used in conjunction with it. The designations used to describe the consistency of cohesive soils are shown in the above-listed table.

## 4.4 Weight Percentages

In nature, soils are consist of particles of varying size and shape and are combinations of the various grain types. The following terms are useful in the description of soil:

| Terms of Identifying Proportion of the Component | Defining Range of Percentages by Weight |
|---|---|
| Trace | 0 - 10 percent |
| Some | 11 - 30 percent |
| Adjective form of the soil type (e.g., sandy) | 31 - 50 percent |

Examples:

- Silty fine sand: 50 to 69 percent fine sand, 31 to 50 percent silt.
- Medium to coarse sand, some silt: 70 to 80 percent medium to coarse sand, 11 to 30 percent silt.
- Fine sandy silt, trace clay: 50 to 68 percent silt, 31 to 49 percent fine sand, 1 to 10 percent clay.
- Clayey silt, some coarse sand: 70 to 89 percent clayey silt, 11 to 30 percent coarse sand.

## 4.5 Moisture

Moisture content is estimated in the field according to four categories: dry, moist, wet, and saturated. In dry soil, there appears to be little or no water. Saturated samples obviously have all the water they can hold. Moist and wet classifications are somewhat subjective and often are determined by the individual's judgment. A suggested parameter for this would be calling a soil wet if rolling it in the gloved hand or on a porous surface liberates water (i.e., dirties or muddies the surface). Whatever method is adopted for describing moisture, it is important that the method used by an individual remains consistent throughout an entire field activity.

## 4.6 Classification of Soil Grain Size for Chemical Analysis

To determine the gross grain size classification (e.g., clay, silt, and sand) from the USCS classification described above, the following table will be used.

| Gross Soil Grain Size Classification | USCS Abbreviation | Description |
|---|---|---|
| Clay | CL | inorganic clays of low to medium plasticity, gravelly clays, sandy clays, silty clays, lean clays,. |
| | CH | inorganic clays of high plasticity, fat clays. |
| | OH | organic clays of medium to high plasticity, organic silts. |
| Silt | ML | inorganic silts and very fine sands, rock four, silty or clayey fine sands with slight plasticity. |
| | OL | organic silts and organic silty clays of low plasticity.. |
| | MH | inorganic silts, micaceous or diatomaceous fine sand or silty soils. |
| Sand | SW | well graded sands, gravelly sands, little or no fines. |

| Gross Soil Grain Size Classification | USCS Abbreviation | Description |
|---|---|---|
| | SP | poorly graded sands, gravelly sands, little or no fines. |
| | SM | silty sands, sand-silt mixtures. |
| | SC | clayey sands, sand-clay mixtures. |

## 4.7 Summary of Soil Classification

In summary, soils will be classified in a similar manner by each geologist/engineer at a project site. The hierarchy of classification is as follows:

- Density and/or consistency
- Color
- Plasticity (optional)
- Soil types
- Moisture content
- Other distinguishing features
- Grain size
- Depositional environment

## 4.0 ATTACHMENTS

1. Figure 1 - Unified Soil Classification System
2. Boring Log

## ATTACHMENT 1

## FIGURE 1 - UNIFIED SOIL CLASSIFICATION SYSTEM

### Unified Soil Classification System

| | | | |
|---|---|---|---|
| Coarse Grained Soils (more than half of soil > No. 200 sieve) | Gravels (More than half of coarse fraction > no. 4 sieve size) | GW | Well graded gravels or gravel-sand mixtures, little or no fines |
| | | GP | Poorly graded gravels or gravel-sand mixtures, little or no fines |
| | | GM | Sandy gravels, gravel-sand-silt mixtures |
| | | GC | Clayey gravels, gravel-sand-silt mixtures |
| | Sands (More than half of coarse fraction < no. 4 sieve size) | SW | Well graded sands or gravelly sands, little or no fines |
| | | SP | Poorly graded sands or gravelly sands, little or no fines. |
| | | SM | Silty sands, sand-silt mixtures |
| | | SC | Inorganic silts and very fine sands, rock flour, silty or clayey fine sands or clayey silts with slight plasticity |
| Fine Grained Soils (more than half of soil < No. 200 sieve) | Silts and Clays LL = < 50 | ML | Inorganic silts and very fine sands, rock flour, silty fine sands or clayey silts with slight plasticity |
| | | CL | Inorganic clays of low to medium plasticity, gravelly clays, sandy clays, lean clays |
| | | OL | Organic silts and organic silty clays of low plasticity |
| | Silts and Clays LL = > 50 | MH | Inorganic silts, micaceous or diatomaceous fine sand or silty soils, elastic silts |
| | | CH | Inorganic silts of high plasticity, fat clays |
| | | OH | Organic clays of high plasticity, organic silty clays, organic silts |
| Highly Organic Soils | | Pt | Peat and other highly organic soils |

### Grain Size Chart

| Classification | Range of Grain Sizes | |
|---|---|---|
| | U.S. Standard Sieve Size | Grain Size In Millimeters |
| Boulders | Above 12" | Above 305 |
| Cobbles | 12" to 3" | 305 to 76.2 |
| Gravel | 3" to No. 4 | 76.2 to 7.76 |
| coarse | 3" to 3/4" | 76.2 to 4.76 |
| fine | 3/4"to No.4 | 19.1 to 4.76 |
| Sand | No. 4 to No. 200 | 4.76 to 0.074 |
| coarse | No.4 to No. 10 | 4.76 to 2.00 |
| medium | No. 10 to No. 40 | 2.00 to 0.420 |
| fine | No. 40 to No. 200 | 0.420 to 0.074 |
| Silt and Clay | Below No. 200 | Below 0.074 |

### Relative Density (SPT)

| SANDS AND GRAVELS | BLOWS/FOOT |
|---|---|
| VERY LOOSE | 0 – 4 |
| LOOSE | 4 – 10 |
| MEDIUM DENSE | 10 – 30 |
| DENSE | 32 – 50 |
| VERY DENSE | OVER 50 |

### Consistency (SPT)

| SILTS AND CLAYS | BLOWS/FOOT |
|---|---|
| VERY SOFT | 0 – 2 |
| SOFT | 2 – 4 |
| MEDIUM STIFF | 4 – 8 |
| STIFF | 8 – 16 |
| VERY STIFF | 16 – 22 |
| HARD | OVER 32 |

## ATTACHMENT 2
## BORING LOG

Tetra Tech NUS, Inc.

# BORING LOG

Page ___ of ___

PROJECT NAME: _____ BORING No.: _____
PROJECT NUMBER: _____ DATE: _____
DRILLING COMPANY: _____ GEOLOGIST: _____
DRILLING RIG: _____ DRILLER: _____

| Sample No. and Type or RQD | Depth (Ft.) or Run No. | Blows / 6" or RQD (%) | Sample Recovery / Sample Length | Lithology Change (Depth/Ft.) or Screened Interval | MATERIAL DESCRIPTION: Soil Density/ Consistency or Rock Hardness | Color | Material Classification | USCS* | Remarks | PID/FID Reading (ppm): Sample | Sampler BZ | Borehole** | Driller BZ** |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |

* When rock coring, enter rock brokeness.
** Include monitor reading in 6 foot intervals @ borehole. Increase reading frequency if elevated repunse read.

Drilling Area Background (ppm): ____

Remarks: _____

Converted to Well: Yes _____ No _____ Well I.D. #: _____

# STANDARD OPERATING PROCEDURE

# SOP-11

# SAMPLE PRESERVATION, PACKAGING, AND SHIPPING

## 1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the procedures for sample preservation, packaging, and shipping to be used in handling soil, sediment, and aqueous samples.

## 2.0 REQUIRED FIELD FORMS AND EQUIPMENT

**Shipping labels**

**Custody seals**

**Chain-of-custody (COC) form(s)**

**Sample containers with preservatives**: All sample containers for analysis by fixed-base laboratories will be supplied, with preservatives added (if required) and deemed certified clean by the laboratory.

**Sample shipping containers (coolers)**: All sample shipping containers are supplied by the laboratory.

**Packaging material**: Bubble wrap, sealable polyethylene bags, strapping tape, etc.

## 3.0 PROCEDURES FOR SAMPLE PRESERVATION, PACKAGING, AND SHIPPING

3.1 The laboratory provides sample containers with preservative already included (as required) for the analytical parameter for which the sample is to be analyzed. All samples will be held, stored, and shipped at 4°C. This will be accomplished through refrigeration (used to hold samples prior to shipment) and/or ice.

3.2 The sampler shall maintain custody of the samples until the samples are relinquished to another custodian or to the common carrier.

3.3 Check that each sample container is properly labeled, the container lid is securely fastened, and the container is sealed in a polyethylene bag.

3.4 If the container is glass, place the sample container into a bubble-out shipping bag and seal the bag using the self-sealing, pressure sensitive tape supplied with the bag.

3.5 Inspect the insulated shipping cooler. Check for any cracks, holes, broken handles, etc. If the cooler has a drain plug, make certain it is sealed shut, both inside and outside of the cooler. If the cooler is questionable for shipping, the cooler must be discarded.

3.6 Put ice into sealable polyethylene bags and place a layer of the sealed bags on the bottom of the cooler. Place the sample containers into the shipping cooler on top of the ice in an upright position (containers will be upright, with the exception of any 40-ml vials). Place sealable polyethylene bags of ice flat against the sides of the cooler. Continue filling the cooler with samples until the cooler is nearly full and the movement of the sample containers is limited.

3.7 Add a final layer of ice sealed in polyethylene bags to the top of the samples just before the cooler is closed and sealed.

3.8 Place the original (top) signed copy of the COC form inside a sealable polyethylene bag. Tape the bag to the inside of the lid of the shipping cooler.

3.9 Close the cooler and seal the cooler with approximately four wraps of strapping tape at each end of the cooler. Prior to wrapping the last wrap of strapping tape, apply a signed and dated custody seal to each side of the cooler (one per side). Cover the custody seal with the last wrap of tape. This will provide a tamper evident custody seal system for the sample shipment.

3.10 Affix shipping labels to each of the coolers, ensuring all of the shipping information is filled in properly. Overnight (e.g., FedEx Priority Overnight) courier services will be used for all sample shipments.

3.11 All samples will be shipped to the laboratory no more than 72 hours after collection. Under no circumstances should sample hold times be exceeded.

# STANDARD OPERATING PROCEDURE

# SOP-12

# CALIBRATION AND CARE OF WATER QUALITY METER

## 1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedures for the calibration and maintenance of field instruments used to measure water quality and for the proper documentation of calibration and maintenance. The multi-parameter water quality meter will be used to measure pH, temperature, oxidation-reduction potential (ORP), specific conductance (SC), and dissolved oxygen (DO). A LaMotte Model 2020 turbidity meter will be used to measure turbidity in water. The water meter will have a multiprobe sensor that can be used in conjunction with a flow-through cell attached to a pump discharge tube to measure water-quality parameters in a groundwater discharge or can be immersed in a surface water body such as a stream, pond, or drainage ditch.

## 2.0 FIELD FORMS AND EQUIPMENT LIST

The following logbooks, forms, equipment, and supplies are required:

**Site logbook**
**Writing utensil (preferably black pen with indelible ink)**
**Equipment calibration log sheet**
**Multi-parameter Water Quality Meter (with flow-through cell)**
**Equipment manual**
**Calibration kit**
**Deionized water, paper towels, spray bottle, etc.**
**Disposable medical-grade gloves (e.g., latex, nitrile)**

## 3.0 PROCEDURES

This section describes the general calibration procedure for the multi-parameter water quality meter. The meter will be supplied with an instruction manual. The manual will be on site and will be used as the calibration guidance document for the meter's calibration. This procedure will list requirements for frequency of calibration and checks to be performed on the meter.

The multi-parameter water-quality meter may be used to measure open water bodies (streams, ponds, springs, etc.) with the probe guard installed. With the flow through cell attached, the meter has the ability to measure water-quality parameters in groundwater via a pump discharge line. By performing the measurements in the discharge line coming directly from the well, the parameters are measured before the groundwater comes in contact with the atmosphere. The parameters to be measured by the meter for this field effort are as follows:

- DO
- SC
- Temperature
- pH
- ORP
- Turbidity (measured via turbidity meter)

## 3.1 Documentation

The Equipment Calibration Log is used to document calibration of measuring equipment used in the field. The Equipment Calibration Log documents that the manufacturer's instructions were followed for calibration of the equipment, including the frequency of calibration, type of standards used, and checks performed on calibration during the course of using the equipment. An Equipment Calibration Log must be maintained for each measuring device that requires calibration. Entries must be made for each day the equipment is used. A blank Equipment Calibration Log form is attached at the end of this SOP.

## 3.2 Calibration

All the parameters listed in Section 3.0 must be calibrated prior to the start of each field effort. After this initial calibration, the meter will be checked each day that it is used. If the check shows any out-of-specification readings, the specific probe will be recalibrated. Meter specifications can be found in the equipment manual. Calibration and calibration checks will be documented in the field logbook and on the Equipment Calibration Log. The name, lot number, and expiration date for all calibration buffers and standards used will be recorded on the Equipment Calibration Log. The meter's model, serial number, and name of rental company will also be recorded on the equipment calibration form.

### 3.3 Tips for Good Calibration

- The DO calibration is a water-saturated air calibration. Make certain to loosen the calibration cup seal to allow pressure to equilibrate before calibrating.
- Make certain that sensors are completely submersed in solution and readings are stable when calibration values are entered.
- Use a small amount of calibration solution (previously used solution may be used, then discarded for this purpose) to pre-rinse the sonde.
- Fill a bucket with ambient temperature water to rinse the sonde between calibration solutions.
- Make sure to rinse and dry the probe between calibration solutions. This will reduce carry-over contamination and increase the accuracy of the calibration.

## 4.0 MAINTENANCE

The water quality meter will be rented for the duration of each brief field effort. Therefore, little field maintenance will be required. For any maintenance other than the routine cleaning, calibrating, or battery charging, the instrument should be returned to the vendor and a replacement sent immediately to the job site.

### 4.1 Meter Storage

For this field effort, the meter storage will be short term, [i.e. over night or between work shifts (4-day break)]. During these breaks, the meter will be charged. One-half inch of tap or distilled water will be placed in the meter calibration cup and the cup threaded onto the sonde. The key for short-term storage of probes is to use a minimal amount of water so the calibration cup will remain at 100 percent humidity. The water level must be low enough so that none of the probes are actually immersed. Proper storage of the sonde between usage will extend its life and will also ensure that the unit is ready for use as quickly as possible for the next application.

**Multi-parameter short term storage key points:**

- Use enough water to provide humidity but not enough to cover the probe surfaces.
- Make sure the storage vessel is sealed to minimize evaporation.

- Check periodically to make certain that water is still present.

## 4.2 Probe Cleaning

- Rinse the probe thoroughly with potable water.
- Wash the probe in a mild solution of Liquinox and water and wipe with paper towels and/or cotton swabs.
- Rinse and soak the probe in deionized water.
- If stronger cleaning is required, consult the equipment manual.

Note: Reagents that are used to calibrate and check the water quality meter may be hazardous. Review the health and safety plan, appropriate Appendix of the equipment manual, and Material Safety Data Sheets (MSDSs), all of which are on file in the field trailer.

## 5.0 ATTACHMENTS

1. Equipment Calibration Log

ATTACHMENT 1
EQUIPMENT CALIBRATION LOG

Tetra Tech NUS, Inc.

# EQUIPMENT CALIBRATION LOG

PROJECT NAME: _____ INSTRUMENT NAME/MODEL: _____

SITE NAME: _____ MANUFACTURER: _____

PROJECT No.: _____ SERIAL NUMBER: _____

| Date of Calibration | Instrument I.D. Number | Person Performing Calibration | Instrument Settings | | Instrument Readings | | Calibration Standard (Lot No.) | Remarks and Comments |
|---|---|---|---|---|---|---|---|---|
| | | | Pre-calibration | Post-calibration | Pre-calibration | Post-calibration | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |

# STANDARD OPERATING PROCEDURE
# SOP-013

## FIELD PORTABLE X-RAY FLUORESCENCE ANALYSIS OF SOIL AND SEDIMENT SAMPLES USING THE INNOV-X ALPHA SERIES INSTRUMENT

### 1.0 PURPOSE

This procedure is for the semiquantitative analysis of metallic lead particles and chemical compounds of lead in soil using a field portable x-ray fluorescence (FPXRF) spectrometer. This procedure is based on the United States Environmental Protection Agency (USEPA)-approved XRF field screening method for elemental analysis (Method 6200).

### 2.0 SCOPE, APPLICATION, AND LIMITATIONS

#### 2.1 Scope of Procedure

Analysis of any other elements beside lead using FPXRF may require changes to this Standard Operating Procedure (SOP), and are therefore outside the scope of this SOP.

Although it is possible to use FPXRF to measure analytes in situ, this SOP requires removal of a soil sample from its native environment prior to analysis. By removing, drying, and homogenizing the sample prior to analysis, more precise and accurate results are obtained.

#### 2.2 Analyst Training

Use of this method is restricted to personnel both trained and knowledgeable in the operation of the Innov-X alpha series XRF instrument or under the supervision of a trained and knowledgeable individual. Proper training for the safe operation of the instrument should be completed by the analyst prior to analysis. This training may be obtained directly from INNOV-X, an INNOV-X instrument distributor or lessor, or another trained Tetra Tech person.

## 3.0 ACRONYMS AND ABBREVIATIONS

FPXRF: Field portable x-ray fluorescence.

mg/kg: milligrams per kilogram.

MDL: Method detection limit.

PQL: Practical quantitation limit.

QC: Quality control.

RPD: Relative percent difference.

USGS: United States Geological Survey.

XRF: X-ray fluorescence.

## 4.0 RESPONSIBILITIES

Analyst/Chemist - Responsible for all aspects of sample preparation and analysis including equipment maintenance, Also responsible for maintaining chain-of-custody of samples after receipt from sampling personnel.

## 5.0 PROCEDURES

### 5.1 Safety

#### 5.1.1 Radiation Safety

Radiation safety practices for the INNOV-X instrument can be found in the operator's manual. Protective shielding should never be removed by the analyst or any personnel other than the manufacturer.

An additional hazard present with x-ray tubes is the danger of electric shock from the high voltage supply. The danger of electric shock is as substantial as the danger from radiation but is often overlooked because of its familiarity.

#### 5.1.2 Protective Equipment

Analysts must wear disposable plastic gloves whenever sample aliquots are being transferred from one vessel to another. Consult the health and safety plan for other protection requirements.

## 5.2 Apparatus and Materials

Apparatus and materials consist of the following:

INNOV-X Alpha Series FPXRF spectrometer with data processing unit (iPAQ) pocket personnel computer):

INNOV-X Alpha Series XRF instrument manual to match the INNOV-X Alpha Series instrument.

Aluminum drying pans or aluminum foil: Sized suitably to hold as much as 50 grams of sample and fit into the drying oven.

Calibration verification check sample: A National Institute of Standards and Technology (NIST) or other Standard reference material (SRM) that contains lead in a concentration range that is compatible with the project objectives to verify the accuracy of the instrument. SRMs can be obtained from the NIST, the U.S. Geological Survey (USGS), the Canadian National Research Council, and the national bureau of standards in foreign nations. Pertinent NIST SRMs for FPXRF analysis include 2704, Buffalo River Sediment; 2709, San Joaquin Soil; and 2710 and 2711, Montana Soil. These SRMs contain soil or sediment from actual sites that has been analyzed using independent inorganic analytical methods by many different laboratories. Acceptable limits for SRM percent recoveries are usually provided with the SRM. In their absence, a limit of ± 30 percent will be used as a guideline.

Instrument Blank: May be silicon dioxide, a Teflon block, a quartz block, "clean" sand, or lithium carbonate.

Lead calibration check standard: Supplied by the FPXRF manufacturer.

Method blank material for performing method blank checks: May be lead-free silica sand or lithium carbonate that undergoes the same preparation procedure as the samples.

Battery charger.

Polyethylene sample cups: 31 millimeters (mm) to 40 mm in diameter with collar, or equivalent (appropriate for FPXRF instrument).

X-ray window film: Mylar™, Kapton™, Spectrolene™, polypropylene, or equivalent; 2.5 to 6.0 micrometers (µm) thick.

Sample containers: glass or plastic to store samples.

Sieves: 60-mesh Stainless steel, Nylon, or equivalent for preparing soil and sediment samples if necessary.

Trowels: for collecting soil samples.

Plastic bags: used for collection and homogenization of soil samples. May also be used as sample presentation device.

Drying oven: standard convection or toaster oven, for soil samples that require drying.

Rolling pin (optional): Wooden rolling pin for crushing samples.

## 5.3 Sample Collection, Preservation, and Handling

Samples shall be provided to the FPXRF analyst in plastic bags. The analyst is responsible for maintaining chain-of-custody of all samples until all analyses have been successfully completed. No sample preservation is necessary. All samples shall be handled in accordance with sample handling SOPs in effect for the field event.

## 5.4 Preventive Maintenance

Refer to the instrument manual for specific manufacturer's recommendations.

## 5.5 Instrument Start-Up

5.5.1 Ensure the pocket PC (iPAQ) is plugged into the FPXRF instrument body and install a fully charged battery into the instrument.

5.5.2 Press the ON/OFF button on the base of the pistol grip of the instrument. If the iPAQ does not automatically power up, press the Power button in the right corner of the iPAQ.

5.5.3 Tap the Microsoft icon at the upper left corner of the iPAQ.

5.5.4 Chose START.

5.5.5 Tap "Soil Mode" on the menu or choose Mode (bottom of screen) and then choose Soil Mode from the drop down menu.

5.5.6 Allow the instrument to warm up (approximately 3 minutes).

5.5.7 Release the manual trigger lock.

5.5.8 Standardize the instrument in accordance with Section 5.6.

## 5.6 Standardization/Calibration Check

It is not possible to start an analysis if the instrument has not been standardized. To verify proper calibration of the instrument it is necessary to periodically standardize it using the automated standardization procedure. This must be done anytime the instrument is restarted and every 4 hours of operation, although re-standardization may be done at any other time (e.g., when instrument drift is suspected).

5.6.1 Click the standardization piece (supplied with the instrument) on the front of the instrument, verifying that the solid portion of the standard completely covers the analysis window.

5.6.2 Select "Tap here to Standardize" or select *File → Standardize*. The red light on top of the instrument will blink indicating that the instrument is producing x-rays and the shutter is open. The amber light on the rear of the instrument will also be illuminated and a status bar will appear to display the progress of the standardization.

5.6.3 Upon successful standardization the message "Successful Standardization" will appear along with the instrument resolution. In this case tap "ok" to dismiss the completion message. If problems are encountered, either follow the prompts that appear and/or repeat the standardization. Contact the FOL if problems persist. Take note of any error messages that appear as they may be useful if the instrument manufacturer must be contacted. Additional assistance is also available in the manufacturer's instrument manual.

## 5.7 Quality Control

The quality control (QC) program includes analysis of blanks calibration verification checks, duplicate analyses, and field duplicate samples. For all the above areas, any identified problems and corrective action must be documented in the instrument run log, analysis narrative report, and instrument maintenance log or standards log (as applicable). Identical operating conditions will be used for each sample.

### 5.7.1 Laboratory Blanks

Two types of blank samples shall be analyzed for FPXRF analysis: instrument blanks and method blanks.

5.7.1.1 At the beginning of each day, at the end of each day, and after every 20th sample or when potential contamination of the instrument is suspected, analyze an instrument blank to verify that no contamination exists in the spectrometer or on the probe window.

If the lead concentration in the blank exceeds the method detection limit (MDL), see Section 5.9.3) check the probe window and other potentially contaminated instrument components for contamination. If contamination is not causing the elevated blank readings, "zero" the instrument according to manufacturer's instructions.

5.7.1.2 After every 20th sample analyze a method blank. If the method blank lead concentration exceeds the practical quantitation limit (PQL, see Section 5.9.4), identify the cause of the elevated lead concentration and reanalyze all samples since the last acceptable method blank.

### 5.7.2 Calibration Verification Checks

5.7.2.1 After performing each blank check (Section 5.7.2), analyze a calibration verification check sample to check the accuracy of the instrument and to assess the stability and consistency of the analysis for the analytes of interest.

5.7.2.2 If the measured lead percent recovery (See Section 5.9.1) is less than 80 percent or greater than 120 percent, reanalyze the check sample. If the value continues to fall outside this acceptance range, the instrument should be recalibrated, or restandardized

according to the manufacture instructions and the batch of samples analyzed before the unacceptable calibration verification check must be reanalyzed.

### 5.7.3 XRF Duplicate Samples

XRF duplicate samples are two portions of the same sample that have been prepared and homogenized together, and then split and analyzed in the same manner by the XRF analyst.

5.7.3.1 Analyze an XRF duplicate at a frequency of 1 per 20 or once per day, whichever is more frequent.

5.7.3.2 If the computed RPD (See Section 5.9.2) exceeds 50 percent reanalyze both samples. If the RPD again exceeds 50 percent RPD consider whether the high degree of imprecision is caused by sample heterogeneity or other causes. This assessment may be aided by repeating the analysis of a sample that was analyzed previously. If the observed imprecision is attributed to sample heterogeneity, increase the number of readings made per sample to try to limit the imprecision and repeat the analyses. If this does not correct the problem notify the FOL.

## 5.8 Sample Analysis

**Note:**

This section provides sample analysis instructions, assuming that appropriate instrument start-up and calibration checks have been completed. The longer the instrument count time, the lower the detection limits and the less uncertainty there is with a recorded result. Count time is user-selectable through the instrument's software. Because the XRF data will be used in a screening capacity to make preliminary decisions concerning the soil concentrations relative to 400 mg/kg, it is not necessary to obtain a high degree of accuracy or precision with the instrument. Therefore, count times should be limited to less than 180 seconds unless an usually high degree of precision is expected. To change the count time, select Options → Setup Testing and enter the same value (in seconds) to minimum and maximum count times.

**Note:**

Section 5.7 identifies the appropriate frequencies for conducting various QC sample analyses and the associated acceptance limits and corrective actions for potentially unsuitable conditions. The

specified QC analysis frequencies are minimum frequencies. More frequent QC sample analyses are permitted, especially when diagnosing quality problems.

5.8.1 Ensure that calibration checks and blanks have been analyzed according to Sections 5.6, 5.7.1, and 5.7.2. Count times shall be at least 60 seconds but generally less than 180 seconds.

5.8.2 Acquire enough soil sample to fill an 8-ounce jar and separate from it all particles greater than the size of a pea.

5.8.3 Homogenize the remaining finer grained portion of the sample by simple mixing until it appears as uniform in texture and composition as practicable. Mixing may be done in a beaker or other suitable lead-free container. If the sample is moist and has high clay content, it may be kneaded in a plastic bag. Mixing shall continue for at least two minutes to ensure that the sample is well mixed. To aid mixing, the sample may be placed into a thick-walled (3 mil or thicker) gallon-sized freezer bag (e.g., ZipLoc®) and rolled flat with a rolling pin to break up large chunks of dirt.

5.8.4 Place approximately 20 to 50 grams (one U.S. nickel weighs about 5 grams) in a suitable container (e.g., aluminum drying pan) for drying.

5.8.5 Dry the homogenized sample from Step 5.8.4 for approximately 20 to 30 minutes in the oven at a temperature not greater than 150°C (a setting of approximately 300°F). If the sample is not visibly dry after this initial drying time, place the sample back into the oven until the sample is dry.

5.8.6 Re-homogenize the dried sample aliquot in a beaker or other suitable lead-free container to obtain a well mixed soil sample. Mixing shall continue for at least one minute.

5.8.7 Place a portion (approximately 1.5 cubic inches) of the dried, homogenized sample aliquot into the instrument manufacturer's recommended sample cup (e.g., a 31.0-mm polyethylene sample cup (or equivalent) or place it in a thin-walled (1.0 mil or thinner) plastic sandwich bag (e.g., ZipLoc®).

5.8.8 If using a disposable plastic sample cup, ensure the cup is at least three-quarters full and cover with mylar (or other) film per the manufacturer's recommendations.

5.8.9 Present the sample to the instrument in Soil Mode.

5.8.10 Perform a single pull of the trigger to start the count. The count time shall be the same as was used for the calibrations, calibration checks, and blank analyses. The message "Test in progress" will appear on the instrument and the red light on top of the instrument and will illuminate.

5.8.11 When the predetermined count period has expired the message "Test complete" will appear on the instrument. A slight delay may also be incurred during which time the message "calculating" may appear to indicate that results are being computed.

5.8.12 Record the displayed results for lead concentration in mg/kg and the associated error on Figure 1.

**CAUTION**

Inconsistent positioning of samples in front of the probe window is a potential source of error because the x-ray signal decreases by the square of the distance from the radioactive source. This error is minimized by maintaining the same distance between the window and each sample. For the best results, the window of the probe should be in direct contact with the sample, which means that the sample surface should be flat and smooth to provide a good contact surface.

5.8.13 Remix the sample in the plastic bag (or rotate the sample cup approximately one-third of a turn) then acquire another measurement by repeating Steps 5.8.10 and 5.8.11. Record the result and associated error on Figure 1.

5.8.14 Repeat Steps 5.8.10 and 5.8.11. Record the result and associated error on Figure 1 (XRF Field Form).

5.8.15 Based on the degree of precision demonstrated by the three individual measurements, determine whether additional readings should be acquired on the sample. This determination shall be based on professional judgment of the FPXRF analyst and shall consider the degree of precision observed during calibration checks and previous sample analyses. The objective will be to ensure that the average reading reported for each

sample is representative of the true sample concentration. If the analyst feels that non-representative readings are being obtained the analyst shall correct the analytical system or notify the FOL prior to continuing with analyses.

5.8.16 Ensure that measured results are reported to the following standards

- Results < 1000 mg/kg (or parts per million) are reported to two significant figures and results > 1000 mg/Kg are reported to three significant figures.
- All values < MDL shall be reported as the MDL and flagged with the letter "U".
- All values > MDL and < PQL shall be reported as is and flagged with the letter "B".

## 5.9 Calculations

5.9.1 Percent Recovery: The equation for determining percent recovery of calibration verification check standards and standard reference materials is:

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

5.9.2 Relative Percent Difference: The equation for determining relative percent difference for laboratory and field duplicate samples is:

$$RPD = \frac{|\text{Amount in Sample 1} - \text{Amount in Sample 2}|}{0.5\,(\text{Amount in Sample 1} + \text{Amount in Sample 2})} \text{ X } 100\,\%$$

5.9.3 Method Detection Limit (MDL): Because the analyses governed by this SOP are semi-quantitative, the manufacturer-specified detection limit will be reported as the MDL unless the specified detection limit is less than 20 mg/kg. Care will taken to ensure that the appropriate count time is consistent with the reported detection limit. However, no value less than 20 mg/kg will be reported as an MDL.

5.9.4 Practical Quantitation Limit (PQL): Multiply the MDL by 3 to obtain the PQL: PQL = MDL*3

## 6.0 REFERENCES

Innov-X Systems, Inc. Innov-X Systems X-Ray Fluorescence Spectrometers Instruction Manual. Woburn, MA. June 2002.

Stephen Shefsky, NITON Corporation. Comparing Field Portable X-Ray Fluorescence (XRF) To Laboratory Analysis of Heavy Metals In Soil. Presented at the International Symposium of Field Screening Methods for Hazardous Wastes and Toxic Chemicals. Las Vegas, Nevada. January 29-31, 1997.

USEPA (U.S. Environmental Protection Agency), Method 6200: Field Portable X-Ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment, Office of Solid Waste, Washington, D.C. January 1998.

USEPA, Region I, Northeast Waste Management Officials' Association (NEWMOA) Technology Review Committee Advisory Opinion. Innovative Technology: X-Ray Fluorescence Field Analysis. September 21, 1999.

# APPENDIX B

# LABORATORY STANDARD OPERATING PROCEDURES

# LABORATORY STANDARD OPERATING PROCEDURES (SOPs)
# TABLE OF CONTENTS


| SOP NUMBER | REV # | DATE | TITLE |
|---|---|---|---|
| | | | **EMPIRICAL LABORATORIES** |
| 100 | 19 | 7/25/06 | Metals Digestion/Preparation Methods 3005A,3010A, 3020A, 3030, 3040A, 3050B, USEPA CLP ILMO 4.1 Aqueous & Soil/Sediment, USEPA Method 200.7 |
| 105 | 14 | 09/09/08 | Metals Analysis by ICP Technique Methods 200.7, SW846 6010B, SM 19th Edition 2340B, USEPA ILMO 4.1 |
| 187 | 6 | 09/05/06 | Electrometric Determination of pH, Methods 150.1, Standard Methods 4500H+B and 9040B for Waters, Liquids and Liquid Wastes, 9045C for Soils and Solid Wastes |
| 221 | 7 | 10/27/08 | Total Organic Carbon SM5310C, USEPA Method 415.1 and SW846 Method 9060 and Lloyd Kahn Method |
| 231 | 3 | 01/16/09 | GC/MS Low Level PAHs By SW-846 Method 8270C SIM |
| 300 | 17 | 09/23/08 | GC/MS- Semivolatile BNA-Aqueous Matrix Extraction Using SW-846 Method 3510C for 8270C/625 Analysis |
| 329 | 16 | 09/24/08 | Soxhlet Extraction- BNA and Pest/PCB Using SW-846 Method 3541 |
| 404 | 12 | 01/05/09 | Laboratory Sample Receiving Log-in and Storage Standard Operating Procedures |
| 405 | 4 | 09/26/03 | Analytical Laboratory Waste Disposal |
| 410 | 6 | 09/08/08 | Standard Operating Procedures for Laboratory Sample Storage, Secure Areas, and Sample Custody |
| | | | **GPL LABORATORIES** |
| F.2 | 19 | 04/08 | Sample Receipt, Inspection, Preservation and Storage Condition Requirements |
| G.22 | 4 | 07/08 | General Laboratory Multi-Incremental Sampling (MIS) Sub-sampling Procedure |
| S.11 | 1 | 05/07 | High Performance Liquid Chromatography (HPLC) Analysis of Nitroaromatics and Nitroamine Explosive Residues in Water, Soil and Sediment Samples |
| D.1 | 5 | 10/06 | Laboratory Waste Handling and Storage Procedure |
| G.10 | 6 | 02/07 | Instrument Maintenance |
| G.12 | 7 | 10/06 | Standard Operating Procedures for Report Generation |

SOP No: D.1

Title: Laboratory Waste Handling and Storage Procedure

Scope: This Standard Operating Procedure details the procedures used to handle, label, store, and dispose of both hazardous and non-hazardous laboratory wastes including samples, sample byproducts, waste chemicals, spent solvents.

TABLE OF CONTENTS

| SECTION | | PAGE |
|---|---|---|
| 1. | INTRODUCTION | 3 |
| 2. | APPLICATION, REFERENCES | 3 |
| 2.1 | APPLICATION | 3 |
| 2.2 | REFERENCES | 3 |
| 3. | RESPONSIBILITIES | |
| 3.1 | CORPORATE | 3 |
| 3.2 | LABORATORY SUPERVISOR | 3 |
| 3.3 | HAZARDOUS WASTE MANAGER | 4 |
| 3.4 | WASTE MANAGEMENT TEAM | 4 |
| 3.5 | ANALYST | 5 |
| 4. | PROGRAM REQUIREMENTS | |
| 4.1 | AQUEOUS WASTE | 5 |
| 4.2 | NON-AQUEOUS WASTE | 6 |
| 4.3 | FREON WASTE | 6 |
| 4.4 | FOREIGN SOIL SAMPLES | 6 |
| 4.5 | ACUMULATION AND STORAGE OF WASTE | 7 |
| 4.6 | WASTE LABELING | 7 |
| 5. | RECORDS | 8 |

## 1.0 INTRODUCTION

1.1 GPL Laboratories, LLLP is a generator of hazardous waste, and as such GPL is subject to the compliance requirements in the Environmental Article, subpart 7-206, of the Annotated Code of Maryland and COMAR 26.13.03. In order to meet or exceed the requirements of COMAR 26.13.03, GPL has set up standard operating procedures for chemical waste handling and storage.

## 2.0 APPLICATIONS, REFERENCES, AND SOURCE DOCUMENTS

2.1 Application

This procedure applies to the handling and storage of all non-redioactive waste generated by the laboratory.

2.2 References

The following documents may need to be referenced when using this procedure:

- COMAR Title 26
- 40 CFR parts 260-265
- Manual of Hazardous Chemical Reactions, 1986 Edition
- 1989/1990 Hazardous Materials, Substances and Wastes Compliance Guide

## 3.0 RESPONSIBILITIES

3.1 Corporate Responsibility (Senior Management)

3.1.1 Overall responsibility for health and safety of the laboratory employees, the public and the environment.

3.1.2 Authorizing payments necessary for the removal of hazardous wastes from the laboratory.

3.1.3 Overseeing all activities performed by the Lab Managers and the Hazardous Waste Manager.

3.2 Lab Supervisor

The lab supervisor shall be responsible for the following activities:

3.3.1 Assuring removal of wastes from the lab to the temporary storage cart inside of the waste disposal/storage area.

3.3.2 Isolating and labeling wastes before they are removed from the lab to the waste storage/disposal area.

3.3 Hazardous Waste Manager

The waste manager is responsible for the following activities:

3.3.1 Keeping the Laboratory General Manager informed of any changes in Federal, State, or local laws that affect waste disposal.

3.3.2 Recognizing opportunities to implement source reduction programs.

3.3.3 Recognizing opportunities to implement waste recycling programs.

3.3.4 Developing waste handling, storage, record keeping, disposal, emergency response.

3.3.5 Purchasing or repairing waste materials emergency response equipment as becomes necessary.

3.3.6 Purchasing and maintaining waste sampling and storage equipment as becomes necessary.

3.3.7 Choosing methods of disposal for wastes.

3.3.8 Keeping records of all samples that have been disposed according to work orders.

3.3.9 Choosing waste disposal companies with which to do business.

3.3.10 Maintaining the analytical profile for all disposed soil waste drum.

3.4 Waste Management Team

The Waste Management Team shall be responsible for the following activities:

3.4.1 Inspection of all laboratory satellite waste containers on a weekly basis. All satellite containers which are at least ½ full are to be emptied into the appropriate waste collection container in the waste management area and returned empty or replaced as needed.

3.4.2 Verifying the suitability of residual water samples for sanitary sewer discharge using the LIMS reporting system and disposing of approved residual water samples once the required post-analysis holding time has elapsed. Once the residual sample has been dumped, the sample label must be removed or defaced in such a way as to permanently obscure identifiable client information, then the empty container is disposed of as non-hazardous waste.

3.4.3 Crushing residual soil samples and jars in drums for storage prior to disposal.

3.4.4 Execution of all other program requirements listed in section 4.0 below.

3.5 Analyst

The analyst who generates a waste is responsible for the following activities:

3.5.1 Minimizing wastes generated.

3.5.2 Storing hazardous, or potentially hazardous wastes in appropriate containers.

3.5.3 Labeling sample and chemical wastes appropriately.

3.5.4 Attending training sessions for waste handling and disposal.

3.5.5 Removing properly labeled wastes from the lab to the waste storage area.

4.0 PROGRAM REQUIREMENTS

4.1 Aqueous Waste

4.1.1 Residual aqueous samples are held for 30 days following analysis, unless contract requirements specify a longer holding time. Once the post-analysis holding requirement is met, the WMT will verify the sample's suitability for sewer disposal using the LIMS reporting system. Samples which meet the laboratory discharge requirements are dumped, the sample labels are permanently defaced, and the empty containers are disposed of as non-hazardous waste. All samples dumped in this manner are recorded in the aqueous sample disposal logbook. Residual aqueous samples which do not meet discharge requirements are returned to the client if possible. If this is not possible, the residual sample is removed by an approved waste hauler as part of the routine waste pickup.

4.1.2 Aqueous metal digestates are neutralized to a pH of 6-9. The neutralized digestate is then disposed of in the same manner as a residual aqueous sample (see 4.1.1)

4.1.3 Some laboratory methods, particularly in the Wet Chemistry Laboratory generate aqueous waste that is known to fall within the laboratory discharge limits. This waste is discharged into the sewer upon the completion of the associated analysis, and the discharge is documented in the analysis run log. Aqueous wastes discharged in this manner must be documented in the relevant analytical SOP and be approved by the HWM.

4.2 Non-Aqueous Waste

4.2.1 Soil samples are placed in steel drums and crushed for storage and eventual disposal using the procedure described in SOP D.2

4.2.2 Residual oil samples are collected in an appropriate container in the waste management area. On a 90 day schedule, the container is sampled, tested for regulated hazardous materials, and transferred to an approved waste hauler for disposal.

4.2.3 Chlorinated waste is collected in clearly labeled Nalgene containers in the waste management area. The containers must be tightly capped to prevent the evaporation of volatile solvents. On a 90 day schedule, the accumulated chlorinated waste is transferred to an approved waste hauler for disposal.

4.2.4 Non-chlorinated waste is collected in clearly labeled Nalgene containers in the waste management area. The containers must be tightly capped to prevent the evaporation of volatile solvents. On a 90 day schedule, the accumulated non-chlorinated waste is transferred to an approved waste hauler for disposal.

4.2.5 Non-aqueous waste that is known or suspected to contain PCBs is subject to a variety of additional regulations. These waste materials are disposed of according to the procedure described in SOP D.4.

4.3 Freon Waste

4.3.1 Based on the analysis methodology or the recommendation of the wet chem manager, determine if a waste is freon.

4.3.2 Combine and contain freon waste with other freon wastes and keep in wet chemistry lab.

4.3.3 Should the freon waste be no longer recyclable, it should be disposed as chlorinated waste.

4.4 Foreign Soil Samples

All foreign/imported soil samples are segregated from other samples in a designated refrigerator. Following analysis, the remaining samples are autoclaved according to the procedure described in SOP G.17. After autoclaving, they are disposed of in the same manner as residual soil samples(see 4.1.1 and SOP D.2)

4.5 Accumulation and Storage of Laboratory Waste

4.5.1 On a regular schedule determined by the HWM, the WMT will inspect each satellite accumulation area in the laboratory.

4.5.1.1 All satellite accumulation containers more than half full are capped, placed on a cart, and transported to the waste management area.

4.5.1.2 All full crimp-top vial containers are closed and transported to the waste management area.

4.5.1.3 The date, time, and laboratories inspected are documented in the satellite waste removal logbook.

4.5.2 Satellite waste containers are emptied into the appropriate 90-day accumulation container in the waste management area. As 90-day containers are filled, new containers are labeled, placed in service, and the start date recorded when the first waste is placed in the new container

4.5.3 Emptied satellite waste containers are replaced in the originating laboratory.

4.6 Waste Labelling

4.6.1 All 90-day waste accumulation containers are labeled based on their waste content: Chlorinated, Non-Chlorinated, Soil for Incineration, Soil for Landfill Disposal, Lab-Pack Waste, Oil Waste, or TSCA PCB waste. All 90-day accumulation containers must show the start date when waste was first placed in the container. When the container is filled, it is taken out of service and the end date is placed on the label

4.6.2 Satellite waste containers must be clearly labeled with the type of waste for which they are used.

## 5.0 RECORDS

The following records are to be maintained by the waste manager.

5.1 A copy of each manifest signed by the facility which received the waste.

5.2 All records of any test results, waste analyses, or other determinations made to characterize our waste as hazardous.

5.3 All correspondence with waste disposal companies pertaining directly to our waste disposal.

SOP No: F.2

Title: Sample Receipt, Inspection, Preservation and Storage Condition Requirements

Scope: This Standard Operating Procedure describes procedures to be used by Sample Control personnel in the inspection of incoming samples and the preservation and storage requirements of those samples.

## 1.0 SAFETY CONSIDERATIONS

1.1 When working with laboratory samples, safety must be of prime concern. It is required that safety glasses, a lab coat, and gloves be worn while unpacking or handling samples. Broken samples should be treated as a chemical spill and should be managed accordingly, using proper spill cleanup procedures (see SOP "Spill Cleanup"). Bench tops should routinely be covered with a bench liner, which should be changed as needed. Good personal hygiene procedures should be followed while in the Sample Control Area, specifically, it is recommended that hands be washed before or immediately after leaving the area, food or drink shall not be consumed, cosmetics shall not be applied, and smoking is forbidden in the Sample Control Area and throughout all areas of the building.

1.2 Sample shipping containers must be placed under the fume hood prior to opening. Once the shipping container is opened, the contents should be examined for hazards prior to removal from the hood. If no obvious hazards are identified, the container may be removed from the hood for unpacking.

1.3 If samples are from DOE site of known or suspected radioactive hazard, the sample shipping containers must be screened for radioactivity prior to opening.

## 2.0 DOCUMENTATION OF INSPECTION

2.1 The inspection of all incoming sample shipments shall be documented on the Sample Receipt Checklist (Figure 1), except that those samples received under the Contract Laboratory Program shall be documented on CLP Form DC-1 (Figure 2). The Checklists shall be used to coordinate the inspection of the shipment and to record important information about the condition of the shipment and the documentation received.

SOP No: F.2

Title: Sample Receipt, Inspection, Preservation and Storage Condition Requirements

Scope: This Standard Operating Procedure describes procedures to be used by Sample Control personnel in the inspection of incoming samples and the preservation and storage requirements of those samples.

## 1.0 SAFETY CONSIDERATIONS

1.1 When working with laboratory samples, safety must be of prime concern. It is required that safety glasses, a lab coat, and gloves be worn while unpacking or handling samples. Broken samples should be treated as a chemical spill and should be managed accordingly, using proper spill cleanup procedures (see SOP "Spill Cleanup"). Bench tops should routinely be covered with a bench liner, which should be changed as needed. Good personal hygiene procedures should be followed while in the Sample Control Area, specifically, it is recommended that hands be washed before or immediately after leaving the area, food or drink shall not be consumed, cosmetics shall not be applied, and smoking is forbidden in the Sample Control Area and throughout all areas of the building.

1.2 Sample shipping containers must be placed under the fume hood prior to opening. Once the shipping container is opened, the contents should be examined for hazards prior to removal from the hood. If no obvious hazards are identified, the container may be removed from the hood for unpacking.

1.3 If samples are from DOE site of known or suspected radioactive hazard, the sample shipping containers must be screened for radioactivity prior to opening.

## 2.0 DOCUMENTATION OF INSPECTION

2.1 The inspection of all incoming sample shipments shall be documented on the Sample Receipt Checklist (Figure 1), except that those samples received under the Contract Laboratory Program shall be documented on CLP Form DC-1 (Figure 2). The Checklists shall be used to coordinate the inspection of the shipment and to record important information about the condition of the shipment and the documentation received.

2.2 The Sample Receipt Checklist documents the following information: The top section of the page documents receipt of the shipment, the identity of the client, the assigned Work Order Number and information relevant to the client's project. The next section of the checklist section contains specific items which must be checked and documented. Included in the section are spaces to document the sample and sample bottle count for comparison to the client supplied paper work and to document the necessity of a pH check, when applicable. If a pH check is required, it shall be documented on the Sample Preservation Check Documentation Form (Figure 3).

2.3 The final section for comments is used to explain any problems or discrepancies with the shipment. Any "no" response to the previous section must be documented in the spaces provided. The sample control personnel will sign and date the form as indicated at the bottom of the page.

2.4 CLP Form DC-1 is used in the same manner as the Sample Receipt Checklist and shall be used in place of that form for the documentation of inspection of CLP sample shipments. All CLP samples received must be documented individually on Form DC-1.

2.5 The completed checklist is included with the original paper work submitted to the Project Management Group to be placed in the central project file. A copy of the completed Sample Receipt Checklist (or Form DC-1) is filed with copies of client supplied paper work, the Work Order and Chain-of-Custody documentation in the Sample Log book maintained by Sample Control.

2.6 Documentation of any problems or discrepancies associated with sample receipt is performed by the Sample Coordinator on the Sample Receipt Checklist (or Form DC-1). The Project Management Group is then informed of these items and is responsible in resolving the issues with the client. Resolution to any problems must be documented by the Project Management Group.

## 3.0 SAMPLE SHIPMENT RECEIPT

3.1 Each shipment of samples arriving by commercial carrier must be accompanied by an airbill, airbill sticker, or manifest. The Sample Coordinator (also referred to as the Sample Custodian for EPA CLP work), upon taking custody of the shipment, shall sign the airbill/manifest and record the date and time the shipment was received in the space provided on the document. If no space is provided, documentation may be recorded on any open space or the back of the document.

3.2 The Sample Coordinator shall next initiate a Sample Receipt Checklist or in the case of a CLP shipment, Form DC-1. The samples must be accompanied with a Chain of Custody (COC) document. The sample acceptance policy must include that:

A. The person submitting the sample must provide full documentation with the sample, which must include:

- Sample Identification
- The location, date and time of collection
- The collector name, preservative added
- Matrix
- Any special remarks concerning the sample

B. Each sample or group of samples must include trip blanks, field blanks, equipment blanks, duplicates or other field submitted quality measures as required by the method.

C. Each sample must show evidence of proper preservation and use of sample containers allowed by the test method.

D. Each sample must be of adequate volume for the requested testing.

Any deviation from the policy, the client will be contacted and documented. The sample(s) will not be processed.

Where required by method, pH of the samples must be determined and reported on the checklist. The client name, date and time received, the carrier name, and the name of the Sample Coordinator shall be recorded in the appropriate sections of the forms. The presence or absence of the airbill/manifest shall be documented and, if present, the airbill/manifest number shall be recorded. Then, the temperature blank vial should be located, and the cooler temperature must be obtained, by inserting the calibrated thermometer into the temp. blank vial, and reading the temp. after 60-90 seconds. If no temp. blank exists, then the thermometer must be inserted in the center of the cooler, where the samples are located, and the temp. should be read after 60-90 seconds of thermometer insertion.

3.3 In the event that samples must be received on a weekend or holiday, special arrangements between the client and the laboratory must be established. Generally, clients must contact the Project Management Group to arrange for the special service. At that time Client Services shall ascertain if any samples requiring immediate analysis are to be included in the shipment. If this is the case, arrangements shall be made to have analytical staff available to perform the required analyses. Project Management Group shall arrange to have a person from Sample Control, or a properly trained alternate, available to receive the shipment. The shipment shall be received and inspected as per this procedure. The appropriate paperwork shall be completed and, if necessary, sample preservation shall be verified. Samples requiring immediate analysis are released upon receipt to the lab by the sample control personnel, before the completion of the transmission of the work order. Thereby enabling the laboratory to meet the required holding time criteria.

3.4 Short Holding Times and Rush Samples

Samples received that require either rush TAT (<72 hours) and or short holdings (<72 hours) require immediate attention by the Sample Management division. It is the responsibility of the Sample Management division to insure the appropriate laboratory receives the samples and are notified of the urgent circumstances. The Sample Management division will bring the samples to the appropriate laboratory manager along with a copy of the original COC or equivalent (written documentation showing the sample ID and the tests required). After the samples are logged in and approved, the analysts will fill out the appropriate internal COC.

## 4.0 SHIPPING CONTAINER INSPECTION

4.1 All sample shipping containers shall be inspected for integrity and physical damage upon receipt. This shall be documented on Line 2 of the Checklist. Any problems found must be documented in the comments section at the bottom of the page. For Form DC-1, comments shall be made in the Remarks section.

4.2 Safety Note: If a strong smell is emanating from a shipping container, place the container in the hood to be unpacked. If a liquid is leaking from a shipping container, place container in the hood on a metal or plastic tray to contain the liquid and prevent gross contamination of the Sample Control Area. Any leaking material should be considered as a hazardous material and should be treated as such. All sample shipments originating from the Sample Management Office (SMO) for the Contract Laboratory Program (CLP) must be opened and unpacked under the hood. In all cases, proceed with caution while checking for broken sample vessels.

4.3 Once the shipping container has been examined for damage, and the presence or absence of custody seals is determined. The sample receiving personnel will evaluate and document the use of chain of custody seals that are of the non-tamper evident variety. Then the overall condition should be ascertained. This check shall be documented in the appropriate spaces on the forms.

## 5.0 SAMPLE VESSEL INSPECTION

5.1 The shipping container shall be carefully opened, any paper work should be removed, and it shall be determined whether ice or cold packs are present. The temperature of the shipping container shall be measured by placing a thermometer in the provided temp blank or among the samples, closing the lid and reopening the container and reading the thermometer for approximately 60-90 seconds. On the sample receipt checklist, document the presence or absence of ice or cold packs and the temperature of the shipping container. Please note that if the samples do not require cold preservation, for example preserved water samples for metals analysis, "No" must be checked and an explanation of N/A included in the Comments section.

5.2 The shipping container should be carefully unpacked and the incoming samples checked for:

- Physical damage due to inadequate packing and/or protection.

- Loss of sample due to inadequate and/or improper sealing of the sample container (i.e., leakage of liquids, loss of particulate material from filters, etc.).

- Possible contamination because of inadequate separation of sample types or bulk sample materials (i.e., charcoal tubes or VOA vials shipped in the same container as bulk liquid organics).

- Adequate containment of volatile organic samples and total organic halide samples in septum vials; there should be zero headspace. If headspace is found, this shall be documented. A widely accepted standard for document the approximate size is to judge the headspace to be smaller of larger than a green pea. This should be noted the Checklist or noted in the sample Remarks section of Form DC-1 and the Project Manager/Client Services Group contacted, as the client may wish to re-sample.

- Proper use of special shipping procedures required to preserve the samples. For example, if shipping instructions note that samples are to be kept frozen, then samples should be frozen upon receipt.

If samples are broken or evidence that sample integrity may have been compromised, the incident shall be documented in the designated space of the Checklist and detailed in the Comments section. For CLP samples this shall be documented on Form DC-1, Line 8 and explained in the remarks section for the affected samples and shall also be noted on the Sample Traffic Report Form. The Project Management Group shall be notified of any such problems as soon as possible.

## 6.0 CONTRACT LABORATORY PROGRAM SAMPLE VESSEL INSPECTION

6.1 For samples submitted by SMO, a review of sample documentation is required once the shipping container has been unpacked. Each shipping container should possess a Chain-of-Custody Form and a Sample Traffic Report Form for the samples contained within the shipping vessel. Each sample should have a Sample Label and a Sample Tag attached to the bottle (where applicable). EPA sample bottles should also have a custody seal over the cap to detect tampering. All items shall be reviewed and documented on Form DC-1.

6.2 Presence or absence of custody seals should be determined. If present, condition of custody seals must be assessed. This shall be documented on Line 1 in the Remarks section of Form DC-1.

6.3 Presence of EPA Sample Tags on sample bottles should be determined. The presence or absence of tags shall be documented on Line 7.

6.4 It shall be determined if the EPA Traffic Report or SAS Packing list was included in the shipment. Documentation shall be made on Line 4.

6.5 It shall be determined if the Chain-of-Custody document was included in the shipment. Documentation shall be made on Line 3.

6.6 The Chain-of-Custody document shall be reviewed to verify the entry of Sample Tag numbers on the Chain-of-Custody. If tag numbers have not been recorded on the Chain-of-Custody, the Sample Coordinator shall perform the document entries and shall document the information in the remarks section for the affected sample.

6.7 The Chain-of-Custody shall be compared with the Sample Labels. Any discrepancies shall be documented. This shall be documented in the remarks section for the affected sample.

6.8 The Chain-of-Custody shall be compared with the Sample Tags. Any discrepancies shall be documented. This shall be documented in the remarks section for the affected sample.

6.9 The Chain-of-Custody shall be compared with the EPA Traffic Reports. Discrepancies shall be documented in the remarks section for the affected sample.

## 7.0 SAMPLE PRESERVATION INSPECTION

### 7.1 EPA Contract Laboratory Program (CLP) Samples

7.1.1 Samples which are submitted under the Contract Laboratory Program for inorganic parameters (metals and cyanide) are preserved in the field and may require pH adjustment. These samples are to be checked for proper preservation upon receipt. Because of the nature of these samples proper safety precautions must be observed. Safety glasses, lab coats and disposable gloves shall be worn when handling the sample bottles while performing pH measurements. Gloves shall be changed when soiled to prevent contamination between both the samples or of the employee.

7.1.2 The pH check shall be performed by removing an aliquot of the sample, pouring it into a small disposable cup and depositing the drop onto a pH strip paper. Match the resulting color of the pH paper to the color scale provided with the pH paper. Various ranges of pH papers are used for preservation check. For low range pH measurement, the pH indicator of 0-6 or 0-2.5 will be used. Other wise, pH indicator range 11-13 or 7.5-14 will be used. This will enable to obtain a definitive measurement of pH.

Samples for metals analysis must have a pH of less than 2, and samples for cyanide must have a pH greater than 12.

7.1.3 Record the pH reading on a Sample Preservation Check Documentation Form (Figure 3). The pH may be recorded as less than the value required for acid preserved samples (i.e. <2), or greater than the required value for NaOH preserved samples (i.e. >12 for NaOH preserved cyanide samples). If a sample is discovered to be inadequately preserved, the exact pH reading from the pH check must be recorded. The form shall be signed and dated by the responsible person.

7.1.4 If the pH check indicates that the sample was not adequately preserved, contact the Project Manager immediately to receive instructions from the Sample Management Office. Documentation of sample preservation deficiencies, and instructions given by the Sample Management Office shall be documented in the case file and the case narrative by the Project Manager. If the Sample Management Office representative dictates that the samples shall be preserved in the laboratory, documentation of this activity shall be indicated on the Sample Preservation Check Documentation Form (Figure 3). The samples may be preserved by Sample Control or laboratory personnel, where applicable. In either case, suitable quantities of the appropriate preservative shall be added to the inadequately preserved samples. Documentation of these activities shall be indicated on the Sample Preservation Check Documentation Form (Figure 3). Copy of the documentation form shall be filed in the case file.

7.1.5 Table 1 provides information on required containers, holding instructions, and holding times for USEPA CLP submitted samples.

7.2 Commercial Samples

7.2.1 EPA requires chemical preservation of water samples to be analyzed for selected parameters. Table 2 presents information which lists parameters, required containers, sample size needed, preservation techniques and holding times for sample analysis performed by methods specified in 40 CFR Part 136, Test Methods for Evaluating Solid Waste - SW 846, and Methods for Chemical Analysis of Water and Wastes (MCAWW). The Sample Coordinator is responsible for verifying that any samples requiring pH adjustment have been appropriately preserved in the field.

7.2.2 The pH check shall be performed by removing an aliquot of the sample, pouring it into a small disposable cup and depositing the drop onto a pH strip paper. Match the resulting color of the pH paper to the color scale provided with the pH paper. Various ranges of pH papers are used for preservation check. For low range pH measurement, the pH indicator of 0-6 or 0-2.5 will be used. Other wise, pH indicator range 11-13 or 7.5-14 will be used. This will enable to obtain a definitive measurement of pH.

Samples for metals analysis must have a pH of less than 2, and samples for cyanide must have a pH greater than 12.

7.2.3 Record the pH reading on a Sample Preservation Check Documentation Form (Figure 3). The pH may be recorded as less than the value required for acid preserved samples (i.e. <2), or greater than the required value for NaOH preserved samples (i.e. >12 for NaOH preserved cyanide samples). As an alternative a check mark may be used to signify that the sample met the preservation criteria. If a sample is discovered to be inadequately preserved the exact pH reading from the pH check must be recorded. The form shall be signed and dated by the responsible person.

7.2.4 If the pH check indicates that the sample was not adequately preserved, contact the Sample Control Supervisor immediately. The Sample Control Supervisor will contact the appropriate Project Manager who will, after consultation with the client, instruct the Sample Control Supervisor of the appropriate action to follow. Generally, the samples are preserved during that time and the necessary documentation of the problem and the resolution are performed. Also, notations are indicated in the final report describing the situation. Samples may be preserved by Sample Control or laboratory personnel. In either case, suitable quantities of the appropriate preservative shall be added to the inadequately preserved samples. See Table 2 for information on preservatives and criteria. Documentation of these activities shall be made on the Sample Preservation Check Documentation Form (Figure 3). Copy of the documentation form shall be filed in the case file.

**Note:** If drinking water metals samples are acidified in the laboratory, the time that the samples are acidified must be recorded on the preservation check form (Fig.3) in order to meet the 16 hour hold time requirement.

**Note:** If Radiochemistry samples are acidified in the laboratory, the time that the samples are acidified must be recorded on the preservation check form (Fig.3) in order to meet the 16 hour hold time requirement.

**Note:** Samples for Radiochemistry Tritium analysis should not be preserved.

7.2.5 Failure to appropriately preserve the samples in the field may result in invalid analytical data. It is the responsibility of Project Management Group to advise the client of this potential.

All acid preservatives are verified upon arrival to the lab.

7.2.6 Samples for State of North Carolina, which are chlorine sensitive, i.e., organics, ammonia, TKN and cyanide must be checked for the presence of chlorine and treated, if required, before storage and analysis. Documentation of these activities shall be noted on the sample residual chlorine check form (Figure 4).

7.3 pH Adjustment Procedure

7.3.1 Samples which require pH adjustment must have appropriate amounts of the required preservative added. This procedure must be carefully performed and must be properly documented. (Figure 3).

7.3.2 Generally, the preservative solutions are relatively concentrated (i.e. 1:1 Nitric or Sulfuric Acid, 5N NaOH) to avoid significant changes in volume. Therefore, only a small quantity of preservative should be added. Due to sample compositions, proper safety precautions must be observed. While performing pH measurements, safety glasses, lab coats and disposable gloves shall be worn. Gloves shall be changed when soiled to prevent sample contamination.

7.3.3 When a particular sample requires additional preservation the sample shall be opened and a small quantity of the preservative added. The amount of preservative added should be in increments of approximately 0.5ml/250ml of sample. Once the preservative is added the bottle shall be securely capped and shaken to distribute the preservative. The bottle shall then be carefully opened, and observed for evidence of escaping pressure before rechecking the pH. The pH check shall be performed by removing an aliquot of the sample, pouring it into a small plastic cup and depositing the drop onto a strip of pH paper. Match the resulting color of the pH paper to the color scale provided with the pH paper. If the pH does not yet meet the method specification, repeat the process until an acceptable value is maintained.

7.3.4 Documentation of the preservation adjustment and reagent lot number shall be indicated on the Sample Preservation Adjustment Documentation Form (Figure 3). Each space shall be completed for each listed sample and the form shall be signed and dated by the responsible person.

Figure 1

SAMPLE RECEIPT CHECKLIST

W.O. No: _____

Client Name: _____

Date Received: _____

Time Received: _____

Received By: _____

Carrier Name: _____

Prepared (Logged In) By: _____ / _____
Initials Date

Project: _____

Site: _____

VOA Holding Blank I.D. No: _____

| | YES | NO |
|---|---|---|
| Airbill/Manifest Present? | ___ | ___ |
| No. _____ | | |
| Shipping Container in Good Condition? | ___ | ___ |
| Custody Seals Present on Shipping Container? | ___ | ___ |
| Condition: Broken _____ | | |
| Intact-not dated or signed _____ | | |
| Intact-dated and signed _____ | | |
| Usage of Tamper Evident Type | ___ | ___ |
| Chain-of-Custody Present? | ___ | ___ |
| Chain-of-Custody Agrees with Sample Labels? | ___ | ___ |
| Chain-of-Custody Signed? | ___ | ___ |
| Packing Present in Shipping Container? | ___ | ___ |
| Type of Packing _____ | | |
| Custody seals on Sample Bottles? | ___ | ___ |
| Condition: Good _____ Broken _____ | | |
| Total Number of Sample Bottles _____ | | |
| Total Number of Samples _____ | | |
| Samples Intact? | ___ | ___ |
| Sufficient Sample Volume for Indicated Test? | ___ | ___ |

| | YES | NO |
|---|---|---|
| Trip Blanks: No. of Sets _____ | ___ | ___ |
| Field Blanks: No. of Sets _____ | ___ | ___ |
| Equip. Blank: No. of Sets _____ | ___ | ___ |
| Field Duplicate: No. of Sets _____ | ___ | ___ |
| MS/MSD: No of Sets _____ | ___ | ___ |
| VOA Vials Have Zero Headspace? | ___ | ___ |
| If no, smaller or greater than a Green Pea (see comments) | | |
| Preservatives Added to Sample? | ___ | ___ |
| pH Check Required? | ___ | ___ |
| Performed By? _____ | ___ | ___ |
| Ice Present in Shipping Container? | ___ | ___ |

| Container # | Temp. | Container # | Temp. |
|---|---|---|---|
| _____ | _____ | _____ | _____ |
| _____ | _____ | _____ | _____ |
| _____ | _____ | _____ | _____ |
| _____ | _____ | _____ | _____ |
| _____ | _____ | _____ | _____ |
| _____ | _____ | _____ | _____ |
| _____ | _____ | _____ | _____ |

Project Manager Contacted?
Name: _____
Date Contacted: _____

Any NO response must be detailed in the comments section below. If items are not applicable to particular samples or contracts, they should be marked N/A/

COMMENTS: _____

_____

_____

_____

Checklist Completed By: _____

Date: _____

Figure 2

SAMPLE LOG-IN SHEET

| | | | | | |
|---|---|---|---|---|---|
| Lab Name | | | | | Page __ of __ |
| Received By (Print Name) | | | | | Log-in Date |
| R ived By (Signature) | | | | | |
| Case Number | | Sample Delivery Group No. | | | NRAS Number |
| | | | Corresponding | | |
| Remarks: | | EPA Sample # | Aqueous Sample pH | Sample Tag # | Assigned Lab # | Remarks: Condition of Sample Shipment, etc. |
| 1. Custody Seal(s) | Present/Absent* Intact/Broken | | | | | |
| 2. Custody Seal Nos. | _____ _____ | | | | | |
| 3. Traffic Reports/Chain of Custody Records or Packing Lists | Present/Absent* | | | | | |
| 4. Airbill | Airbill/Sticker Present/Absent* | | | | | |
| 5. Airbill No. | _____ _____ | | | | | |
| 6. Sample Tags | Present/Absent* | | | | | |
| Sample Tag umbers | Listed/Not Listed on Traffic Report/Chain of Custody Record | | | | | |
| 7. Sample Condition | Intact/Broken*/ Leaking | | | | | |
| 8. Cooler Temperature Indicator Bottle | Present/Absent* | | | | | |
| 9. Cooler Temperature | _____ | | | | | |
| 10. Does information on Traffic Reports/Chain of Custody Records and sample tags agree? | Yes/No* | | | | | |
| 11. Date Received at Lab | _____ | | | | | |
| 12. Time Received | _____ | | | | | |
| Sample Transfer | | | | | | |
| Fraction | Fraction | | | | | |
| Area # | Area # | | | | | |
| By | By | | | | | |
| On | On | | | | | |

* Contact SMO and attach record of resolution

| | | |
|---|---|---|
| Reviewed By | | Logbook No. |
| Date | | Logbook Page No. |

Figure 3

Sample Preservation Check Documentation Form

Work Order: _____

| Parameter: Preservative: pH Value | Metals (*A) HN03 <2 | Phenol H2SO4 <2 | TPH O&G H2SO4 <2 | Classical Parameters H2SO4 <2 | Cyanide NaOH >12 | Sulfide NaOH >9 | Radiology (*B) HN03 <2 | Other Preservation Adjustment | Reagent Lot # | see checklist for comments |
|---|---|---|---|---|---|---|---|---|---|---|
| GPL ID | | | | | | | | | | |
| | | | | | | | | Y / N | | |
| | | | | | | | | Y / N | | |
| | | | | | | | | Y / N | | |
| | | | | | | | | Y / N | | |
| | | | | | | | | Y / N | | |
| | | | | | | | | Y / N | | |
| | | | | | | | | Y / N | | |
| | | | | | | | | Y / N | | |
| | | | | | | | | Y / N | | |
| | | | | | | | | Y / N | | |
| | | | | | | | | Y / N | | |
| | | | | | | | | Y / N | | |
| | | | | | | | | Y / N | | |
| | | | | | | | | Y / N | | |
| | | | | | | | | Y / N | | |
| | | | | | | | | Y / N | | |
| | | | | | | | | Y / N | | |
| | | | | | | | | Y / N | | |
| | | | | | | | | Y / N | | |
| | | | | | | | | Y / N | | |
| | | | | | | | | Y / N | | |
| | | | | | | | | Y / N | | |
| | | | | | | | | Y / N | | |
| | | | | | | | | Y / N | | |
| | | | | | | | | Y / N | | |
| | | | | | | | | Y / N | | |
| | | | | | | | | Y / N | | |

(*A). If drinking water metals sample is acidified in the laboratory,
the TIME that the samples are acidified MUST be recorded in order to meet the 16 hour hold time requirement.
(*B). If Radiochemistry sample is acidified in the laboratory,
the TIME that the samples are acidified MUST be recorded in order to meet the 16 hour hold time requirement.
Tritium analysis should NOT be preserved.

Sample Preservation Check Performed By: _____ Date: _____

Figure 4

North Carolina Sample Residual Chlorine Check Form

Work Order: _____

| Client ID | Organics | TKN | Ammonia | Cyanide | Comments |
|---|---|---|---|---|---|
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |

Sample Preservation Check Performed By: _____ Date: _____

Table 1
Summary of Contract Laboratory Program Required
Containers, Preservation Techniques, and Holding Times[1]

| Parameter | Container | Holding Instructions | Maximum Sample Holding Time |
|---|---|---|---|
| Metals | P, G | Room Temperature | 180 days from VTSR |
| Mercury | P, G | Room Temperature | 26 days from VTSR |
| Cyanide (Total and amenable to chlorination) | P, G | Room Temperature | 12 days from VTSR |
| Volatiles | G(TS) | Store at 4° ± 2°C | 10 days from VTSR |
| Semivolatiles (water) | G(TC) | Store at 4° ± 2°C (in dark) | 5 days from VTSR [a] |
| Semivolatiles (soil/sediment) | G(TC) | Store at 4° ± 2°C (in dark) | 10 days from VTSR [a] |
| Pesticides/PCBs (water) | G(TC) | Store at 4° ± 2°C (in dark) | 5 days from VTSR [a] |
| Pesticides/PCBs (soil/sediment) | G(TC) | Store at 4° ± 2°C (in dark) | 10 days from VTSR [a] |

[a] - Extract holding time is 40 days after extraction
P = plastic
G = glass
G(TC) = glass, TFE-lined cap
G(TS) = glass, TFE-lined septum

---

[1] - USEPA Contract Laboratory Program; Statement of Work for Inorganics Analysis; Multi-media, Multi-concentration; SOW No. 3/90

USEPA Contract Laboratory Program; Statement of Work for Organics Analysis; Multi-media, Multi-concentration; SOW No. 3/90

Table 2
Summary of Required Sample Containers, Sample Volumes,
Preservation Techniques And Storage Requirements
(from 40 CFR Part 136)

| Parameter Name | Container[1] | Minimum Sample Size (ml) | Preservation[2,3] | Maximum Holding Time[4] |
|---|---|---|---|---|
| **BACTERIAL TESTS** | | | | |
| Coliform, fecal and total | P, G (sterile) | 200 | Cool, 4°C, 0.008% $Na_2S_2O_3$[5] | 6 hours |
| Fecal Streptococci | P, G (sterile) | 200 | Cool, 4°C, 0.008% $Na_2S_2O_3$[5] | 6 hours |
| **INORGANIC TESTS[7]** | | | | |
| Acidity | P, G | 200 | Cool, 4°C | 14 days |
| Alkalinity | P, G | 200 | Cool, 4°C | 14 days |
| Ammonia | P, G | 200 | Cool, 4°C, $H_2SO_4$ to pH < 2 | 28 days |
| Biochemical Oxygen Demand | P, G | 1,000 | Cool, 4°C | 48 hours |
| Bromide | P, G | 250 | None | 28 days |
| Chloride | P, G | 50 | None Required | 28 days |
| Chlorine, total residual | P, G | 500 | None Required | Analyze immediately |
| Color | P, G | 500 | Cool, 4°C | 48 hours |

Table 2
Summary of Required Sample Containers, Sample Volumes,
Preservation Techniques And Storage Requirements
(from 40 CFR Part 136)

| Parameter Name | Container[1] | Minimum Sample Size (ml) | Preservation[2,3] | Maximum Holding Time[4] |
|---|---|---|---|---|
| Cyanide, total and amenable to chlorination | P, G | 1,000 | Cool, 4°C, $H_2SO_4$ to pH > 12, 0.6g ascorbic acid[5] | 14 days[6] |
| Fluoride | P | 1,000 | None Required | 28 days |
| Hardness | P, G | 200 | $HNO_3$ or $H_2SO_4$ to pH < 2 | 6 months |
| Hydrogen ion (pH) | P, G | 250 | None Required | Analyze Immediately |
| 3jeldahl and organic nitrogen | P, G | 500 | Cool, 4°C, $H_2SO_4$ to pH < 2 | 28 days |
| Chromium VI | P(A), G(A) | 300 | Cool, 4°C | 24 hours |
| Mercury | P(A), G(A) | 500 | $HNO_3$ to pH < 2 | 28 days |
| Metals, except Chromium VI and Mercury | P(A), G(A) | 500 | $HNO_3$ to pH < 2 | 6 months |
| Nitrate | P, G | 200 | Cool, 4°C | 48 hours |
| Nitrate-nitrite | P, G | 200 | Cool, 4°C, $H_2SO_4$ to pH < 2 | 28 days |
| Nitrite | P, G | 200 | Cool, 4°C | 48 hours |

Table 2
Summary of Required Sample Containers, Sample Volumes,
Preservation Techniques And Storage Requirements
(from 40 CFR Part 136)

| Parameter Name | Container[1] | Minimum Sample Size (ml) | Preservation[2,3] | Maximum Holding Time[4] |
|---|---|---|---|---|
| Oil and Grease | G, (wide mouth) | 2000 | Cool, 4°C, $H_2SO_4$ to pH < 2 | 28 days |
| Organic Carbon | P, G | 100 | Cool, 4°C, $H_2SO_4$ to pH < 2 | 28 days |
| Orthophosphate | P(A), G(A) | 200 | Filter Immediately, Cool 4°C | 48 hours |
| Oxygen, dissolved (probe) | G Bottle and Top | 300 | None Required | Analyze Immediately |
| Winkler | G Bottle and Top | 300 | Fix on site and store in dark | 8 hours |
| Phenols | G only | 1000 | Cool, 4°C, $H_2SO_4$ to pH < 2 | 28 days |
| Phosphorous, elemental | G | 200 | Cool, 4°C | 48 hours |
| Phosphorous, total | P, G | 200 | Cool, 4°C, $H_2SO_4$ to pH < 2 | 28 days |
| Residue, total | P, G | 200 | Cool, 4°C | 7 days |
| Residue, filterable (TDS) | P, G | 200 | Cool, 4°C | 48 hours |
| Residue, nonfilterable (TSS) | P, G | 200 | Cool, 4°C | 7 days |
| Residue, settleable | P, G | 200 | Cool, 4°C | 48 hours |

Table 2
Summary of Required Sample Containers, Sample Volumes,
Preservation Techniques And Storage Requirements
(from 40 CFR Part 136)

| Parameter Name | Container[1] | Minimum Sample Size (ml) | Preservation[2,3] | Maximum Holding Time[4] |
|---|---|---|---|---|
| Residue, volatile | P, G | 200 | Cool, 4°C | 7 days |
| Silica | P | 500 | Cool, 4°C | 28 days |
| Specific Conductance | P, G | 500 | Cool, 4°C | 28 days |
| Sulfate | P, G | 50 | Cool, 4°C | 28 days |
| Sulfide | P, G | 200 | Cool, 4°C, add zinc acetate plus sodium hydroxide to pH > 9 | 7 days |
| Sulfite | P, G | 100 | None Required | Analyze Immediately |
| Surfactants | P, G | 500 | Cool, 4°C | 48 hours |
| Temperature | P, G | 250 | None Required | Analyze |
| Turbidity | P, G | 250 | Cool, 4°C | 48 hours |

Table 2
Summary of Required Sample Containers, Sample Volumes,
Preservation Techniques And Storage Requirements
(from 40 CFR Part 136)

| Parameter Name | Container[1] | Minimum Sample Size (ml) | Preservation[2,3] | Maximum Holding Time[4] |
|---|---|---|---|---|
| **ORGANIC TESTS[8]** | | | | |
| Purgeable Halocarbons | G, Tef.-lined septum | 2X 40 | Cool, 4°C, 0.008% $Na_2S_2O_3$[5] | 14 days |
| Purgeable aromatic hydrocarbons | G, Tef.-lined septum | 2X 40 | Cool, 4°C, 0.008% $Na_2S_2O_3$[5], HCl to pH 2[9] | 14 days |
| Acrolein and acrylonitrile | G, Tef.-lined septum | 2X 40 | Cool, 4°C, 0.008% $Na_2S_2O_3$[5], Adjust pH to 4-5[10] | 14 days |
| Phenols[11] | G, Tef.-lined cap | 2000 | Cool, 4°C, 0.008% $Na_2S_2O_3$[5] | 7 days until extraction; 40 days after extraction |
| Benzidines[11] | G, Tef.-lined cap | 2000 | Cool, 4°C, 0.008% $Na_2S_2O_3$[5] | 7 days until extraction[13] |
| Phthalate esters[11] | G, Tef.-lined cap | 2000 | Cool, 4°C | 7 days until extraction; 40 days after extraction |
| Nitrosamines[12,14] | G, Tef.-lined cap | 2000 | Cool, 4°C, store in dark, 0.008% $Na_2S_2O_3$[5] | 7 days until extraction; 40 days after extraction |
| PCBs[11] acrylonitrile | G, Tef.-lined cap | 2500 | Cool, 4°C | 7 days until extraction; 40 days after extraction |

Table 2
Summary of Required Sample Containers, Sample Volumes,
Preservation Techniques And Storage Requirements
(from 40 CFR Part 136)

| Parameter Name | Container[1] | Minimum Sample Size (ml) | Preservation[2,3] | Maximum Holding Time[4] |
|---|---|---|---|---|
| Nitroaromatics and isophorone[11] | G, Tef.-lined cap | 2000 | Cool, 4°C, 0.008% $Na_2S_2O_3$[5]. Store in dark | 7 days until extraction; 40 days after extraction |
| Polynuclear aromatic hydrocarbons[11] | G, Tef.-lined cap | 2000 | Cool, 4°C, 0.008% $Na_2S_2O_3$[5] | 7 days until extraction 40 days after extraction |
| Haloethers[11] | G, Tef.-lined cap | 2000 | Cool, 4°C, 0.008% $Na_2S_2O_3$[5] | 7 days until extraction; 40 days after extraction |
| Chlorinated hydrocarbons[11] | G, Tef.-lined septum | 2X 40 | Cool, 4°C | 7 days until extraction; 40 days after extraction |
| **PESTICIDES** | | | | |
| Pesticides[11] | G, Tef.-lined cap | 1000 | Cool, 4°C, pH 5-9[15] | 7 days until extraction 40 days until extraction |
| **RADIOLOGICAL TESTS** | | | | |
| Alpha, beta, radium | P, G | 2000 | $HNO_3$ to pH 2 | 6 months |

1. Polyethylene (P) or Glass (G), G(A) or P(A) = rinsed with 1:1 Nitric Acid

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 Mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of Table II, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight 0000000or less (pH about 1.96 or greater); Nitric acid ($HNO_3$) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid ($H_2SO_4$) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

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 time, and has received a variance from the Regional Administrator under 136.3(e). Some samples may not be stable for the maximum time period given in the table. A permittee, or monitoring laboratory, is obligated to hold the sample for a shorter time if knowledge exists to show that this is necessary to maintain sample stability. See 136.3(e) for details.

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 in order 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 seven days before extraction and for forth 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 and 13 (re: the analysis of benzidine).

12. If 1,2-Diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0 $\pm$ 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% $Na_2S_2O_3$.

UNCONTROLLED DOCUMENT
AND
BUSINESS CONFIDENTIAL



SOP No: G.10

Title: Instrument Maintenance

1.0 Scope and Application

1.1 This Standard Operating Procedure describes the maintenance required by laboratory instrumentation and the records to be maintained.

2.0 Summary

2.1 To minimize downtime and interruption of analytical work, preventive maintenance should be routinely performed on each analytical instrument. Designated laboratory personnel are trained in routine maintenance procedures for all major instrumentation. When repairs are necessary, the equipment shall be taken out of service. A tag will be posted and clearly identify the instrument is out of order. The repairs performed by either trained staff or trained service engineers, and an evaluation of the impact on previous calibrations or tests performed. The detailed logs for each instrument documenting the preventive maintenance and repairs performed are maintained.

3.0 Instrument Maintenance

3.1 All maintenance procedures are performed by the chemist/technician using the particular instrument.

3.2 The individual chemist/technician is responsible for reporting any damage or improperly functioning instrument to the Section Manager or Laboratory Director. The Section Manager or Laboratory Director will assign a chemist/technician to repair the instrument. If in-house personnel cannot repair the instrument, Laboratory Director will contact the service company to perform the repair. All maintenance work will be recorded in the designated instrument maintenance logbook.

3.3 Laboratory has a contingency plan in place in case of an instrument shut down. Designated backup instrument for each type of analysis should be in operation or on standby mode to continue with the analysis. Backup equipment must meet all the quality control criteria and must be routinely checked for the operational status if they are on standby mode. The contingency plan is designed to provide backup for all methods practiced in the lab. It is the responsibility of the Section Manager of the Laboratory to ensure that the preventive maintenance schedules are followed and that all equipment is functioning properly.

3.4 The instrument that has been subjected to overloading or mishandling, gives suspect results, or has been shown to be defective or outside specified limits, shall be taken out of service. It shall be isolated to prevent its use or clearly labeled or marked as being out-of service, until it has been repaired and shown by calibration or test to perform correctly.

3.5 For complete details on maintenance procedures, the chemist/technician should refer to the designated instrument/equipment manual for instructions. These manuals are located in each laboratory area.

3.6 Instrument maintenance Procedures:

- **ICP 61/61E and ICP/MS X-series**

  Daily

  - Check the pump tubing and sample introduction line. Adjust for smooth flow. Replace when needed.
  - Check the waste bottle and drain line system. Drain when half full.
  - Check nitrogen level. (for Purge instrument only)
  - Clean autosampler tray and rails.
  - Check the condition of torch glassware and replace, if needed.
  - Check the injector tube for wear or deposits and replace, if needed.
  - Reassemble the torch and check out system.

  Weekly

  - Check the gas lines and fittings for leaks.
  - Check cones and O rings (ICP/MS)

Quarterly

- Clean the inlet filter to the spectrometer.
- Clean instrument and area.
- Check the file system integrity. Delete and destroy unneeded files. (ICP only)
- Perform instrument detection limit study.
- Check air filter (RF generator, Chillers)
- Check IEC tables.

Yearly

- Replace vacuum pump oil.
- Replace air filters.
- Perform interelement correction study.
- Drain and replace chiller water

- **Leeman Labs Hydra AA Automated Mercury Analyzer**

Daily

- Check pump tubing replace when needed.
- Check drying tube
- Check waste bottle and drain line system
- Drain waste bottle when 3/4 full.
- Check gas/liquid separator, clean when needed.

Weekly

- Check optics, clean when needed.

Quarterly

- Replace drying tube.

- **Conductivity Meter- YSI 3100**

  Each Use

  - Rinse probe several times with DI water.
  - Calibrate instrument with a standard.
  - Perform final rinse of probe using DI water (store probe in DI water bottle)
  - Complete the logbook.

  Monthly

  - Clean cells using a solution of 100ml isopropyl alcohol, 100ml ethyl ether, 50ml concentrated HC1, and 50ml distilled water.

  Quarterly

  - Inspect platinum black cell coating for thinning and/or flaking.

- **Infrared Spectrophotometer- Buck Scientific 404**

  Each use

  - Check the zero setting and adjust, if necessary.
  - Inspect lenses and light path for dust or streaking

- **Spectronic 20D Spectrophotometer**

  Each Use

  - Rinse sample tubes with DI water before and after use.
  - Calibrate with analytical standards.
  - Clean sample compartment of any spills immediately

  Quarterly

  - Check the absorbance to transmittance calibration. Adjust, if necessary.
  - Check the lamp alignment. Adjust, if necessary.

Yearly

- Check wavelength calibration and photometric linearity with Cobalt Chloride solutions. See manual for procedure.

- **Tekmar Phoenix 8000 TOC**

Daily

- Check levels of persulfate and phosphoric acid solutions. Rinse and add fresh DI water to reservoir.
- Check and drain mist trap if necessary.
- Check level of water in gas/liquid separator and fill to waste outlet level if necessary.
- Prime reagent lines.
- Check that nitrogen flow into and out of the detector is 200 +/- 10 cc/min. Adjust at gas tank if necessary.
- Check Sn/Cu scrubber. Repack if Cu is dull or granules are clumped.
- Clean the sample boat (soils) and add clean quartz wool.
- Check the catalyst (soil). Replace if there is excess acid residue
- Check the oxygen and nitrogen tanks and replace if below 250psi.

Monthly

- Check that all 8-port valve thumb screws are finger tight.
- Run the cleaning cycle for the sparger and reaction chamber.
- Check permeation drier(s) for excess water vapor.

- **pH Meter - Acumet**

Each use

- Calibrate, using at least two buffers.
- Rinse electrodes with water before and after use.
- Check level of filling solution and add if necessary.

- Allow electrodes to set in a neutralized buffer solution between use.

Weekly

- Change the storage solution.

Monthly

- Change the electrode filling solution.

- **Analytical Balance/Top Loader**

Daily

- Complete the required weight calibrations and record values in logbook.

- Refer to SOP entitled "Balance Calibration Maintenance, and Use".

Each Use

- Clean the sample holder using the brushes located adjacent to the balances.

- Adjust the zero setting.

- **Gas Chromatograph - HP 5890 Series/ Agilent 6890N**

Daily

- Change the septa.

- Replace liners, glass wool and ferules.

- Check the carrier gas pressure and replace, if falls below 250psi.

- Maintain optimum level of solvents for the auto injector.

As Needed

- Break off a short portion (about one meter) of the column from the end near the injector.

- Replace the GC column.

- Check the flow rate.

- Bake out the electron capture detector at 310°C for 4 hours.
- Change the seal for the injector port.
- Clean or replace syringe.
- Bake out GC oven temperature at 300°C for 4 hours.
- Bake out injector port at 265° for 4 hours.

Note: Detectors are leak tested semi-annually by independent vendor.

- **GC/MS - HP5970 Series / 5972 Series**

<u>As Needed</u>

- Clean ion source.
- Replace filament.
- Clean or replace jet separator.
- Change gas cylinders.
- Replace the injection liner.
- Change pump fluid twice a year.
- Replace electron multiplier.
- Tune Mass Spectrometer.
- Clean or replace syringe.
- Fill solvent vial.

- **Tekmar - LSC2000/LSC3000**

<u>As Needed</u>

- Rinse positions thoroughly.
- Purge and bake each position for approximately 8 minutes as needed.
- Bake trap at 240°C for about 10 minutes. Change the trap on an as needed basis.

- Change purge valves, lines, and sparger needles as needed.

- **HPLC - Waters/ HP 1100**

  Daily

  - Check the pressure of the pump (volumes vary with analyses).
  - Wash the system with methanol for 10-15 minutes (whenever is necessary)
  - Check the waste container.

  Monthly

  - Change the pre-column filter (μ-Bondapak C18, Guard-Pak) as needed.
  - Wash the system with MeOH for 2-3 hours.
  - Replace the pump seals (if necessary).

  Semi-yearly

  - Back flush the column using methanol.
  - Replace the column (if poor response is observed).

- **IC - Dionex 500/600**

  Daily

  - Check the gas pressure (~120psi).
  - Sparge the eluents.
  - Wash the ATC with 190μM NaOH solution for 10-15 minutes. (for method UT03/LT03 only)
  - Pump working eluent through the system for 20-30 minutes before the analysis.
  - Check eluent level to ensure proper volume to complete schedule.
  - Check the waste container (empty the container for disposal).

As needed

- Prepare new acid regen.solution
- Reverse flow wash the ATC with 190mm NaOH.
- Clean columns with 190mm NaOH flow for 10-15 minutes.
- Change the column (if necessary).

4.0 Documentation

4.1 Records (Instrument Maintenance Logbooks) are maintained for each major item of equipment. These records include documentation on all routine and non-routine maintenance activities.

4.2 The Instrument Maintenance Logbook includes:

- The name of the item of equipment
- The manufacturer's name, type identification and serial number or other unique identification
- Date received and date placed in service (if available)
- Current location, where appropriate
- Copy of the manufacturer's instructions, where available
- Details of maintenance carried out to date

UNCONTROLLED DOCUMENT
AND
BUSINESS CONFIDENTIAL



SOP No: G.12

Title: Standard Operating Procedures for Submitting Reports

Scope: This standard operating procedure describes the procedures to be used by reports generation personnel in the generation of sample data reports and electronic data deliverables (EDD) to be sent out to clients.

1.0 WORKORDER RECEIPT, CHECKING AND FILING

1.1 Upon delivery of samples, the coolers are unpacked by the Sample Receiving personnel and the samples are checked against the chain of custody for discrepancies.

1.2 The samples are logged into the LIMS system and a work order is created. The work order is then forwarded by Sample Receiving to the Project Manager for approval. Once the workorder is approved, it is forwarded to the reporting generation department who then assembles a work order file consisting of a work order with fraction test list, the chain-of-custody form, the subcontracted chain-of-custody form (if applicable), the sample receipt form and the shipping label, if applicable.

2.0 DATA ENTRY INTO LIMS

2.1 All data acquired from sample analysis for each Workorder as per required test or Job must be entered into the LIMS database either manually or 99.9% of sample analysis is acquired automatically with XENCO.

2.1.1 Organics and Inorganics analyses

Results for all analyses are exported to the LIMS database by the analyst or supervisor in the laboratory. The exporting of data to the LIMS is accomplished through use of various software.

2.1.2 Subcontracted Analysis

The subcontracted analysis report must have the following parameter fields; analyte, result (concentration), analyzed date, preparation date (if required), detection limit and units. If any information is missing, the subcontracted lab has to be contacted to obtain the required parameters.

## 3.0 GENERATION OF LIMS REPORT

Data that has been exported into the LIMS data entry system database can be used to generate a LIMS report which can then be used for different reporting purposes.

### 3.1 Reporting Options

To generate a LIMS report using GPL LRD, first open GPL LRD by double clicking the GPL LRD icon on desktop. Choose the "STATUS" tab from the list of 5 tabs across top of screen by clicking on it once. Enter GPL work order number in box "SDG". Upon entering work order number click the "GO>" button once, which is located directly right of the GPL work order number. After clicking the "GO>" button a list of batches in LRD will appear. Once the batches in LRD appear, click the "Full Summary" box on the right hand side of screen one time. After the "Full Summary" box has been clicked, the LIMS report will be generated and appear on user's screen. Review data on screen by clicking arrow buttons back and forth to different pages. Print report by pressing print button on menu bar.

## 4.0 REPORTING DATA TO THE CLIENT

After data has been entered and a LIMS report generated and checked, the data is ready to be reported to the client. Data can be reported in various fomats depending on the clients needs. The client may opt for faxed or e-mailed results, complete hard copy results, CD's, or all 4 options.

### 4.1 Faxed or E-mailed Results

4.1.1 Clients often request to have their sample results faxed or e-mailed by a specific date. The date that the results are due depends on the turn around time requested by the client and can be found on the work order sheet. The data required for fax or e-mail is sample results only.

4.1.2 The LIMS report is faxed or e-mailed in its entirety or partiality as requested by the client. When faxing, each work order is logged into the fax logbook which can be found next the fax machine. The date, time, client, contact, fax number and any comments is logged into the fax logbook. The above information is taken from the client information section of the work order sheet. After the results have been faxed, the confirmation form from the fax is placed in the work order file.

### 4.2 Packages (when required)

4.2.1 Depending on the report type requested by the client, a completed hardcopy and/or CD is sent to the client on the due date as determined by the contract turn around time.

4.2.2 After the completed package from each department is submitted to the report generation department, its status is updated on the daily status reports from missing to complete. When packages for work orders that do not require faxing get turned in to the reports generation as complete, their status is noted as complete in the daily status report.

4.2.3 All packages that are ready to be sent to the client are paginated and copied. The client gets the copies, the number of which depends on the contract or client request. Different clients get different types of GPL report packages. The different types of report packages are GPL Level I, II, III and IV packages. All completed packages are scanned in its entirety and a CD is burned and stored in the Reporting Dept.

4.3 Data package Verification

4.3.1 When a department turns in a completed hardcopy package, it must have been checked for completeness and accuracy by the preparer (analyst) and that department's supervisor or designate. Both personnel must sign on the case narrative and conformance summary report.

4.3.2 When the package gets to reports generation, the technician that prepares the work order package and sends it out to the client also checks for the completeness of the package and, when completed, a data package check list must be signed.

4.3.3 A complete report package will always include:

4.3.4.1 A cover page

- Signatures by the Laboratory Director and Project Manger or designees, with the printed title.
- The address and phone number of GPL Laboratories, LLLP.
- The client's name, project name and project manager name, which appears on the cover page.
- The date the report was issued.

4.3.4.2 A copy of the chain-of-custody, which includes the date and time of sample collection, preservations, sample analysis requested and date and time of the sample receipt. The shipping label is included also.

4.3.4.3 A sample receipt checklist that is used to document the condition of the samples upon arrival at the laboratory.

4.3.4.4 An Analytical Summary Report that includes the reporting limits, test results, methods, date and time of sample collection, analysis and preparation for each sample. The Report also contains the work order number, fraction numbers, and the client's identification for each sample.

4.3.4.5 A case narrative that details the condition of the samples upon arrival at the laboratory. The case narrative also documents any quality control failures or deviations from the methods cited.

4.3.4.6 A definition page that provides a key for the notes used in the analytical summary report.

4.3.4.7 An original copy of all subcontracted data and supporting documentation (if applicable).

4.3.4.8 For the non-NELAP accredited analysis, the report will be flagged and the information will be stated in the case narrative

4.3.5 After the package has been checked for completeness, it is assembled according to the package type as designated on the work order sheet. It is then paginated. A table of content sheet is generated according to the content of the package. The final package is copied and the copies packaged for shipping. The analytical summary report (including the Cover Page), Chain-of-Custody, Sample Receipt Checklist, Qualifier page, and the Case Narrative will be paginated.

4.3.6 Prior to signature by the Project Manager, the entire data package is reviewed for: completeness, confirmation that correct data package was presented, and that the correct mailing address was used. The Case Narrative is also reviewed, edited and signed by the Project Manager and Lab Director. The project manager then returns the completed package to the reporting department for corrections, if necessary, and the completed package is sent to the client.

4.3.7 After the report has been sent, the sent date and the mailing method, e.g., Federal Express, UPS, Courier or mail is noted on the work order sheet.

## 5.0 GENERATION OF STATUS REPORT

5.1 The daily status report is generated each and every work day. It is used in the daily production meeting to track the status of all work orders in house.

## 6.0 GENERATION OF ELECTRONIC DATA DELIVERABLES (EDD/ADR)

Most clients will request to have their data submitted separately in an electronic format (EDD/ADR). The format of an EDD/ADR varies greatly from client to client and even within different projects for a single client. EDDs/ADRs are produced using customized programs and applications that query the GPL database and then reformat the data to suit individual client needs. The resulting EDD/ADR file is then submitted to the client via e-mail. The process has 3 steps:

- Locking the work order in the database
- Generating and reformatting the data, and
- Submitting the data to the client.

### 6.1 Locking the work order

6.1.1 This process is accomplished by opening an application that links to the GPL database and allows the user to enter the work order number to be locked. The user then selects a check box in the application and locks the work order. Locking a work order prevents any further modifications being made to the data by the lab. Only after locking occurs can data be generated for an EDD/ADR.

### 6.2 Generating and Reformatting the EDD/ADR

6.2.1 Most EDDs/ADRs are generated by querying the GPL database using Microsoft Excel and it's query feature. For each client the EDD/ADR analyst will prepare a separate customized generator based on the client's specified EDD/ADR requirements. The generator lays out the data fields and automatically converts certain data (for example dates) to the format required by the client within the framework of the Excel program. The data in the EDDADR is then reviewed against the hard copy report and any correction of data is performed. Once the data has been reviewed the EDD/ADR file can be saved either as an Excel file if that is the client's need or saved and converted to various other file formats directly from Excel or by exporting the data to Microsoft Access.

6.2.2 If a client requests an EDD/ADR but does not supply requirements, GPL has a standard format that can be used.

### 6.3 Archiving and Submitting the EDD/ADR

6.3.1 Completed EDD/ADR files are saved to a specified directory on GPL's server. Each client has a separate folder to hold that client's EDDs/ADRs. The files are stored in the EDD/ADR directory indefinitely and the folder is routinely backed up to tape.

6.3.2 Once the file(s) has been saved the ED/ADR analyst will generally then e-mail the EDD/ADR file(s) for the work order to that clients GPL project manager who will submit the EDD/ADR to the client as required. Other methods of delivery include direct e-mail, saving the file to the data package CD or direct transmittal of the EDD/ADR from the analyst to the client.

## 7.0 ARCHIVING COMPLETED WORKORDERS

Work orders that have been sent out, checked for completeness and the data package check list signed by the project manager, is ready to be archived. Completed work orders are archived either in-house or offsite.

### 7.1 In-house Archiving

7.1.1 Work orders that are ready to be archived are filed in a file cabinet in the reports generation section by month. As space fills up work order files from earlier months are removed to make room for work order files from recent months. As workorders from earlier months are removed, placed in archived boxes and the boxes are given a number in sequence, generated from a list that details work orders and their box numbers. This list is maintained conservatively. As the boxes are filled up with workorders and given a number, they are moved into an in-house storage space designated for the storage of archived work orders. The in-house storage area is kept locked and accessible only to authorized personnel.

### 7.2 Retrieving Work Orders from in-house Archive Location

7.2.1 When a file is stored in-house and is later needed by a Project Manager, the Project Manager must sign out the report and/or CD on the sign out sheet, which is located in the reporting department table next to the storage area. When the report and/or CD is returned back to the storage area, it is then signed back in on the sign in sheet. The storage of CD's are kept in a secure area and only accessible to authorized personnel.

### 7.3 Offsite Archiving

7.3.1 When the space designated for in-house storage becomes full, older archived boxes are moved to an offsite location. A list is maintained that catalogs the date that the boxes were moved to an offsite location (Iron Mountain), the boxes that were moved and the corresponding box number that were designated for each box by the offsite warehouse.

7.4 Retrieving Work Orders from Offsite Archive Location

7.4.1 Archived work orders from the offsite location can be requested to be sent back to the lab if needed. To do this a call is placed to the offsite location (Iron Mountain). The operator will request that the caller provide them with our account number, password, the box the desired work order is located in and it's corresponding offsite number. When the work order is no longer needed, it is returned to the warehouse.

7.5 Revisions, Additions or Deletions to Previously Issued Reports of Analysis

7.5.1 Any changes made to a final Report must be documented using "Report of Analysis Revision/Addition/Deletion Form"(Fig 1). Copies of the form are available from the Reporting Department supervisor. The form will be completed by the person requesting a revision to a previously issued Report of Analyses. The appropriate section of the form will be checked and the reason for the revision, addition or deletion will be described in detail.

7.5.2 The completed Form will be returned to the Reporting Department along with any supporting documentation (i.e., copies of logbooks, chromatograms, etc.). The requested changes will be made to the Report of Analysis. The entire data package, including the completed "Report of Analysis Revision/Addition/Deletion Form' will be given to the Lab Director for review. If the Lab Director approves the changes, he will sign on the indicated line of the form.

7.5.3 When the data package and signed Form is returned to the Reporting Department, they will send the revised Report of Analysis to the client along with any required supplemental documentation. The original form and all supplementary documentation will be archived with original data package.

Fig 1

***GPL Laboratories, LLLP***

## Report of Analysis Revision/ Addition/ Deletion Form

Work Order#: _____

Client: _____

**REVISION** Date of Revision: _____

Reason for Revision: _____

**ADDITION:** Date of Addition: _____

Reason for Addition: _____

**DELETION:** Date of Deletion: _____

Reason for Deletion: _____

Revision / Addition / Deletion Approved by:

Project Manager: _____ Lab Director: _____

SOP No: G.22

Title: General laboratory Multi Incremental Sampling (MIS) sub-sampling procedure.

Scope: This SOP addresses the laboratory's procedures for drying, particle size reduction, and sub-sampling of multi-incremental soil samples for metals or/and explosives samples.

## 1.0 METHOD SUMMARY

1.1 The purpose of this Standard Operating Procedure is to describe in detail the methodology and protocol used in obtaining representative environmental samples. It describes in detail sample handling preparing, drying, sieving grinding, and pulverizing. It also describes the procedure for sub-sampling of the processed samples.

## 2.0 REFERENCES

- SW846 Method 8330B revision 2 October 2006.
- EPA/600/R-03/027, Guidance for Obtaining Representative Laboratory Analytical Subsamples from particulate Laboratory Samples, November 2003.
- ASTM D6323 Standard guide for laboratory for laboratory subsampling of Media Related to Waste Management Activities.

## 3.0 INTERFERENCES

3.1 Sample processing hardware must be clean to minimize interferences.

## 4.0 APPARATUS AND MATERIAL

- Puck Mill (LABTECHNICS Type LM1-P)
- Flying saucer bowls (Part #1658-000160)
- Puck-off-center hole.
- Bowl lid.
- Sieve appropriate sizes mesh (Brass, USA Std. Testing Sieve, ASTM E-11 specification.)
- Drying tray (Steel or Aluminum, 18" x 26", 1" deep)

- Tray rack (Rubbermaid model# 3320)
- Tray rack (Restaurant Product model# 511-012)
- Tongue Depressor (Wooden spatulas)
- Sub-Sampling 7" spoon .25 TSP/ 1.23 ml (Polystyrene, flat bottomed. Fisher part# 14-375-254.)
- Balance capable of weighing 10kg sample with 2g precision (Taylor TE22)
- Ottowa Sand.
- Plastic film.
- Tray liner paper (Lab Soaker VWR item #52857-120)

5.0 REAGENTS AND STANDARDS.

5.1 Reagents.

5.1.1 HPLC Grade Acetonitrile.

5.2 Laboratory Control Spike solutions.

5.2.1 Laboratory Control Spike solutions are obtained as single component standards for 14 compounds at varied concentrations (See appendix B). Typically 100-200ug/mL. The standards for the matrix spike solution must be from a separate vendor or a different lot than the standards used for the calibration solutions.

5.2.2 Nitroglycerine standard is obtained as a single component at a concentration 5.0 mg/Kg. (See appendix C).

5.2.3 PETN standard is obtained as a single component at a concentration 5.0 mg/Kg. (See appendix D)

5.2.4 3,5-Dinitroanoline d standard is obtained as a single component at a concentration 5.0 mg/Kg. (See appendix E)

5.2.5 These amputated standards are good for six months or the date assigned by the manufacturer.

5.2.6 All the above solutions must be refrigerated at 4°C (± 2°C) and stored in the dark.

6.0 SOIL AND SEDIMENTS SAMPLE PREPARATION

6.1 Samples are expected to arrive in plastic bags or other suitable containers, with an average weight of 1-4kg. Empty the entire sample onto a drying tray, lined with liner paper. If the sample is particularly wet, liner paper may not be suitable. In this case, plastic film will be used. Moist soil samples are broken up into small pieces with a gloved hand prior to drying. Air-dry the soil sample at room temperature being careful not to expose the samples to direct sunlight. Record the date and time. The drying tray rack must be located in a well-ventilated room,

with the ventilating system in operation.

6.2 To ensure that the drying process is completed, the sample weight is monitored and recorded in the MIS sample preparation logbook. The drying process is considered complete when the weight change over a period of at least ½ hour is <1%. Record the initial and subsequent results.

6.3 Pass the samples through a #10 (2mm) sieve. Any clumps and agglomerates of soil are broken up into small pieces with a gloved hand. In addition, mosses and other types of fine vegetation will be physically shredded while sieving to release entrapped fibrous residues. Remove the oversized fraction of the sample that was retained on the #10 (2mm) sieve. Weigh both fractions and record both weights in a logbook. The oversized fraction is retained with the original sample, but is not ground or extracted unless specified in the project requirements.

6.4 To prevent cross contamination of samples, each sieve must be cleaned thoroughly and dried before new sample is loaded. Refer to GPL SOP G.2 sections 7.3-7.5 for specific instructions

6.5 If required by the project, the fraction that did not pass through the sieve is processed to reduce the particle size to <2mm and added to the sample prior to grinding as follows. Vegetable matter is removed and processed in a laboratory blender until it will pass through the #10 sieve. Any non-vegetable matter, as well as any material that could not be reduced in the blender is shipped to Penniman and Brown in Baltimore, MD for pulverization. The returned sample is passed through the #10 sieve and added to the remainder of the sample. If any material remains that still will not pass through the #10 sieve, the client will be contacted for further instructions.

6.6 Samples are now ready for further preparation or sub-sampling, depending on specific project requirements.

6.7 If analysis requested are only for explosives, proceed to section 8.0.

7.0 Sample Splitting for Explosives and Metals Analyses

7.1 If independent (separate) samples containers are not collected for metals. The existing dried and sieved samples will sub-sampled for Metals analysis prior to sample grind. Five-gram sub-sample will be needed to perform Metals digestion and analysis. The five-gram sample will be sub-sampled in the following manner. Using the sub-sampler scoop, take about ¼ scoop sample from randomly chosen locations, by sampling the whole profile of the sample tray. Approximately 15-20 different sub-sampled scoops will be necessary to obtain the five-gram sub-sample. Repeat the process and collect a duplicate sample, should re-digestion is necessary. Record the exact weight of the sub-sample in the appropriate sample digestion logbook and digest the entire sample. Proceed with metal digestion as described in SOP H.21. The entire sub-sampled must be digested. The remainder of the sample (if explosive analysis by 8330B are necessary) will be processed further, using the grinding process outlined in section 8.0.

8.0 GRINDING SAMPLE FOR SOIL AND SEDIMENTS USING PUCK MILL.

8.1 To prepare samples for explosive analysis, place a portion of the sample (up to 500g) in the grinder bowl of the Puck Mill and grind the samples for 60 second. Empty the ground sample portion on to a clean lined tray. Repeat the process,

until the entire sample is ground. To prevent the Puck mill from warming up to the temperatures where some energetic compounds may be lost, allow a three minutes cool down time period between five 60 seconds grinding cycles. To demonstrate that the process is complete, pass a portion of ground sample and passed through a #200 sieve. If the sample portion does not pass through the #200 sieve, continue the process above, until the sample portion passes through.

8.2 To prevent cross contamination of samples, each bowl, puck, lid and sieve must be cleaned thoroughly and dried before new sample is loaded. Refer to GPL SOP G.2 sections 7.3-7.5 for specific instructions

9.0 SUB-SAMPLING OF SOIL AND SEDIMENTS SAMPLES FOR EXPLOSIVE ANALYSIS.

9.1 The soil or sediment sample (the fine powder) is emptied back onto clean drying tray, freshly lined with liner paper. Using a wooden spatula, evenly distribute the ground sample on the tray.

9.2 Sub-sampling is performed using a flat-bottomed, straight walled scoop.

9.3 A 10-gram sub-sample will be collected for subsequent sonication and analysis of explosives. The 10g sub-sample will be collected in the same manner that is disribed in section 7.1. A total of 30-40 sub-sampling increments will be necessary to obtain the 10g required for sonication and analysis. Record the exact weight of each sub-sample in the appropriate sample extraction logbook and sonicate the entire portion of the sub-sample.

10.0 QUALITY CONTROL.

10.1 To ensure that the process does not introduce target analytes or other interferences into the ground samples, each batch of samples will include one bottle containing 500g of Ottowa sand, which is dried, sieved, and ground with the same equipment and methodology as the client samples. This blank material is then sub-sampled in the same manner as the client samples to obtain the background matrix for the Method Blank.

10.2 If the contamination in the blank mentioned in section 10.1 is > ½ reporting limit, all samples associated with contaminated blank must be re-extracted and re-analyzed if sufficient sample exists or all samples associated with the contaminated blank must be qualified with " B " qualifier.

10.3 To demonstrate that the grinding process does not cause undue analyte loss, each batch of samples will include Laboratory Control Sample (LCS) spiked with target compound prior to grinding. The LCS will be comprise of 500g of Ottawa sand spiked as followed:

10.3.1 Using a syringe, spike 2.5 ml of the LCS spike solution (Appendix B), 0.5 ml of Nitroglycerine solution (Appendix C), 0.05 ml of PETN solution (Appendix D), 0.25 ml of 3,5-Dinitrotolune solution (Appendix E)

10.4 The LCS standard must meet criteria for recovery as described in the DoD QSM. If QC criteria specified in the DoD QSM are not met, all samples associated with LCS will be re-analyzed and the data will be flagged as prescribed in the DoD QSM.

10.5 To demonstrate that the process adequately homogenizes and accurately sub-

samples each multi-incremental sample, for every twenty-field samples processed under this SOP, one sample is sub-sampled in triplicate and analyzed. The triplicate analyses must yield a RSD of 20% or less. If the %RSD IS >20%, the sample will be re-digest/re-extract and re-analyzed if additional sample volume is available.

10.6 A separate sample is also sub-sampled in triplicate for MS/MSD or MD/MS extraction, as specified by the relevant analytical method. The MS/MSD are spiked after grinding at sample extraction level as specified by GPL SOP S.11. The percent recoveries of MS/MSD must meet the LCS limits. The MS/MSD analyses must yield a RPD of 20% or less. If the RPD exceeds 20%, corrective action as specified in the DoD QSM will be followed.

Appendix B
Explosives analytes

| ANALYTE | SPIKE level |
|---|---|
| | ug/Kg |
| 1,3,5-Trinitrobenzene | 100 |
| 1,3-Dinitrobenzene | 100 |
| 2,4,6-Trinitrotoluene | 100 |
| 2,4-Dinitrotoluene | 100 |
| 2,6-Dinitrotoluene | 100 |
| 2-Amino-4,6-dinitrotoluene | 100 |
| 4-Amino-2,6-dinitrotoluene | 100 |
| HMX | 200 |
| RDX | 200 |
| Tetryl | 200 |
| 2-Nitrotoluene | 200 |
| 3-Nitrotoluene | 200 |
| 4-Nitrotoluene | 200 |
| | |
| | |
| | |

| Appendix C | ug/kg |
|---|---|
| Nitroglycerin | 5000 |

| Appendix D | ug/kg |
|---|---|
| PETN | 5000 |

| Appendix E | ug/kg |
|---|---|
| 3,5-Dinitroaniline | 1000 |

| MIS Sample Preparation for GPL Analysis | |
|---|---|
| Work Order # : | Client: |

Note:

Equipment: 2.0mm Sieve (#10), Scale readable to 1 grams.

| | | Moisture Calculation for Drying Process | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| GPL Fraction | Mass (g) Tray & Paper | Wet Mass (g) Sample + Tray | 1 Dry Mass (g) Sample + Tray | 2 Dry Mass (g) Sample + Tray | 3 Dry Mass (g) Sample + Tray | 4 Dry Mass (g) Sample + Tray | 5 Dry Mass (g) Sample + Tray | Oversized Mass (g) Sample Only[1] | Sample Mass (g) Sample Only[2] |
| 1 | | | | | | | | | |
| 2 | | | | | | | | | |
| 3 | | | | | | | | | |
| 4 | | | | | | | | | |
| 5 | | | | | | | | | |
| 6 | | | | | | | | | |
| 7 | | | | | | | | | |
| 8 | | | | | | | | | |
| 9 | | | | | | | | | |
| 10 | | | | | | | | | |
| 11 | | | | | | | | | |
| 12 | | | | | | | | | |
| 13 | | | | | | | | | |
| 14 | | | | | | | | | |
| 15 | | | | | | | | | |
| 16 | | | | | | | | | |
| 17 | | | | | | | | | |
| 18 | | | | | | | | | |
| 19 | | | | | | | | | |
| 20 | | | | | | | | | |

mments:

Analyst: Date:

Supervisor: Date:

[1]Sample material which did not pass through a 10-mesh sieve

[2]Sample material which passed through a 10-mesh sieve

GPL LABORATORIES, LLLP

FORM# Q-10

# SOP REVIEW FORM

SOP NO: G22 VERSION NO: 23 DATE: 07/29/08

SOP TITLE: MIS sub-sampling procedure

REVIEWER: [illegible] DATE: 7/29/08

SUPERVISOR: _____ DATE: _____

CHANGES MADE: (Y OR N)

IF YES, NEW VERSION NO: 23 DATE: 07/29/08

PLEASE GIVE A BRIEF DESCRIPTION OF THE CHANGES:

- All Roller Mill procedures were removed
- Reagents and Standards section was added.
- LCS spike will be performed prior to grinding.

SOP CHANGES ACCEPTED AND APPROVED BY:

LABORATORY DIRECTOR: [signature] DATE 07/29/08

SOP No: S.11

Title: HPLC Analysis of Nitroaromatic and Nitramine Explosive Residues in Water, Soil, and Sediment Samples

Scope: This SOP describes the analytical methodology employed in the analysis of water, soil, and sediment extracts for explosive residues by Method 8330B

## 1.0 METHOD SUMMARY

1.1 The purpose of this Standard Operating Procedure is to describe in detail the methodology used in the analysis of water, soil, and sediment extracts for explosive residue compounds. The methodology conforms to that specified in SW846 method 8330A/8330. The compounds to be analyzed in this method are listed in Table I.

TABLE I

| COMPOUND NAME | CAS NO. |
|---|---|
| HMX | 2691-41-0 |
| 1,3,5-TNB | 99-35-4 |
| Tetryl | 479-45-8 |
| TNT | 118-96-7 |
| 4-A-2,6-DNT | 1946-51-0 |
| 2,6-DNT | 606-20-2 |
| 4-Nitrotoluene | 99-99-0 |
| RDX | 121-82-4 |
| 1,3-DNB | 99-65-0 |
| Nitrobenzene | 98-95-3 |
| 2-A-4,6-DNT | 355-72-78-2 |
| 2,4-DNT | 121-14-2 |
| 2-Nitrotoluene | 88-72-2 |
| 3-Nitrotoluene | 99-08-1 |
| 3,5-Dinitroaniline | 618-87-1 |
| PETN | 78-11-5 |
| Nitroglycerin | 55-63-0 |

2.0 REFERENCES

- SW846 Method 8330B revision 2 October 2006.
- SW846 Method 8330A revision 1 January 1998.
- SW846 Method 8330 revision 0 September 1994.

3.0 INTERFERENCES

3.1 Glassware and other sample processing hardware must be clean to minimize interferences.

4.0 APPARATUS AND MATERIAL

4.1 HPLC Instrument.

4.1.1 A Hewlett Packard HPLC system – HP1100 series.

4.1.2 A Waters Model 501 Solvent Delivery System (HPLC Pump), Waters WISP 712 Autosampler and Waters Variable Wavelength Detector Model #486 or LINEAR UVIS-201 absorbance detector is used as an analytical system complete with primary column (Phenomenex Ultracarb 5um ODS (30) 250x4.6mm or equivalent), confirmatory column (Phenomenex Synergi 4um POLAR-RP 250x4.6mm or equivalent) and pre-column HPLC inserts of corresponding stationary phase.

4.2 Other equipment.

- Temperature controlled ultrasonic path.
- Balance- Capable of weighing +\- 0.01g
- Magnetic stirrer with PTFE stirring bars.
- Vacuum pump (GE Motors/Industrial System).
- Filter holder Manifold
- Millipore-47mm Glass Filter Holder
- Chiller Model No 1156 VWR.

4.3 Material and supply.

- Disposable cartridge filters –0.45um PTFE filter.
- Pipets class A glass, appropriate sizes.
- Volumetric flask –10ml, 25ml, 100ml, and 1L, with ground glass stoppers.
- Glass Vials-40ml, 60ml.
- Gastight Syringes- 1ml, 500ul and 25ul.

- Mortar and pestle,
- Sieve 10 mesh.
- Class A TC and TD Graduate cylinders-1L and 10ml.
- Wide mouth flask-125ml.
- Disposable aluminum dish.
- Deposable aluminum weighing dish.
- Disposable pipettes.
- Disposable filter device-Whatman brand.
- Depressor Tongue (Wooden spatulas)
- Fisher magnetic stir bar
- Amber Vials-10ml, 20ml
- Fisher brand Sodium chloride
- Fisher brand Acetonitrile HPLC grade.
- Sand - Fisher brand.

5.0 OPERATING PARAMETERS

- Mobile phase: 50% HPLC grade Methanol / 50% deionized H2O for primary. 60% $H_20$, 5% MeOH and 35% Acetonitrile for confirmation.
- Flow rate: 0.9ml/min for primary and 1.0ml/min for confirmation analysis.
- Injection volume: 100ul fixed loop.
- Wavelengths: 210nm and 254nm for primary and confirmation.

6.0 REAGENTS AND STANDARDS

6.1 Reagents.

- HPLC Grade methanol and Acetonitrile
- Calcium Chloride Desiccant (High Purity)
- Standards can be obtained as an ampule from Absolute, Restek, or other suitable vendor.
- Sodium Chloride, NaCl, Reagent Grade
- Tetrabutylammonium dihydrogen phosphate, 97%

6.2 Standards.

6.2.1. Source Standards.

6.2.1.1 Source standard solutions may be obtained as single component standards or as mixtures at concentrations suitable for preparation of the intermediate standard, typically 1000-5000ug/mL. The source standards may be retained until the expiration date provided by the vendor.

6.2.1.2 The source standards should be stored refrigerated at 4°C (± 2°C) in the dark.

6.2.2 Intermediate mix.

6.2.2.1 The intermediate mix is prepared by combining the necessary aliquots of the source standard(s) in a volumetric flask and dilution with Acetonitrile to volume. The final concentration of the Intermediate Standard should be 20ug/mL for HMX, RDX, Tetryl, 2-NT, 3-NT, 4-NT, 3,5-DNA, and 1,2-DNB (surrogate) and 10ug/mL for 1,3,5-TNB, 1,3-DNB, NB, 2,4,6-TNT, 2-A-4,6-DNT, 4-A-2,6-DNT, 2,4-DNT, and 2,6-DNT. NG and PETN are not added to the Intermediate, since separate calibration curves are used for them. A separate PETN intermediate is prepared at 50ug/mL in Acetonitrile. A NG intermediate may be prepared if necessary, or the individual calibration points for the NG curve may be prepared directly from the source solution. The intermediate standards are discarded after 30 days.

6.2.2.2 Intermediate standard should be refrigerated at 4°C (± 2°C) in the dark.

6.2.3 Working mix.

6.2.3.1 A Working mix is prepared as needed by adding 25uL of the intermediate standard (Section 6.2.2.1) to mix of 500uL calcium chloride and 475ul Acetonitrile to yield a solution with a concentration of 0.25 and 0.50ug/mL. In the same way, a PETN working solution is prepared as needed with a concentration of 1.25ug/mL.

6.2.3.2 The working standards are not stored, but prepared fresh as needed.

6.2.4 Surrogate spike solution.

6.2.4.1 A 1,2-Dinitrobenzene source solution is obtained at a nominal concentration of 1000ug/mL.

6.2.4.2 The surrogate spike solution is prepared to a nominal concentration of 6.0ug/mL by placing an aliquot of the source solution in a volumetric flask and dilution to volume with Methanol.

6.2.4.3 The surrogate spike solution is good for 30 days.

6.2.4.4 1mL of the Surrogate spike solution is spiked to all samples, Method Blank, MS/MSD's and LCS's prior to extraction.

6.2.4.5 The surrogate solutions must be stored refrigerated in the dark at 4°C (± 2°C).

6.2.5 Matrix spike solutions.

6.2.5.1 Matrix spike source solutions may be obtained as single component standards or as mixtures at concentrations suitable for preparation of the intermediate standard, typically 1000-5000ug/mL. Standards for the matrix spike solution must be from a separate vendor or a different lot than the standards used for the calibration solutions. The source standards may be retained until the expiration date provided by the vendor.

6.2.5.2 The matrix spike solution is prepared by placing aliquots of the source solutions in a volumetric flask and diluting to volume with Acetonitrile. All analytes except NG should be present at 5ug/mL in the final solution. A separate NG matrix spike solution is prepared in Acetonitrile at a concentration of 100ug/mL.

6.2.5.3 These solutions are good for 30 days from the time of preparation.

6.2.5.4 1mL of the matrix spike solution is added to the LCS and MS / MSD samples prior to extraction. 1mL of the NG matrix spike solution is added to a separate LCS, MS, and MSD prior to extraction.

6.2.5.5 All the above solutions must be refrigerated at 4°C (± 2°C) and stored in the dark

6.2.6 Second source standard.

6.2.6.1 The matrix spike solution and NG matrix spike solution may be used to prepare a second source standard, which is used to verify each calibration curve prior to use of the curve for sample analysis. This standard is prepared as needed and used immediately.

## 7.0 SAMPLE COLLECTION, HANDLING AND HOLDING TIMES

7.1 Water samples may be collected in 1L or (quart) amber glass container. Soil samples may be collected in glass containers or closed end tubes.

7.2 All samples must be iced or refrigerated at 4°C (± 2°C) from the time of collection until extraction.

7.3 Extraction holding times for water is seven days from the date sampled and fourteen days from the date sampled for soil. Analysis holding time is 40 days from the date of extraction for both soil and water samples.

## 8.0 CALIBRATION AND STANDARDIZATION

8.1 For primary and confirmation analyses, calibration is performed for sixteen analytes and the surrogate with the concentrations as listed on Table II-A. Table II-B lists the standards used to prepare the calibration curve. NG is calibrated separately using the concentrations listed on table III.

TABLE II-A

| COMPOUNDS | 1st LEVEL (ppb) | 2nd LEVEL (ppb) | 3rd LEVEL (ppb) | 4th LEVEL (ppb) | 5th LEVEL (ppb) | 6th LEVEL (ppb) |
|---|---|---|---|---|---|---|
| HMX | 20 | 100 | 500 | 1000 | 2000 | 5000 |
| RDX | 20 | 100 | 500 | 1000 | 2000 | 5000 |
| Tetryl | 20 | 100 | 500 | 1000 | 2000 | 5000 |
| 3,5-DNA | 20 | 100 | 500 | 1000 | 2000 | 5000 |
| 1,3,5-TNB | 10 | 50 | 250 | 500 | 1000 | 2500 |
| TNT | 10 | 50 | 250 | 500 | 1000 | 2500 |
| 4-A-2,6-DNT | 10 | 50 | 250 | 500 | 1000 | 2500 |
| 2,6-DNT | 10 | 50 | 250 | 500 | 1000 | 2500 |
| 4-NT | 20 | 100 | 500 | 1000 | 2000 | 5000 |
| 1,3-DNB | 10 | 50 | 250 | 500 | 1000 | 2500 |
| Nitrobenzene | 10 | 50 | 250 | 500 | 1000 | 2500 |
| 2-A-4,6-DNT | 10 | 50 | 250 | 500 | 1000 | 2500 |
| 2,4-DNT | 10 | 50 | 250 | 500 | 1000 | 2500 |
| 2-NT | 20 | 100 | 500 | 1000 | 2000 | 5000 |
| 3-NT | 20 | 100 | 500 | 1000 | 2000 | 5000 |
| PETN | 50 | 250 | 1250 | 2500 | 5000 | 12500 |
| 1,2-DNB (surrogate) | 20 | 100 | 500 | 1000 | 2000 | 5000 |

TABLE II-B

| | ACN | CaCl | Intermed. Standard | Working Standard | Final Conc. (ug/L) |
|---|---|---|---|---|---|
| Level-1 | 460uL | 500uL | 0 | 40uL | 10/20/50 |
| Level-2 | 300uL | 500uL | 0 | 200uL | 50/100/250 |
| Level-3 | 475uL | 500uL | 25uL | 0 | 250/500/1250 |
| Level-4 | 450uL | 500uL | 50uL | 0 | 500/1000/2500 |
| Level-5 | 400uL | 500uL | 100uL | 0 | 1000/2000/5000 |
| Level-6 | 250uL | 500uL | 250uL | 0 | 2500/5000/12500 |

TABLE III

| COMPOUNDS | 1st LEVEL (ppm) | 2nd LEVEL (ppm) | 3rd LEVEL (ppm) | 4th LEVEL (ppm) | 5th LEVEL (ppm) |
|---|---|---|---|---|---|
| Nitroglycerine | 1 | 5 | 10 | 20 | 50 |

8.2 Initial Calibration is performed by analyzing six calibration levels (five levels for Nitroglycerin). PETN calibration is performed with the detector set to 210nm. All other analytes use 254nm. Percent RSD (Relative Standard Deviation) must be below 20%. Calculate response factor (RF) for each level of standard using peak area. Peak heights will be used to calculate the response factors if it is required by specific project.

$$\%RSD = \frac{\text{Standard Deviation}}{\text{Average RF}} \times 100$$

$$RF(\text{Each Level}) = \frac{\text{Peak Area/Height of Analyte}}{\text{Concentration}}$$

8.2.1 Prior to use for sample analysis, the acceptability of the initial calibration curve must be verified through analysis of calibration verification (ICV) solutions obtained from a second source. Calibration verification analysis should meet the same acceptance criteria used for daily calibration.

8.3 Continuing Calibration:

8.3.1 A continuing calibration is performed at the beginning of each shift by analyzing a mid-level standard. The calibration difference must be below 15%. When a Continuing Calibration is passed, the calibration is acceptable for a 12-hour period starting from the beginning of the injection of the standard.

8.3.2 Mid-point calibration standard is run every ten samples and a closing standard is run at the end of the batch or 24-hour period, whichever comes first. Each mid-point and closing standard must have a %Difference below 15.

$$\%\ \text{Difference} = \frac{RFc - RF_i}{RF_i} \times 100$$

where:

$RF_i$ = Mean response factor from the most recent initial calibration

$RF_c$ = Response factor from continuing calibration

8.3.3 If the instrument does not meet the acceptance criteria, a new initial calibration must be performed.

8.3.4 A new initial calibration is also required if a column is replaced or other major instrument maintenance is performed.

8.3.5 Minor corrective action like changing pre-column filter or washing column and lines with pure methanol may not require performing a new initial calibration, provided the daily calibration that originally failed, now passes the acceptance criteria.

9.0 METHOD DETECTION LIMIT

9.1 Method detection limits for this method are listed in the GPL Laboratory Method Detection Limit and Reporting Limit table.

10.0 METHOD PERFORMANCE

10.1 The MDL concentrations listed in the GPL MDL table are generally obtained using organic-free reagent water. Results were also obtained by extracting seven spiked replicates the same way as the samples and analyzing them. 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. Precision and accuracy studies are performed once a year at a minimum. Single operator precision, overall precision and method accuracy were found to be directly related to the concentration of the parameter.

11.0 PREPARATION PROCEDURE

The appropriate sample preparation techniques must be used according to the project requirement: (Figure 1)

11.1 Aqueous Samples – (salting-out extraction)

11.1.1 Place 770ml aliquots of the aqueous sample, blank and blank matrix spike, in 1000ml erlenmeyer flasks. Add 1.00ml of 6ppm of the surrogate solution (section 6.2.4.3) to each sample and 1.0ml of matrix spike solution (Section 6.2.5.3) to each LCS and sample matrix spike. Matrix spike solution is prepared in methanol at 5.0ug/ml concentration using all target compounds listed in Table I.

11.1.2 Spike samples should be allowed to equilibrate for at least one hour prior to processing of the sample.

11.1.3 Add 251.3g of NaCl plus a magnetic stir bar in to the flask and stir the sample using a magnetic stirrer, starting from medium to high speed until all of the NaCl is completely dissolved. Be sure to dissolve all salt before adding acetonitrile, or the dissolution process takes much longer.

11.1.4 Using a graduated cylinder, add a164mL of Acetonitrile to each sample and stir on high speed for 20 minutes. Let the phases separate for about 10 minutes.

11.1.5 Collect the upper layer (Acetonitrile) in a 100ml erlenmeyer flask. Approximately 10ml should be collected.

11.1.6 Add another 10ml of acetonitrile into the 1000ml erlenmeyer flask containers sample and stir on high speed for 15 minutes. Allow the phases to separate for about 10 min. Collect the upper layer and combine with the first extract in the 100ml volumetric flask.

11.1.7 Prepare a salt water solution in a separate flask by dissolving 325g of NaCl in 1000ml of D.I. water. Add 84ml of the salt water solution to the extract, which was collected in a 125ml erlenmeyer flask. Place the 125ml erlenmeyer flask on the magnetic stirrer with a magnetic bar and stir on high speed for 15 min. Allow the separation of the layers. Collect the top layer (Acetonitrile), extract one more time with an additional 1ml of acetonitrile, adding this to the first extract and adjust the volume to 10ml using Class A graduated cylinder or volumetric flask.

11.1.8 Before analysis dilute the sample 1:1 with DI water, (with pH<3 if tetryl is a suspected analyte) prior to analysis.

11.2 Aqueous Samples – (solid-phase extraction)

The Cartridges or Disks extraction media type will be used according to the project requirement:

11.2.1 Cartridge procedure:

11.2.1.1 Prepare the material and equipment to be used like plastic tubings strata SPE phase: SDBL 500mg/6ml cartridges as specified, florisil clean-up apparatus, aluminum foils, 10ml pipets, 20ml scintillating vials, and syringes. Precondition strata SPE phase: SDBL 500mg/6ml cartridge by running them with 5ml MeOH (methanol) and 15ml DI water.

11.2.1.2 To the original sample containers, prior to transfer, Add 5ml of MeOH and spike with 100ul of 6ug/ml surrogate for blank, samples and QC samples. Also add 50ul of 5ul/ml (Section 6.2.5.3.) explosive mix standard to the LCS and MS/MSDs.

11.2.1.3 Spike sample should be allowed to equilibrate for at least one hour prior to processing the sample.

11.2.1.4 Measure out the entire sample volume provided using clean class A TC graduate cylinder. If more than one-liter sample is provided, use second graduate cylinder to measure the sample volume, QC and samples. Record the sample volume in the extraction log book provided.

11.2.1.5 Prepare the extraction apparatus by installing the plastic tubing in cartridge and priming with the last 5ml DI water and proceed the extraction procedure by increasing the vacuum pressure and proceed carefully, gradually decreasing until all the samples are properly running at normal pressure which is about 5psi. Be careful not to spill

your samples, continue until all the samples are extracted and take note of the collecting flask, which may overflow due to excessive flow.

11.2.1.6 Wash the cartridge with 15ml DI water and thoroughly dry for 30-60 seconds. Wipe the underside of the cartridge apparatus with clean wipes to collect the excess water underneath.

11.2.1.7 Put the properly labeled scintillating vials underneath the cartridge corresponding to the sample numbers they are to be collected.

11.2.1.8 Rinse the sample bottles with 5ml of CH3CN (acetonitrile) and pipet or decant into the cartridge until all the CH3CN are collected. Be sure that the cartridge is in the stop position.

11.2.1.9 Start the vacuum pump until the CH3CN just begins to drip and close it, just gradually, opening slowly with the use of gravity, start the collection flow through the cartridge until all the CH3CN are visibly gone in the cartridge. Complete the collection process with drying by running the vacuum for 1 minute.

11.2.1.10 Blow down the collected extract of about 5ml to 0.5ml final volume in the nitrogen bubble bath, keeping the temperature below 30 degrees centigrade.

11.2.2 Disk procedure

11.2.2.1 Prepare the material and equipment to be used. Empore, C18 disks- 47-mm diameter, aluminum foils,10ml pipets, 20ml scintillating vials, and syringes.

11.2.2.2 Precondition Disk by pre-Wet disk with 15ml of CH3CN for three minutes. Allow not to dry.

11.2.2.3 To the original sample container, prior to transfer, add 5ml of MeOH and spike with 1mL of 6ug/ml surrogate solution (Section 6.2.4.3.) for blank, QC and samples. Also add 1mL of matrix spike solution (Section 6.2.5.3.) @ 5ug/mL) to the LCS and MS/MSDs. Spike samples should be allowed to equilibrate for at least one hour prior to the sample processing.

Measure out the entire sample volume provided using class A TC graduated cylinder. If more than one liter sample is provided, use second class A TC graduated cylinder to measure the sample volume. Record the volume into the sample log book.

11.2.2.4 Pour sample into the reservoir and under full vacuum,

filter it as quickly as the vacuum will allow. Rinse the bottle with DI water and filtered as previously stated.

11.2.2.5 After the sample has passed through the disk, dry the disk by maintaining vacuum for about 3 minutes. Turn of the vacuum and allow the disk to soak.

11.2.2.6 Soak the SPE Disk with 5ml of CH3CN for 3 minutes.

11.2.2.7 Collect eluted sample by vacuuming to a collection tube.

11.2.2.8 Adjust the sample collected in the collection tube to 10ml using class A graduated cylinder or volumetric flask.

11.3 Soil and Sediment Samples

11.3.1 Sample homogenization:

11.3.1.1 Samples are expected to arrive in plastic bags, with an average weight of 1-4 kg. Empty the entire contents of the plastic bag in an drying tray, lined with aluminum foil. Air dry the soil sample at room temperature for at least 24 hours, being careful not to expose the samples to direct sunlight. The drying tray rack should be located in a well ventilated room, like the sample management storage room area, with the ventilating system in operation.

11.3.1.2 After the drying process is complete (and that can be verified by conducting two succeeding weight readings of the same sample aliquot, with no substantial difference) the soil sample must be sieved using a 2-mm sieve. The sieving must be performed in accordance with the method 8330B requirements.

11.3.1.3 Visually inspect the sample to insure that no rocks, stones or twigs exist. If stones, rocks or sizeable twigs exist, they must be removed, prior to the sieving. Once the sieving process is completed, the material that has been retained by the sieve, is returned to the original sample plastic bag. This material will not be used in generating the sub-sample to be used for analysis.

11.3.1.4 Weigh the sample and record the results in the sample drying logbook. Continue to monitor the sample weight every half hour until constant weight is reached. The acceptance criterion is <± 1% variation in weight. Record the initial and subsequent results.

11.3.1.5 Homogenize the dried sample thoroughly, by grinding the material that was processed through the 2-mm sieve (10-mesh). The grinding process must be performed by taking the sieved portion of the sample and placing it in a 2 liter HDPE wide mouth bottle. If a lot of sample material exists, then a second or third bottle may be appropriate, for the

same sample. Insert 20-30 teflon-coated grinding cubes into the sample container. Place the sample container on the Rotation Instrument, and rotate the sample container for at least 3 hours, or until the final product is a fine, uniform soil powder. Once this is achieved, the grinding operation can be terminated. After the grinding process is completed, the contents of the sample container (the fine powder) must be emptied back onto the original drying tray, freshly lined with aluminum foil. Then using a disposable wooden spatula, the sample must be spread evenly on the drying rack. Only after this uniformity of the sample is obtained, then a sub-ample can be obtained to perform the sonication and analysis for explosive compounds.

11.3.1.6 To prevent cross contamination of samples, each sieve must be cleaned thoroughly and dried after every use.

11.3.1.7 After the soil sample is generated and collected, place a 10.0g sub sample of each soil sample in a 60ml glass vial. Add 19.0ml of acetonitrile and 1.0ml of 6ppm surrogate to each sample. Add 18.0ml of acetonitrile and 1ml of 8330 matrix spike, 1.0ml surrogate to each LCS and sample matrix spike.

11.3.1.8 Place samples in a cooled ultrasonic bath for 18 hours. After sonication, allow sample to settle for 30 minutes. Remove 5.0ml of supernatant, and combine with 5.0ml of calcium chloride solution before filtering through a 0.45um teflon filter. Allow samples to equilibrate for 15 minutes before analysis.

## 12.0 SAMPLE ANALYSIS

12.1 Sample analysis may begin when calibration is complete.

12.2 Mid-Point standards are run every ten samples in order to examine both the variation of retention time and to check the calibration of the instrument.

12.3 A dilution must be performed when the peak response exceeds the calibration range of the compounds.

12.4 Peak identification is based on the comparison of the retention times using both the primary and confirmatory columns. The width of the retention time window is calculated by determining the average retention time, average peak width, and retention time standard deviation for each analyte in three continuing calibration standards run within a 72 hour period. The RT window width is the greater of +/- three times the RT standard deviation or ½ of the average peak width for each analyte. The ½ average peak width value may not be used if this would result in a window that encompasses any other analytes. The center of each retention window is determined on an ongoing basis using the retention times measured for each continuing calibration.

12.5 Each sample extract is analyzed twice using the same analytical conditions; once with the detector set to 210nm for PETN analysis, and once with the detector set

to 254nm for the remainder of the analytes. Alternatively, one analysis may be used with a detector capable of monitoring 210 and 254nm simultaneously. Two calibration standards are used for the analysis at 254nm; one with NG only, and one with the remainder of the analytes. Since NG and TNB exhibit very similar retention times on the primary column, it is necessary to report results for these two analytes from the confirmation column if both are detected in the same sample.

## 13.0 SAMPLE QUANTITATION

The concentration of all target compounds should be calculated using the following equations.

13.1 Water Sample:

$$\text{Conc. (ug/L)} = \frac{\text{(Peak Area/Height of Sample)(Final Volume in ml)(Dilution)}}{\text{(Avg RF of Std.)(Initial Volume in ml)}}$$

13.2 Soil Sample:

$$\text{Conc. (ug/Kg)} = \frac{\text{(Peak Area/Height of Sample)(Final Volume in ml)(Dilution)}}{\text{(Avg RF of Std.)(Initial Wt. In gm)}}$$

## 14.0 QUALITY CONTROL

14.1 A method blank is extracted and analyzed with every batch of 20 samples or less. The level of target analyte contaminants in the blank must be less than one half of the reporting limit.

14.1.1 Purified sand should be used as a soil method blank. The soil sand blank needs to be processed after several environmental samples have been processed (e.g., after the mortar, pestle and sieve have been used to process at least one environmental sample and have been cleaned). Method blanks should come in contact with all equipment used to process the samples (e.g., the sieve, mortar, pestle and aluminum plate).

14.1.2 If the contamination in the blank is > ½ reporting limit, all samples associated with contaminated blank must be re-extracted and re-analyzed if sufficient sample exists and holding times have not elapsed.

14.1.3 Blank must be spiked with method specified surrogate. If the surrogate recovery in the method blank does not meet the acceptance criteria, first re-analyze the method blank. If the surrogate recovery does not meet the acceptance criteria after the re-analysis, the method blank and the sample associated with the blank must be re-extracted and re-analyzed.

14.2 Sample analysis

14.2.1 Sample must be extracted and analyzed within the method specified holding time.

14.2.2 The samples must have associated method blank meeting the blank

acceptance criteria.

14.2.3 Samples must be spiked with method specified surrogate. If the surrogate recovery does not meet the established acceptance criteria, first check calculation, sample preparation logs, and the instrument condition. If calculation was incorrect, correct the calculation and verify that the surrogate compound recovery meets the acceptance criteria. If the instrument malfunctioned, correct the instrument problem and re-analyze the sample extract.

14.2.4 If the above action does not correct the problem, re-extract and re-analyze the sample.

14.2.5 If the surrogate compound recovery meets the acceptance criteria in the re-extraction, submit the data from the re-extraction.

14.2.6 If the surrogate compound recovery fails to meet the acceptance criteria in the re-extracted samples, then the problem may be due to matrix effect. To determine if there was matrix effect, review the surrogate recoveries of blank, LCS & MS/MSD analyzed and extracted in the same batch.

14.3 Laboratory Control Sample

Laboratory Control Sample (LCS) is extracted and analyzed for every batch of 20 samples or less. The recovery limits are determined by taking three standard deviations of at least 30 consecutively analyzed LCS's. Data points used in the data set must not be selectively included or excluded.

The LCS is analyzed to assess general method performance. The LCS is spiked with all target analyses before it is carried through the sample preparation. For soil samples, a purified solid matrix (e.g., ottawa sand, sodium sulfate, or other purified solid) would typically be used. For aqueous analyses, use analyte-free reagent water. The concentration used to spike the LCS is the 3rd level of the initial calibration mix (see tables II ).

14.3.1 Each LCS must be evaluated against the Control Limits and Marginal Exceedance limit. If any LCS is outside the Control Limit, it should be also compared to the Laboratory Marginal Exceedance Limits to ensure that it does not exceed. If a single analyte in the LCS exceeded the Marginal Exceedance Limit, the LCS had failed and corrective action needs to be initiated.

14.3.2 If the LCS has more than the allowable number of Marginal Exceedance, the LCS has failed and corrective action needs to be initiated.

14.3.3 The corrective action for failed LCS is based on professional judgment in conjunction with matrix spike and surrogate recoveries in the same batch. If after checking the associated QC's it was determined by the section supervisor or manager that the LCS had failed, all affected samples associated with the out of control LCS should be reprocessed and re-analyzed.

14.3.4 If samples cannot be reprocessed due to lack of sample volume or the holding times has lapsed, the results should be reported with appropriate

flag. The report case narrative must include a discussion of the failed LCS, and its impact on the data quality.

14.3.5 When LCS is spiked with large number of analytes, the laboratory should add up total number of exceedances for the LCS based on the number of analyte spiked in the LCS. The total of exceedance should be compare with the allowable number from the following chart.

| Number of analyte in the LCS | Allowable Number of Marginal Exceedances of LCS |
|---|---|
| < 11 | 0 |
| 11-30 | 1 |
| 31-50 | 2 |
| 51-70 | 3 |
| 71-90 | 4 |
| >90 | 5 |

14.4 Matrix spike and duplicate analyses are performed per batch of 20 samples or less.

The MS/MSD is evaluated by comparing the precision of target analytes to the recovery windows established. MS/MSD data evaluation is more complex than method blank or LCS data since MS/MSD measure matrix effect in addition to sample preparation and analysis error. MS/MSD that fail to meet the acceptance criteria would indicate that a potential matrix effect is present. The laboratory must assess the batch to determine whether the spike results are attributable to matrix affect, or the result of another problem in the analytical process. If all the QC batch elements, which are not affected by the sample matrix, are in control (e.g., method blank, LCS), and if there is no evidence that the spiking was not properly performed, the poor spike recovery may be attributed to matrix effect. If the LCS compounds that are not affected by the sample matrix are out of control, and if the same compounds in the MS/MSD are outside control limit, then matrix spiked sample(s) must be re-processed through the entire analytical procedure.

For DOD projects use QSM LCS limits to evaluated the MS\MSD.

14.5 Confirmation for all target compounds detected on the Ultracarb column is performed on a Polar RP column. Analytes are identified when peaks are observed in the retention time window for the analyte on both columns. Confirmation of peak on the secondary column is based on comparison of the retention times with the corresponding peak of the standard analyzed before the samples. When identification has been confirmed on the confirmation column, the analyst should evaluate the agreement of the quantitated results on both columns. For any result where the RPD of the values measured on the primary and secondary columns is greater than 40%, the result is reported with a J flag and noted in the case narrative.

14.6 If the agreement between the quantitated results on both columns is higher than 70%, consult your supervisor/ manager before results can be reported.

In addition, for DOD projects when the RPD is greater than 40%, report the

higher of two confirmed results unless an overlapping or interference peak is causing erroneously high results. In this case, report the non-affected result and document in the case narrative.

14.7 Surrogate recoveries are quantified for all blanks, samples, matrix spikes and lab control spikes. Surrogate recoveries should be established and monitored by plotting control charts. Recoveries of surrogate for the blank and samples should be within the specified ranges. If the recoveries of surrogate are outside the QC limits re-analyze the extract. Re-extraction of the sample should also be considered if re-injection of the sample produces similar results. The judgment of the experienced analyst is heavily relied upon when re-extraction and/or re-injection are necessary.

## 15.0 SAFETY

15.1 Standard precautionary measures used for handling other organic compounds like safety glasses, laboratory coats, gloves should be sufficient for the safe handling of the analytes handled by this method. The only extra caution should be taken is when handling the analytical standard neat material.

15.2 Visual observation of soil sample is important when the sample is taken from a site expected to contain explosives lump of material that have a chemical appearance should be suspect and not ground. Explosives are generally a very finely ground grayish-white material.

## 16.0 POLLUTION PREVENTION

16.1 GPL Laboratory operates in a safe manner to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. For more detail on pollution prevention, refer to GPL SOP D.5.

## 17.0 WASTE MANAGEMENT

17.1 Several wastes that GPL generates can be handled in a fairly routine manner. The process of describing the method for waste disposal of chemicals including standards and reagent solutions, and process waste, and samples is described in Standard Operating Procedures D.1 and D.2.

## 18.0 DEFINITIONS

18.1 For definitions of terms used in this document, refer to GPL Laboratory SOP G.14.

Figure 1

# METALS DIGESTION/PREPARATION

## METHODS

**USEPA SW846**

**3005A, 3010A, 3030C, 3031, 3050B**

**USEPA CLPILM 04.1 Aqueous & Soil/Sediment (NJDEP does not accept CLPILM 04.1 after June, 2003)**

**Addendum for USEPA CLPILM 05.2 Aqueous & Soil/Sediment**

**USEPA Methods for Chemical Analysis of Water and Wastes**

**200.7, Standard Methods 3030C**

**SOP NUMBER:** **SOP-100**

**REVISION NUMBER:** **19**

**APPROVED BY:**

**SECTION MANAGER**

**QUALITY ASSURANCE OFFICER**

**07/25/06**
**EFFECTIVE DATE**

**04/10/08**
**DATE OF LAST REVIEW**

# METALS DIGESTION/PREPARATION

**References:**
**Methods 3005A/USEPA CLPILM0 4.1 Aqueous, 3010A, 3030C, 3031, 3050B**
**USEPA CLPILM0 4.1 (Soil/Sediment), 200.7, Standard Methods 3030C**
**See Addendum for USEPA CLPILM 05.2 (Aqueous & Soil/Sediment)**

## I. SCOPE AND APPLICATION

A. AQUEOUS

1. Method 3005A and USEPA CLP ILM0 4.1, "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by ICP Spectroscopy".
   a. This method is used to prepare surface water, ground water, drinking water and wastewater samples for analysis by inductively coupled argon plasma spectroscopy (ICP).

2. Method 200.7, "Determination of Metals and Trace Metals in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry"
   a. This method is used to prepare surface water, ground water, drinking water and wastewater samples for analysis by inductively coupled argon plasma spectroscopy (ICP).

3. Method 3010A, "Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP Spectroscopy".
   a. This method is used to prepare aqueous samples, EP and mobility-procedure extracts, and wastes that contain suspended solids for analysis by ICP. The procedure is used to determine total metals.

4. Method 3030C (Standard methods), "Preliminary Treatment for Acid-Extractable Metals".
   a. This method is used to prepare ground water samples from North Carolina for analysis by ICP.

B. SOLIDS

1. Method 3050B, "Acid Digestion of Sediments, Sludges and Soils".

   a. This method is used to prepare sediments, sludges and soil samples for analysis by ICP. Since certain matrices may result in poor recovery, the method of standard additions may be used when analyzed.

   b. It should be noted that some metals could be biased high with the soil digestion when dilution is necessary. Take necessary measures to ensure that dilutions are made as accurately as possible.

2. USEPA CLP ILM0 4.1, "Acid Digestion of Soil/Sediment"

   a. This method is used to prepare sediments and soil samples for analysis by ICP. Since certain matrices may result in poor recovery, the method of standard additions may be used when analyzed.

C. OILS

1. Method 3031, "Digestion Procedure for Oils".

   a. This method is used to prepare samples containing oils, greases or waxes for analysis by inductively coupled argon plasma emission spectroscopy (ICP).

D. **NOTES:**

1. "Total Metals" includes all metals, inorganically and organically bound and both dissolved and particulate.

2. "Dissolved metals" includes all metals present in a sample after filtration through a 0.45 micron filter followed by digestion.

## II. SUMMARY OF METHODS

A. A representative sample of water, soil or oil is put into an acid medium and exposed to heat for a certain amount of time. This allows for reduction of interferences by organic matter and converts metals bound to particulates to form the free metal that can be determined by ICP-Atomic Emission Spectrometry.

NOTE: When a reporting limit is required for a project lower then is customary, a four times concentration must be used in order to reach that lower level. Care

must be taken to matrix match this concentrated aliquot. A blank and laboratory control sample (at a reduced concentration) are required with this concentration. A matrix spike ( not at reduced concentration) and duplicate or matrix spike and matrix spike duplicate is needed per 20 samples or per batch.

## III. SAMPLE HANDLING AND PRESERVATION

A. AQUEOUS

1. Samples are taken in high density polyethylene, one liter bottles. Samples should be preserved with concentrated $HNO_3$ to a pH <2 immediately once sampled. If dissolved metals are to be analyzed the sample should be filtered before the $HNO_3$ is added. The samples should be maintained at 4°C until analysis. The holding time for metals samples is 180 days or approximately 6 months.

B. SOLIDS

1. Samples are taken in high density polyethylene(CLP only) or glass bottles. The samples should be maintained at 4°C until analysis. The holding time for metals samples is 180 days or approximately 6 months.

C. OILS

1. Samples are taken in high density polyethylene bottles. The samples should be maintained at 4°C until analysis. The holding time for metals samples is 180 days or approximately 6 months.

## IV. INTERFERENCES

A. AQUEOUS

1. Method 3005A and USEPA CLPILM0 4.1, "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by ICP Spectroscopy", SW846, July, 1992.

   a. This digestion procedure may not be sufficiently vigorous to destroy some metal complexes.

2. Method 200.7

3. Method 3010A

a. See method 6010B.

B. SOLIDS

1. Method 3050B

a. Sludge samples can contain diverse matrix types, each of which may present its own analytical challenge. Spiked samples and any relevant standard reference material should be processed to aid in determining whether this method is applicable to a given waste.

2. USEPA CLP ILM0 4.1

a. Sludge samples can contain diverse matrix types, each of which may present its own analytical challenge. Spiked samples and any relevant standard reference material should be processed to aid in determining whether this method is applicable to a given waste.

C. OILS

1. Method 3031

a. These digestates can have very high dissolved solids, which may necessitate the use of internal standards, dilutions, or the method of standard addition.

## V. SAFETY

A. Normal accepted laboratory safety practices should be followed while performing this analysis.

B. Be certain the exhaust hood is functioning before you begin the digestion procedure.

C. Hot acids can be extremely corrosive. Avoid inhalation or contact with skin.

## VI. EQUIPMENT/APPARATUS

A. Fume hood, Labconco or equivalent.

B. Hot plate, Thermolyne cimarec-3 or equivalent source for use at 95°C. The temperature of the hot plate must be monitored via the use of a temperature blank.

C. Thermometer capable of reading 80 to 120 degrees C – ERTCO cat# 611-3-SC or equivalent.

D. Vacuum pump for filtering dissolved metals- Gast or equivalent.

E. Analytical balance capable of weighing to 0.01 gram. Mettler model BB300 or equivalent.

F. Beckman CS-6R centrifuge.

G. Various class A volumetric glassware and ribbed watchglasses, Pyrex or equivalent.

H. Whatman No. 41 filter paper or equivalent.

I. Whatman No. 42 filter paper or equivalent.

J. Whatman 0.45 micron filter paper or equivalent.

K. 250 mL beaker or other appropriate vessel such as polypropylene block digester tubes, watch glasses and caps.

L. Stirring device, e.g. magnetic stirrer, glass rod or equivalent.

M. Manual Sample Mill

N. Wiley Sample Mill

O. Clippers for cutting vegetation

NOTE: All glassware should be acid washed.

## VII. REAGENTS AND STANDARD PREPARATION

### A. REAGENTS

1. Metals grade Nitric acid ($HNO_3$). Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.

2. Metals grade Hydrochloric acid (HCl). Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.

3. 30% hydrogen peroxide reagent, ACS Grade. Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.

4. Metals grade Sulfuric acid ($H_2SO_4$). Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.

5. Reagent water (Deionized water).

6. Potassium Permanganate - Ultra pure grade. Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.

7. Ammonium hydroxide, concentrated, reagent grade. Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.

8. Ammonium phosphate, reagent grade- Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.

9. Base oil, analyte-free. Oil should be analyzed to determine level of impurities. If method blank is < MDL, then the reagent can be used.

## B. STANDARDS

### 1. Traceability

a. A bound logbook record shall be maintained on all reference materials. The record shall include date of receipt, source, purity, all compositional information, storage conditions and expiration date. These materials/solutions are to be identified by a unique number in the logbook as well as on the container's label.

b. All working standards made from reference materials shall be labeled with a unique ID number with complete information on preparation date, concentration of each compound, solvent, preparer's name, expiration date and the logbook where information is recorded. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded in a bound logbook. Measurements made during standards preparation (e.g., from weighing operations, volume diluted to, etc.) shall also be recorded. There should be no container with sample, sample extract, standard solution, etc. that is not correctly labeled and properly stored.

c. The analyst must initial and date each entry made in a logbook. Each analyst must be sure to "Z" out the unused area of each logbook page.

## 2. PREPARATION

A. Laboratory control sample

1. Aqueous

a. This solution is prepared as follows: 50 mL concentrated HCl, 20 mL concentrated $HNO_3$, 1 mL of CLP-CAL-1, Solution A, 1 mL of CLP-CAL-1 Solution B, 0.25 mL of CLP-CAL-2, and 0.25 mL of CLP-CAL-3 diluted to 1 L in a volumetric flask. Use 50 mL (100 mL for strict CLPIIM0 4.1) for digestion. This solution is given a unique identifier and recorded in sample digestion logbook.

b. For four times concentrated samples: The solution is prepared as follows: 50 mL concentrated HCl, 20 mL concentrated $HNO_3$, 1mL CLPP-SPK-4 (Inorganic Ventures) (This solution contains 10 mg/L Selenium, 100 mg/L Antimony, 50 mg/L Cadmium and Thallium, 40 mg/L Arsenic and 20 mg/L Lead) to 1 L in a volumetric flask. This solution is given a unique identifier. Use 12.5 mLs to 50 mLs and prepare two aliquots. Heat at 90 to 95°C to reduce the volume in each vessel to ten mLs and then combine each 10 mL aliquot into one vessel and take to a final volume of 25 mLs. Take care to matrix match acids so that the final 25 mL portion will contain 2% $HNO_3$ and 5% HCl. Use 0.125 mLs $HNO_3$ and 0.3125 mLs HCl to each 50 mL vessel.

2. Solids

a. A 1.0 ±0.02 gram aliquot of teflon chips is weighed and spiked using the same spiking solution used for matrix spikes. This sample is given a unique identifier according to the Lot# for the teflon chips used and when digested is given the descriptor. i.e. LCSS(date)A and then B etc. plus the unique identifier number assigned. Alternatively a solid matrix standard reference material is obtained from the manufacturer. This sample is given a unique identifier and recorded in the sample digestion logbook.

3. Oils

a. **An analyte free oil MUST be used or explosive reactions can occur.** An analyte free oil (wesson oil which has been analyzed previously to prove that it is < MDL.) is spiked using the same spiking solution used for matrix spikes. This sample is given a unique identifier. i.e. LCSO(date)A and then B etc.

B. Spiking solution

1. Sample is spiked using 0.1 mL of CLP-CAL-1, Solution A, 0.1 mL of CLP-CAL-1 Solution B, 0.025 mL of CLP-CAL-2 and 0.025 mL of CLP-CAL-3 for a final volume of 100 mL. If only 50 mL is used, decrease amount used appropriately. These solutions are given unique identifiers. Record the amount spiked and the unique identifier of the standard.

2. CLP sample is spiked using 0.1 mL CLPP-SPK-1 and 0.1 mL CLPP-SPK-4 for a final volume of 100 mL. If only 50 mL is used, decrease amount used appropriately. These solutions are given unique identifiers.

3. For samples that require four times concentration, the sample is spiked using 0.0125 mLs of CLPP-SPK-4 to each of two vessels with 50 mLs of sample in each. The volume of each of the vessels is lowered to less then 10 mLs and combined and the final volume of this concentrated sample is 25mLs.

## VIII. CALIBRATION

**A.** The temperature of the samples must be maintained at 95°C and monitored via a temperature blank. 85° for oil samples. Record in digestion logbook.

## IX. PROCEDURE

A. Glassware preparation for oil digestion or when the hot-block can not be used:

1. Wash glassware with hot soapy water and rinse thoroughly. (Beakers must be washed as soon as possible after being used, dirty beakers must not be allowed to sit overnight.)
2. Rinse glassware with reagent water that contains 5% $HNO_3$ and 5% HCl followed by a rinse with reagent water.
3. Prior to use, all glassware must be confirmed clean via a glassware check. Otherwise, repeat step "2" until the glassware check passes.

B. Aqueous sample filtration (for dissolved metals):

1. Thoroughly clean a flask and funnel with hot soapy water. Next, rinse the flask and funnel with 1:5 $HNO_3$ followed by a thorough D.I. water rinsing. This step is very important because the filters contain some metals (namely Zn) which could contaminate the samples.
2. Rinse a 0.45 micron filter with 1:5 $HNO_3$ thoroughly, followed by D.I. water.
3. Filter the unpreserved sample. If dissolved Hg analysis is requested for the sample, filter at least 200 mL.
4. Discard the first 50 to 100 mL.
5. A preparation blank must be taken through the filtration step and analyzed with the sample.
6. Preserve the sample with $HNO_3$ to pH<2.
7. Soluble samples that are clean and clear do not have to be digested. Use 100 mL sample, add 5 mL of concentrated HCl and 2 mL of concentrated $HNO_3$. **Samples must be digested unless approval for analysis without digestion is received from the project manager.**

C. Aqueous sample preparation

1. Method 3005A and USEPA CLP ILM0 4.1, "**Acid digestion procedure for total recoverable or dissolved metals for analysis by ICP ".**

a. Shake sample thoroughly and pour 50 mL of the well-mixed sample into a digestion vessel. For samples which require concentration pour 50 mLs of the well-mixed sample into two digestion vessels.

b. Add 0.50 mL ( 1 mL of (1+1) when strict CLP ILM0 4.1 is required) concentrated $HNO_3$ to the sample. For samples which require concentration, add 0.125 mL (0.25 mL of (1+1) when strict CLP ILM0 4.1 is required) concentrated $HNO_3$ to the sample.

c. Add 2.5 mL ( 5 mL of 1+1) when strict CLP ILM0 4.1 is required) concentrated HCl to the sample. For samples which require concentration, add 0.3125 mL (0.625 mL of (1+1) when strict CLP ILM0 4.1 is required) concentrated HCl to the sample.

d. Cover the sample with a ribbed watch glass or equivalent source.

e. Transfer the digestion vessel to a pre-heated hot plate or hot block at 90 to 95°C. A temperature blank will assure correct temperature. The temperature must be recorded in the digestion log book. Take the volume down to between 5 to 10 mL, ( 12 to 25 mLs when strict CLP ILM0 4.1 is required) **making certain that the sample does not boil. This is extremely important. Boiling may lead to vaporization of certain analytes.** Remove the sample from the hot plate and cool

f. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.

g. Bring sample to its predigestion volume ( or when samples require concentration, to a volume four times lower then what was started with) with DI water in the digestion vessel. The final volume must be recorded in the digestion log book.

h. The sample is now ready for analysis.

i. The digestion log must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards plus identification #'s for standards used for spiking and the volume spiked into the sample.

2 Method 200.7, "**Acid digestion procedure for total recoverable metals**".

a. Shake sample thoroughly and pour 50 mL of the well-mixed sample into the digestion vessel. If sample contains undissolved solids >1% refer to Section 11.3 of Method 200.7 for subsequent procedures.

b. Add 1.0 mL concentrated $HNO_3$ to the sample.

c. Add 2.50 mL concentrated HCl to the sample.

d. Cover the sample with a ribbed watch glass or equivalent source.

e. Transfer the digestion vessel to a pre-heated hot plate or equivalent source at 85°C. Take the volume down to between 10 to 15 mL, **making certain that the sample does not boil. This is extremely important. Boiling may lead to vaporization of certain analytes.**

f. Leave sample on hot plate and gently reflux for 30 minutes. Remove from hot plate and cool.

g. Bring sample to its predigestion volume with DI water in the digestion vessel.

h. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.

i. The sample is now ready for analysis.

j. The digestion log must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards.

3. Method 3010A, "**Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP Spectroscopy**".

a. Shake sample thoroughly and pour 50 mL of the well-mixed sample into the digestion vessel.

b. Add 1.5 mL concentrated $HNO_3$ to the sample.

c. Cover the sample with a ribbed watch glass.

d. Transfer the digestion vessel to a pre-heated hot plate or hot block at 90 to 95°C. A temperature blank must be used, with the temperature being recorded in the log book. Take the volume down to a low

volume (~5 mL), **making certain that the sample does not boil. This is extremely important. Boiling may lead to vaporization of certain analytes. Also make certain that no portion of the bottom of the digestion vessel is allowed to go dry. This may lead to low recoveries.** Remove the sample from the hot plate and cool.

e. Add another 1.5 mL portion of concentrated $HNO_3$ to the sample.

f. Cover the sample with a ribbed watch glass.

g. Transfer the vessel to the hotblock or equivalent source. Increase the temperature so a gentle reflux occurs. Continue heating, adding additional acid as necessary, until the digestion is complete (generally indicated when the digestate is light in color or does not change in appearance with continued refluxing).

h. Uncover the vessel and evaporate to a low volume (~3 mL) **making certain that no portion of the bottom of the digestion vessel is allowed to go dry.** Remove and cool.

i. Add 2.5 ml of 1:1 HCl (10 mL/100 mL of final solution).

j. Cover the digestion vessel and reflux for an additional 15 minutes.

k. Bring sample to its predigestion volume in digestion vessel.

l. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.

**Note:** When preparing USACE project samples, if any sample in a digestion batch requires filtration, all samples (including QC samples) must be treated in the same manner.

m. The sample is now ready for analysis.

n. The digestion log must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards.

4 Method 3030C (Standard Methods), "**Preliminary treatment for Acid-Extractable Metals**"

a. Shake sample thoroughly and pour 50 mL of the well-mixed sample into a 50 mL digestion vessel.

b. Add 2.5 mL 1:1 HCl to the sample.

c. Heat 15 minutes in a hot bath.

d. Filter through a membrane filter.

e. Adjust filtrate volume to 50 mL with DI water.

f. Transfer to ICP analyst.

D. Solid sample preparation

*It is extremely important that waste (when appropriate), soil and sediment samples be mixed thoroughly to ensure that the sample is as representative as possible of the sample media. The most common method of mixing is referred to as quartering. The quartering procedure should be performed as follows:*

- *The material in the sample pan(inorganic-plastic/organic-aluminum) should be divided into quarters and each quarter should be mixed individually.*
- *Two quarters should then be mixed to form halves.*
- *The two halves should be mixed to form a homogenous matrix.*

*This procedure should be repeated several times until the sample is adequately mixed.*

**NOTE: Samples that are clay type materials should be handled in a different manner. Due to these type sample matrices having an affinity to stick to most anything that touches it, another approach must be followed. Obtain a representative sub-sample aliquot from the center or middle section of the sample container.**

**Grinding of Vegetation Samples**

Remove sample from shipping container and brush off dirt particles. Chop sample into about half inch pieces with clippers or other cutting tool. Place the sample in an aluminum pan and air-dry in an exhaust hood to the appropriate dryness for grinding. It should be dry enough where it won't stick to the inside of the mill. Grind the dried sample to fineness in either the manual sample mill

or the Wiley mill or both if needed. Place the ground sample in a container and label immediately.

1. USEPA CLP ILM0 4.1, **"Acid digestion of Soil/Sediment"**

   a. Mix the sample thoroughly to achieve homogenity. For each digestion procedure, weigh (to the nearest 0.01 g) a 1.0 to 1.5 g portion of sample and transfer to a digestion vessel.

   b. Add 10 mL of 1:1 nitric acid ($HNO_3$), mix the slurry, and cover with a watch glass or equivalent source. Heat the sample to 92 to 95°C and reflux for 10 minutes without boiling. Allow the sample to cool, add 5.0 mL of concentrated $HNO_3$, replace with watch glass or equivalent source, as appropriate, and reflux for 30 minutes. Do not allow the volume to be reduced to less than 5 mL while maintaining a covering of solution over the bottom of the heating vessel.

   c. After the second reflux step has been completed and the sample has cooled, add 2 mL of Type II water and 3.0 mL of 30% hydrogen peroxide ($H_2O_2$). Return the heating vessel to the hot plate or equivalent heating source for warming to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides, and cool the heating vessel.

   d. Continue to add 30% $H_2O_2$ in 1 mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged. (NOTE: Do not add more than a total of 10 mL 30% $H_2O_2$.)

   e. If the sample is being prepared for ICP analysis of Al, As, Sb, Ba, Be, Ca, Cd, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Ag, Na, Tl, V, and Zn, add 5 mL of 1:1 HCl and 10 mL of Type II water, return the covered heating vessel to the hot plate or equivelent heating source, and heat for an additional 10 minutes. After cooling, filter through Whatman No. 42 filter paper (or equivalent) and dilute to 50 mL with Type II water. NOTE: In place of filtering, the sample (after dilution and mixing) may be centrifuged or allowed to settle by gravity overnight to remove insoluble material. Dilute the digestate to 144 mL with DI water, add 5 mLs concentrated HCl and 1 mL of concentrated $HNO_3$, mix well and place into the appropriate container. The diluted sample has an approximate acid concentration of 2.5% (v/v) HCl and 5% (v/v) $HNO_3$. The sample is now ready for analysis.

f. The digestion log must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards and ID of matrix spikes and the amounts used for spiking.

2. Method 3050B, **"Acid digestion of Sediments, Sludges and Soils"**

a. Mix the sample thoroughly for 5 minutes using a plastic spatula or Teflon coated spatula in a glass or plastic weigh boat to achieve homogeneity.

b. Weigh approximately (to the nearest 0.01 g) a 1 to 1.5 g portion of the sample directly into a digestion vessel. For samples with low percent solids a larger sample size may be used as long as digestion is completed. Record the exact mass in the digestion log.

**NOTE: To achieve the lowest reporting limit possible, use a 2.0 g portion of sample with an ending volume of 100 mLs.**

c. Add 5 mL D.I. water and 5 mL concentrated $HNO_3$(1:1), mix the slurry and cover with a watch glass. Place the sample in a preheated hot block and reflux at 95°C for 10 to 15 minutes being certain that the sample does not boil. Record temperature in digestion log book

d. Allow the sample to cool. Add 5 mL concentrated $HNO_3$, replace the watch glass and heat/reflux again for 30 minutes. If brown fumes are generated, indicating oxidation of the sample by $HNO_3$, repeat this step (addition of 5 mL of concentrated $HNO_3$) over and over until no brown fumes are given off by the sample indicating the complete reaction with $HNO_3$. Using a watch glass or equivalent allow the solution to evaporate to approximately 5 mL without boiling at 95°C ± 5°C for approximately two hours. Maintain a covering of solution over the bottom of the vessel at all times. Do not allow the volume to be reduced to less than 5 mL while maintaining a covering of solution over the bottom of the beaker. If the volume does get low, add 2.5 mL of D.I. water to bring volume back up.

e. Take the sample off the hot block and allow it to cool. Next, add 2 mL of D.I. water and 3 mL of 30% Hydrogen Peroxide. (The sample will bubble upon the addition of $H_2O_2$ if it is still warm.) Cover the vessel with a watch glass and return the sample to the hot block or equivalent source and heat until the bubbling subsides. Care must be taken to

ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides and cool the beaker. Add two more 3 mL portions of $H_2O_2$ to the sample in the same manner as before. (NOTE: Do not add more than a total of 10 mL 30% $H_2O_2$.)

f. Cover the sample with a ribbed watch glass and continue heating the acid-peroxide digestate at 95°C ± 5°C without boiling for approximately two hours until the volume has been reduced to approximately 2.5 mL. Maintain covering of solution over the bottom of the vessel at all times.

g. Add 2.5 mL of DI water and 2.5 mL of concentrated HCl and 10 mL of DI water, cover the sample with a ribbed watch glass and continue refluxing for an additional 10 minutes without boiling

h. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.

i. Bring sample up to 50 mL with D.I. water in the vessel. Add 150 ml of DI water to a 250 ml sample bottle. Invert the 50 ml sample digestion vessel several times to mix the sample and pour sample into the 150 ml of the sample bottle. Pour some sample back into the 50 ml sample digestion vessel to rinse and pour back into the 250 ml sample bottle and cap and mix.

**NOTE1:** When preparing USACE project samples, if any sample in a digestion batch requires filtration, all samples (including QC samples) must be treated in the same manner.

**NOTE2:** To achieve the lowest reporting limit possible use 2.0 grams of sample with an ending volume of 100 mLs.

j. The sample is now ready for analysis.

k. The digestion log must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards.

E. Oils

1. Method 3031, "**Digestion Procedure for Oils**"

**NOTE: THIS METHOD IS VERY TIME CONSUMING--DISCUSS SUB-CONTRACTING SAMPLES WITH YOUR SUPERVISOR AS SOON AS THEY COME IN THE DOOR.**

a. Homogenize sample and Weigh approximately (to the nearest 0.01 g) a 0.5 g representative portion of the sample into a 250 mL beaker. Separate and weigh proportional aliquots of the phases if more than one phase is present. Record the exact mass in the digestion log. Larger or smaller sample sizes can be used if needed.

g. Add 0.5 g of potassium permanganate powder. If larger sample sizes are used, increase the amount of potassium permanganate so that the ratio of oil to postassium permanganate is still 1:1. Mix the oil and permanganate thoroughly until homogenous. Thick oils and tars that cannot be mixed should be heated to achieve mixing (the oil may react mildly). It is important to record the amount of potassium permanganate used for each sample if analysis is by ICP-AES and correction is to be made for the amount of manganese. If more than 10% of the sample is aromatic material, such as xylene, then the reaction will be incomplete. If this is the case, increase the amount of potassium permanganate. If the sample is a mixture of oil and other non-organic materials, reduce the amount of potassium permanganate.

NOTE: All steps requiring the use of acids should be conducted under a fume hood by properly trained personnel using appropriate laboratory safety equipment. This should include face shields and latex gloves.

h. Cautiously add 1.0 mL concentrated $H_2SO_4$, and stir with an appropriate stirring device. If larger sample sizes are used, increase the volume of the sulfuric acid so that the ratio of oil to sulfuric acid is 1 g to 2 mL. The $H_2SO_4$ can be added dropwise or all at once, depending on analytical needs. (Generally, dropwise is preferred when low reporting limits are needed.)

NOTE: To prevent a strong exothermic reaction, $H_2SO_4$ should be added dropwise to all samples unfamiliar to the analyst and to all samples that are known to be highly reactive.

The reaction can take several seconds to begin, but when it occurs it will be very quick, vigorous, and exothermic. Generally larger sample sizes will react faster than smaller. Likewise, lower average molecular weight materials will react faster than heavier. Do not be mislead by an initial lack of reactivity. A grey-white vapor will be ejected from the beaker ($SO_3$) and splattering and bubbling can occur. The beaker will become very hot. This step is complete when no more gases are given off and the sample would be a thick black lumpy paste. Allow the beaker to cool as needed.

NOTE: Care must be taken when working with very light organic materials, such as diesel fuels, as they may flash. Generally, the lower the average molecular weight of the meterial correlates to a greater danger of flashing. The danger of flashing is reduced by adding the sulfuric acid dropwise.

NOTE: If more than 10% of the sample is aromatic material, such as xylene, only a little grey-white vapor will form. This will reduce accuracy and complicate nebulization. If there is a signigicant amount of non-hydrocarbon material, a sputtering recation will occur and black $MnO_2$ particulates will be given off. See section (b.) above under procedure.

i. Add 2 mL of concentrated $HNO_3$ and stir. This reaction will be slightly exothermic. If larger sample sizes are used, it is not always necessary to increase the volume of $HNO_3$ proportionately, depending on analytical needs. Some reddish-brown vapor ($NO_2$) may be given off. Allow the reaction to continue until complete, that is when the digestate no longer gives off fumes. Allow the beaker to cool as needed.

j. Add 10 mL of concentrated HCl and stir. If larger sample sizes are used, it is not always necessary to increase the volume of HCl proportionately, depending on analytical needs. This reaction will be slightly exothermic and gas formation and foaming will occur. Lighter oils will foam more than will heavier oils. If excess foaming occurs, add water to prevent sample loss. Allow the beaker to cool as needed.

k. Heat the beaker until there is no further gas evolution. (temperature should not exceed 150 °C to prevent volatilization). There may be additional foaming or other milder reactions which may result in overflow from the beaker. If excess foaming occurs, either remove the beaker from the heating source until foaming subsides or add sufficient

water to prevent overflow. The final digestate should be a clear yellow liquid with black or dark reddish-brown particulates.

l. Filter the digestate through Whatman 41 filter paper and collect filtrate in a volumetric flask or beaker.

m. Wash the digestion beaker and filter paper, while still in the funnel, with no more than 5 mL of hot HCl.

NOTE: The purpose of this next step is to recover antimony, barium, and silver that may not have been completely solubilized. If the sample is not being prepared for these analytes, the next step may be skipped.

n. (Optional) After having washed the filter paper, remove the filter and residue from the funnel and place it back in the beaker. Add 5 mL of conc. HCl and place the beaker back on the heating source until the filter paper dissolves (temperature should not exceed 150 °C ±5 °C to prevent volatilization). Remove the beaker from the heating source and wash the cover and sides with reagent grade water and then filter the residue and collect the filtrate in the same flask or beaker as in sections f. and g. above. Allow the filtrate to cool and quantitatively transfer to a volumetric flask. Bring to volume.

o. (Optional) If the filtrate is collected in a beaker, the filtrate can be heated again to drive off excess HCl. This can reduce matrix effects in sample introduction (temperature should not exceed 150 °C ± 5 °C to prevent volatilization). When sufficient HCl has been removed, remove the beaker from the heating source, allow to cool, and then transfer the contents to a volumetric flask and bring to volume. However, if too much HCl is removed, barium, silver and antimony can be lost.

p. Analyze the flitrate by ICP-AES. Depending on the final volume selected, the total solids in the digestate may be high enough to cause nebulization problems. Problems due to high dissolved solids may be corrected by 1) following optional Section i., 2) using internal standards, 3) using flow injection analysis, or 4) using other matrix correction procedures.

Manganese Removal Steps

NOTE: The purpose of these next steps is to remove the manganese in the digest by precipitating it as manganese ammonium phosphate

under alkaline conditions. Elements that do not form insoluble phosphates, such as arsenic, are filtered out and can be analyzed at lower concentrations.

q. Take the digestate, or portion of digestate and reduce the volume to remove as much HCl as possible without going below 10 mL. Then add conc. $NH_4OH$ until pH is 7 or greater. For most matrices, the digestate will change colors (often from yellow to brown) at pH 7. A mild exothermic reaction will occur immediately.

r. Add at least 2 g ammonium phosphate for each 1 g of potassium permanganate used in the digestion and stir. An excess of phosphate is needed for good analyte recovery. Then add enough water and mix to ensure maxium precipitation. A pink or yellow silky amorphous precipitate, manganese ammounium phosphate, will form. If too much $NH_4OH$ is used some of the manganese ammonium phosphate can be solubilized. Stir until precipitation is complete. Some ammonium phosphate may remain unreacted at the bottom of the beaker.

s. Filter the digestate through Whatman 41 filter paper (or equivalent) and collect filtrate in a volumetric flask or beaker.

t. Heat the filtrate to volatilize the ammonia (temperature should not exceed 150 °C ± 5 °C to prevent volatilization). The volume of filtrate can be reduced by heating to no less thatn 10 mL. If too much water is removed ay ammonium chloride formed will solidify. If this occurs, either add enough water to dissolve the solids or filter out the solids and wash the residue with deionized water. The filtrate can be analyzed by ICP-AES.

u. The digestion log must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards.

## X. CALCULATIONS

A. The analyst must be supplied with both beginning sample masses/volumes and final digestate volumes. This information must be recorded in the digestion log.

## XI. QUALITY CONTROL

A. Digestion

1. Temperature blank

   a. The temperature of the hot plate/hot block must be monitored for temperature during the digestion process.

   b. The thermometer must be tagged with annual calibration information. Record the thermometer reading, correction factor and the corrected temperature in the digestion log.

2. Blanks

   a. Digest a blank with every batch of samples digested (20 sample maximum). The blank is prepared by adding all the same reagents added to the samples to a clean dry beaker and taking it through the same process as the samples. **NOTE: The blank for OILs MUST include an analyte-free oil or explosive reactions can occur.**

   b. Also, there must be a blank for every different method of digestion that is set up that day, every 20 samples.

   c. There must also be a blank for every different matrix of samples that is to be digested, every 20 samples.

   d. Sample is given a unique identifier in the digestion log.

3. Laboratory Control Samples

   a. For water samples, one LCS is digested with every batch of samples digested (20 sample maximum).

   b. For water samples, a LCS is digested every day for each type of digestion, every 20 samples.

   c. For soil/sediment samples, a soil matrix standard reference material (SRM ) must be digested per batch (20 samples maximum) or alternatively a spiked teflon chip sample.

   d. Sample is given a unique identifier in the digestion log.

   e. Recoveries of standard reference materials or laboratory control samples spiked with organo-metallic standards recoveries should be **±25% of their true values for OILS.**

4. Duplicates

a. A duplicate is prepared every 20 samples. This usually takes the form of a matrix spike duplicate.

**NOTE:** Certain projects require a sample duplicate and a matrix spike duplicate with each set of twenty samples.

5. Blank Spike

a. This is required for certain projects.

B. Sample Matrix

**NOTE:** Field blanks/duplicates, trip blanks, or equipment blanks are not to be used for sample matrix QC samples.

1. Matrix spike

a. Digest a spike and spike duplicate every 20 samples where sample volume is adequate to do so. Choose a sample (if possible) that has a lot of metals requested to be analyzed.

**NOTE:** For some projects, a sample duplicate and sample spike may be required instead of a spike and spike duplicate. Your supervisor should make you aware of these projects.

b. The following metals do not get digested spikes when using CLP spike.

Calcium
Magnesium
Sodium
Potassium

v. For TCLP samples, a spike must be digested for every matrix. You should inspect the sample (original sample prior to extraction) or check the log book to determine matrix type. (Also the matrix spike aliquot must be added to the extract after filtration but before preservation.)

**d. The CLH project requires that a high and a low spike be prepared and analyzed. Spikes shoud be prepared at 40 mg/Kg and 400 mg/Kg for soil samples and 200 ug/L and 2000 ug/L for aqueous samples.**

## XII. CORRECTIVE ACTIONS

A. Sample boils during digestion.

1. Redigest another sample aliquot.

B. Sample goes dry or portion of beaker bottom is exposed due to excess evaporation during digestion.

1. Redigest another sample aliquot.
2. Glass beaker dry for an extended period of time? Discard beaker.

## XIII. SPECIAL NOTES

A. **Never** take for granted how a sample should be digested. If the sample looks strange or unusual, or if you are not sure what metals the sample gets, what detection limits are required, whether the sample is total or dissolved, or even what method of digestion should be used, always ask your supervisor or the person who is to analyze the sample. How metals need to be digested changes too often to take it for granted.

B. **Antimony (Sb) soils** should be analyzed within 48 hours of digestion whenever possible. When a soil requesting Antimony analysis is received, you must coordinate with the person who will be analyzing it to be sure that they can analyze it on the same day that it is digested.

C. Labels for the digested sample must be written in a neat and legible manner. The labels must include such information as sample number, client name, the date digested, and the volume or mass digested.

D. There are several precautions that must be taken to minimize the possibility of contamination.

1. All metals glassware must be kept separate from all other laboratory glassware.

2. Metals glassware must be washed as soon as possible after being used. **Dirty metals beakers must not be left overnight.**

3. Acid to be used for metals digestions must be kept separate from all other laboratory acid.

E. Samples must be digested in a timely manner to ensure ICP analysis remains on schedule for data generation. Samples received on or before Wednesday of week X must be prepared for ICP digestion by the end of week X. Your supervisor must be consulted if this schedule can not be met at a particular time.

F. Please consult Waste Disposal SOP-405, for information concerning disposal of waste generated from this area. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

## Addendum for USEPA CLPILM 05.2 AQUEOUS &SOIL/SEDIMENT

The following is a list of changes for sample preparation when the 5.2 statement of work is required:

1. Soluble samples are required to be digested unless the chain of custody specifically states that digestion is not required. An MDL study must be done on the unprepared MDL solution in order to provide MDL levels for samples that are not digested. When digestion is not required an LCSW and post digestion spike are not required.
2. Digestates must be stored until 365 days after delivery of a complete, reconciled data package.
3. Preparation codes are used on form 13's. They are found in the 5.2 statement of work page B-39 3.4.12.2.4.

**DEFINITIONS** – Refer to SOP-431 for common environmental laboratory definitions.

# METALS ANALYSIS

# BY INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROMETRY (ICP-AES) TECHNIQUE

## METHODS 200.7, ( SW846) 6010B, (SM 19th Edition 2340B) Hardness Calculation, (USEPA CLP) ILMO 4.1 (NJDEP does not accept CLPILM 04.1 after June, 2003)

## Addendum for USEPA CLPILM 05.2

**SOP NUMBER:** SOP-105

**REVISION NUMBER:** 14

**APPROVED BY:**

SECTION MANAGER

TECHNICAL DIRECTOR

**EFFECTIVE DATE:** 09/09/08

**DATE OF LAST REVIEW:** 09/09/08

**ICP METHOD**
**SOP**
**References: SW-846, Method 6010B, December 1996**
**USEPA, Method 200.7, June 1991, Standard Methods 19th Edition 2340B, 1995**
**USEPA CLP, ILM 04.1. See Addendum for USEPA CLPILM 05.2**

## I. SCOPE AND APPLICATION

A. Inductively Coupled Argon Plasma (ICP) determines trace elements in solution. **We use the ICP to determine the concentration of the following metals: Ag, Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, Pb, Sb, Se, Sn, Sr, Ti, Tl, V and Zn.** All matrices, including ground water, aqueous samples, TCLP, SPLP and EP extracts, industrial and organic wastes, soils, sludges, sediments, and other solid wastes, require digestion prior to analysis.

B. **Detection limits, sensitivity, and optimum ranges of the metals may be found in the ICP method file.** Use of this method is restricted to spectroscopists who are knowledgeable in the correction of spectral, chemical, and physical interferences.

## II. SUMMARY OF METHOD

A. Prior to analysis, samples must be solubilized or digested using appropriate Sample Preparation Methods (e.g., Methods 3005-3050 and SOW ILM 04.1/05.2). When analyzing for dissolved constituents, acid digestion is not always necessary if the samples are filtered and acid preserved prior to analysis. If particulates form after filtration and preservation the sample must be digested prior to analysis.

NOTE: When selenium is required soluble samples must always be digested.

B. This method describes the simultaneous multi-elemental determination of elements by ICP. The method measures element-emitted light by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific atomic-line emission spectra are produced by a radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the lines are monitored by photomultiplier tubes. Background correction is required for trace element determination. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background-intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interference and reflect the same change in background intensity as occurs at the analytic wavelength measured. Background correction is not required in cases of line

broadening where a background correction measurement would actually degrade the analytical result.

C. ICP's primary advantage is that it allows simultaneous determination of any elements in a short time. The primary disadvantage of ICP is background radiation from other elements and the plasma gases. Although all ICP instruments utilize high-resolution optics and background correction to minimize these interferences, analysis for traces of metals in the presence of a large excess of a single metal is difficult. Examples would be traces of metals in an alloy or traces of metals in a limed (high calcium) waste. ICP and Flame AA have comparable detection limits (within a factor of 4) except that ICP exhibits greater sensitivity for refractories (Al, Ba, etc.). Furnace AA, in general, will exhibit lower detection limits than either ICP or FAA.

D. It is standard procedure to use an internal standard (Yttrium) with samples to increase the stability of the instrument as recommended by the manufacturer (TJA). (When samples are suspected of containing Yttrium internal standard cannot be used.)

## III. SAMPLE HANDLING AND PRESERVATION

A. Preliminary treatment of most matrices is necessary because of the complexity and variability of sample matrices. Water samples which have been prefiltered and acidified will not need acid digestion as long as the samples and standards are matrix matched and particulates do not form after the filtration and preservation take place. Solubilization and digestion procedures are presented in Sample Preparation Methods (Methods 3005A-3050A).

B. Sample digestates are stored at room temperature for at least 2 months unless a longer time is requested by the client. The samples contain an acid matrix of 3:1. Since the most concentrated acid matrix allowed for direct disposal down an acid sink is a ratio of 20:1, the samples must be diluted with 1 part water to 2 parts sample prior to pouring down the sink while the tap water is running.

C. **The appropriate SOPs should be consulted regarding sample preparation.** The following is a brief summary of the methods we use for metals preparation.

- Method 3005A prepares groundwater and surface water samples for total recoverable and dissolved metals determination by ICP. The unfiltered or filtered sample is heated with dilute HCl and $HNO_3$ prior to metal determination.

- Method 3010A prepares waste samples for total metal determination by ICP. The samples are vigorously digested with a mixture of nitric acid and hydrochloric acid followed by dilution with laboratory water. The method is applicable to aqueous samples, TCLP and mobility-procedure extracts.

- Method 3031 prepares oily waste samples for total metal determination by ICP. The samples are mixed with permanganate, $H_2SO_4$, $HNO_3$, and HCl, then filtered. The filter is then added back to the filtrate and heated with more $HNO_3$ and filtered once again. If manganese is needed a manganese removal step is necessary.
- Method 3050B prepares waste samples for total metals determination by ICP. The samples are vigorously digested in nitric acid and hydrogen peroxide followed by dilution with either laboratory water or hydrochloric acid and laboratory water. The method is applicable to soils, sludges, and solid waste samples.

## IV. INTERFERENCES

A. Spectral interferences are caused by background contribution from continuum or recombination phenomena, stray light from the line emission of high-concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.

1. Background emission and stray light can usually be compensated for by subtracting the background emission determined by measurements adjacent to the analyte wavelength peak. Spectral scans of samples or single element solutions in the analyte regions may indicate when alternate wavelengths are desirable because of severe spectral interference. These scans will also show whether the most appropriate estimate of the background emission is provided by an interpolation from measurements on both sides of the wavelength peak or by measured emission on only one side. The locations selected for the measurement of background intensity will be determined by the complexity of the spectrum adjacent to the wavelength peak. The locations used for routine measurement must be free of off-line spectral interference (interelement or molecular) or adequately corrected to reflect the same change in background intensity as occurs at the wavelength peak. For multivariate methods using whole spectral regions, background scans should be included in the correction algorithm. Off-line interferences are handled by including spectra on interfering species in the algorithm.

2. To determine the appropriate location for off-line background correction, the user must scan the area on either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. This spectral information must be documented and kept on file. The location selected for background correction must be either free of off-line interelement spectral interference or a computer routine must be used for automatic correction on all determinations. If

a wavelength other than the recommended wavelength is used, the analyst must determine and document both the overlapping and nearby spectral intereference effects from all method anlytes and common elements and provide for their automatic correction on all analyses. Tests to determine spectral interference must be done using anlyte concentrations that will adequtetely describe the interfernce. Normally, 100 mg/L single element solutions are sufficient; however, for analytes such as iron that may be found at high concentration , a more appropriate test would be to use a concentration near the upper analytical range limit.

3. Spectral overlaps may be avoided by using an alternate wavelength or can be compensated by equations that correct for interelement contributions. Instruments that use equations for interelement correction require the interfering elements be analyzed at the same time as the element of interest. When operative and uncorrected, interferences will produce false positive determinations and be reported as analyte concentrations. More extensive information on interferant effects at various wavelengths and resolutions is available in reference wavelength tables and books. Users may apply interelement correction equations determined on their instruments with tested concentration ranges to compensate (off line or on line) for the effects of interfering elements. Some potential spectral interferences observed for the recommended wavelength are listed in the method in table 2. For multivariate methods using whole spectral regions, spectral interferences are handled by including spectra of the interfering elements in the algorithm. The interferences listed are only those that occur between method analytes. Only interferences of a direct overlap nature are listed. These overlaps were observed with a single instrument having a working resolution of 0.035 nm.

4. When using interelement correction equations, the interference may be expressed as analyte concentration equivalents (i.e. false analyte concentrations) arising from 100 mg/L of the interference element. For example, assume that As is to be determined (at 193.696 nm) in a sample containing approximately 10 mg/L of Al. According to Table 2 from the method, 100 mg/L of Al would yield a false signal for As equivalent to approximately 1.3 mg/L. Therefore, the presence of 10 mg/L of Al would result in a false signal for As equivalent to approximatley 0.13 mg/L. The user is cautioned that other instruments may exhibit somewhat different levels of interferences than that shown in Table 2 from the method. The interference effects must be evaluated for each individual instrument since the intensities will vary.

5. Interelement corrections will vary for the same emission line among instruments because of differences in resolution, as determined by the grating, the entrance and exit slit widths, and by the order of

dispersion. Interelement corrections will also vary depending upon the choice of background correction points. Selecting a background correction point where an intefering emission line may appear should be avoided when practical. Interelement corrections that constitute a major portion of an emission signal may not yield accurate data. Users should not forget that some samples may contain uncommon elements that could contribute spectral interferences.

6. The interference effects must be evaluated for each individual instrument whether configured as a sequential or simultaneous instrument. For each instrument, intensities will vary not only with optical resolution but also with operating conditions (such as power, viewing height and agron flow rate). When using the recommended wavelengths, the anlaylst is required to determine and document for each wavelength the effect from referenced interferences as well as any other suspected interferences that may be specific to the instrument of matrix. The analyst is encouraged to utilize a computer routine for automatic correction on all analyses.

7. If the correction routine is operating properly, the determined, apparent analyte(s) concentration from analysis of each interference solution should fall within a specific concentration range around the calibration blank. The concentration range is calculated by multiplying the concentration of the interfering element by the value of the correction factor being tested and divided by 10. If after the subtraction of the calibration blank the apparent analyte concentration falls outside of this range in either a positive or negative direction, a change in the correction factor of more than 10% should be suspected. The cause of the change should be determined and corrected and the correction factor updated. The interference check solutions should be analyzed more than once to confirm a change has occurred. Adquate rinse time between solutions and before analysis of the calibration blank will assist in the confirmation.

8. When interelement corrections are applied, their accuracy should be verified, daily, by analyzing spectral interference check solutions. If the correction factors or multivariate correction matrices tested on a daily basis are found to be within 20% criteria for 5 consecutive days, the required verification frequency of those factors in compliance may be extended to a weekly basis. Also, if the nature of the samples analyzed is such they do not contain concentrations of the interfering elements at ± one reporting limit from zero, daily verification is not required. All interelement spectral correction factors or multivariate correction matrices must be verified and updated every six months or when an instrumentation change, such as in the torch, nebulizer, injector, or plasma conditions occurs. Standard solution should e

inspected to ensure that there is no contamination that may be perceived as a spectral interference.

B. Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by diluting the sample or by using a peristaltic pump, by using an internal standard or by using a high solids nebulizer. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, affecting aerosol flow rate and causing instrumental drift. The problem can be controlled by wetting the argon prior to nebulization, using a tip washer, using a high solids nebulizer or diluting the sample. Also it is has been reported that better control of the argon flow rate, especially to the nebulizer, improves instrument performance: this may be accomplished with the use of mass flow controllers.

C. Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from the build up of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the elements and can be minimized by flushing the system with a rinse blank between samples. The possibility of memory interferences should be recognized within an analytical run and suitable rinse times should be used to reduce them. The rinse times necessary for a particular element must be estimated prior to analysis. This may be achieved by aspirating a standard containing elements at a concentration ten times the usual amount or at the top of the linear dynamic range. The aspiration time for this sample should be the same as a normal sample analysis period, followed by analysis of the rinse blank at designated intervals. The length of time required to reduce analyte signals to within a factor of two of the method detection limit should be noted. Until the required rinse time is established, this method suggests a rinse period of at least 60 seconds between samples and standards. If a memory interference is suspected, the sample must be reanalyzed after a rinse period of sufficient length. Alternate rinse times may be established by the analyst based upon their DQOs.

D. Users are advised that high salt concentrations can cause analyte signal suppressions and confuse interference tests. If the instrument does not display negative values, fortify the interference check solution with the elements of interest at 0.5 to 1 mg/L and measure the added standard concentration accordingly. Concentrations should be within 20% of the true spiked concentration or dilution of the samples will be necessary. In the absence of measurable analyte, overcorrection could go undetected if a negative value is reported as zero.

## V. SAFETY

A. Normal accepted laboratory safety practices should be followed while performing this analysis.

1. Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of appropriate safety gloves and lab coats is highly recommended.
2. Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples that need special consideration have applicable notes on the sample logs.
3. MSDS sheets are available for all reagents and standards that have been purchased. These are located in the bookshelves in the Quality Assurance Officers office.

## VI. EQUIPMENT/APPARATUS

A. Inductively coupled argon plasma emission spectrometer: Thermo Jarrell Ash (TJA) 61E trace or equivalent.

B. Computer-controlled emission spectrometer with background correction: Thermo Jarrell Ashe (TJA) 61E trace or equivalent.

C. Radio frequency generator compliant with FCC regulations: TJA 61E trace or equivalent.

D. Argon gas supply – Liquid Argon

E. Class A volumetric flasks

F. Class A volumetric pipettes

G. Analytical balance - capable of accurate measurement to a minimum of three significant figures (.001gm): Mettler model AE100

H. Variable Eppendorf Pipettes 1000µL; 5000µL

## VII. REAGENTS AND STANDARD PREPARATION

**A. Notes**

1. Reagent Water. All references to water in the method refer to reagent grade water unless otherwise specified. Reagent water will be interference free.

2. Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. If the purity of a reagent is in question analyze for contamination. If the concentration is less than the MDL then the reagent is acceptable.

## B. REAGENTS

1. Hydrochloric acid (concentrated), HCl.
2. Nitric acid (concentrated), $HNO_3$.

## C. STANDARDS

### 1. Matrix

a. All standards contain 2% $HNO_3$ and 5% HCl.

### 2. Storage

a. The standards are stored at room temperature in 500 mL Teflon bottles.

### 3. Traceability

a. A bound logbook record shall be maintained on all reference materials. The record shall include date of receipt, source, purity, all compositional information, storage conditions and expiration date. These materials/solutions are to be identified by a unique number in the logbook as well as on the container's label.

b. All working standards made from reference materials shall be labeled with a unique ID number with complete information on preparation date, concentration of each compound, solvent, preparer's name, expiration date and the logbook where information is recorded. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded in a bound logbook. Measurements made during standards preparation (e.g., from weighing operations, volume diluted to, etc.)

shall also be recorded. There should be no container with sample, sample extract, standard solution, etc. that is not correctly labeled and properly stored.

c. The analyst must initial and date each entry made in a logbook. Each analyst must be sure to "Z" out the unused area of each logbook page.

**4. Calibration standards**

a. All standards have an acid matrix of 2% $HNO_3$ and 5% HCl and should be prepared using class A volumetric flasks, class A volumetric pipettes (or calibrated Eppendorfs).

b. STD-1 is the calibration blank: Reagent grade water **matrix matched as in (a) above. Note: when this standard is analyzed the intensities should be compared to a previous run to make sure that no contamination has occurred. Prepare this solution fresh daily.**

c. Stock QC21 solution: (100 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier and includes the following metals - Sb, As, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Li, Mg, Mn, Mo, Ni, Se, Sr, Tl, Ti, V, and Zn.

d. Stock Boron solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

e. Stock Tin solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

d. QC21 High (10 ug/mL). Dilute 50 mL of stock QC21 solution, 5 mL of stock Boron solution and 500 uL of stock Tin solution to 500 mL in a 500 mL volumetric flask, with 25 mL TRACE, Concentrated HCl and 10 mL TRACE, Concentrated HNO3. Prepare this solution fresh every three to six months.This standard is used to calibrate the following-As,B,Be,Cd,Co,Cr,Cu,Mn,Mo,Ni,Pb,Sb,Se,Sn,Ti,Tl,V,Zn.

e. Stock Silver solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

f. Stock Aluminum solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

g. Stock Calcium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

h. Stock Magnesium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

i. Stock Iron solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

j. Stock Potassium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

k. Stock Barium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

l. Stock Sodium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

m. NAK High solution: (500000 ug/L Al,Fe), (2000 ug/L Ag), (5000 ug/L Ba), (500 mg/L Mg). Dilute 25 mL stock Al, Fe, Mg; and 1 ml stock Ag solution; and 0.25 mL of stock Ba solution to 500 mL in a 500 mL volumetric flask with 25 mL TRACE, Concentrated HCl and 10 mL TRACE, Concentrated HNO3. Solution is stable for 6 months.

n. NAK 100ppm solution: (100 mg/L Ca, Na, K.) Dilute 5.0 mL stock Ca, Na, K solutions into a 500ml flask with 25 mL TRACE, Concentrated HCl and 10 mL TRACE, Concentrated HNO3. Solution is stable for 6 months.

o. Stock Arsenic solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

p. Stock Cobalt solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

q. Stock Chromium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

r. Stock Copper solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

s. Stock Manganese solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

t. Stock Nickel solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

u. Stock Lead solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

v. Stock Selenium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

w. Stock Thallium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

x. Stock Beryllium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

y. Stock Cadmium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

z. Stock Antimony solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

aa. Stock Molybdenum solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

bb. Stock Strontium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

cc. Stock Titanium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

dd. Stock Vanadium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

ee. Stock Zinc solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier.

ff. Stock Ultimate Low Standard: (200 µg/L Ag, As, Co, Cr, Cu, Mn, Ni, Pb, Se, Tl); (2500 µg/L Al); (2000 µg/L Fe); (50 µg/L Be, Cd); (10 mg/L B, Sn, Ca, Mg); (500 µg/L Sb, Zn); (1000 µg/L Mo, Sr, Ba, Ti); (200 µg/L V). Dilute 100 uL of stock Ag, As, Co, Cr, Cu, Mn, Ni, Pb, Se, V and Tl solutions; 125 µ/L of stock Al solution; 100 uL of stock

Fe solution; 5 mL of stock B solution; 500 uL of stock Sn, Ca, and Mg solutions; 250 uL of stock Sb and Zn solutions; 500 uL of stock Mo, Sr, Ba and Ti solutions; 1.0 mL of stock Cd and Be diluted to 100 mL and then use 2.5 mL of that solution diluted to 500 mL in a 500 mL volumetric flask with 25 mL TRACE, Concentrated HCl and 10 mL TRACE, Concentrated HNO3. Solution is stable for 6 months.

gg. Ultimate Low Standard: (20 µg/L Ag, As, Co, Cr, Cu, Mn, Ni, Pb, Se, Tl); (250 µg/L Al); (200 µg/L Fe); (5.0 µg/L Be, Cd); (1.0 mg/L B, Sn, Ca, Mg); (50 µg/L Sb, Zn); (100 µg/L Mn, Sr, Ba, Ti); ( 20 µg/L V). Dilute 10 mL of Stock Ultimate Low Standard to 100 mL in a 100 mL volumetric flask with 5 mL of TRACE, Concentrated HCl and 2 mL TRACE, Concentrated HNO3. This solution is prepared daily.

**Stock Ultimate Low Standard and Ultimate Low Standard are only used when specified by client.**

**5. Calibration Verification standards**

a. ICV Prep- 1.0 ml of QC-23, 1.0 ml of QC-7, 0.1 ml of 10,000 ppm Na, 0.09 ml of Fe at 10,000 ppm, and 0.09 ml of Al at 10,000 ppm diluted to 100 ml with 2% HNO3 and 5% HCL matrix. Mix well. CCV Prep- 1.0 ml of QC-21, 1.0 ml of QC-7, 1.0 ml of SN at 100 ppm, 0.1 ml of Na at 10,000 ppm, 0.09 ml of FE at 10,000 ppm,and 0.09 ml of Al at 10,000 ppm diluted to 100 ml with 2% HNO3 and 5% HCL matrix and mix well. This solution is prepared daily. These solutions are given a unique identifier and recorded in the standard log. ICV is prepared from a source independent from that used in the initial calibration.

b. The CRI solution is analyzed to check the accuracy of the instrument down near the contract required detection limits (CRDL). It is analyzed in conjunction with the interference check sample. The sample is prepared from a purchased solution which contains 120 µg/mL Sb, 100 µg/mL Co and V, 80 µg/mL Ni, 50 µg/mL Cu, 40 µg/mL Zn, 30 µg/mL Mn, 20 µg/mL As, Cr, Ag and Tl, 10 µg/mL Be, Cd and Se along with 6 µg/mL Pb. 500 µ/L of the solution is diluted to 500 mL. This solution is stable for 6 months.

b. The interference check solutions ( ICSA and ICSAB ) are prepared to contain known concentrations of interfering elements that will provide an adequate test of the IECs. A solution containing 500 ug/mL Al, Ca, Mg and 200 ug/mL Fe is diluted 10x to prepare the ICSA. The ICSAB is prepared by diluting 100x a solution containing 10 ug/mL of As and Tl; 20 ug/mL Ag; 50 ug/mL Ba, Be, Cr, Co, Cu, Mn, and V; 100 ug/mL Cd, Ni and Zn; 5 ug/mL Pb and Se; and 60 ug/L Sb. Add to this a solution containing 500 ug/mL Al, Ca, Mg and 200 ug/mL Fe diluted 10x. These solutions are prepared as needed or monthly.

d. Reporting Limit Standard- Prepared 1.0 ml of RL Stock solution A and 1.0 ml of RL Stock Solution B diluted to 100 ml with 2% HNO3 and 5% HCL matrix ; mix well. Solution stable for 3 months

**6. Digestion standards**

a. The Laboratory control sample ( LCS ) is prepared from High Purity solutions CLP-CAL-1 solution A and B; CLP-CAL-2 and CLP-CAL-3. 0.50 mL of CLP-CAL-1 A and B is diluted to 500 mL with 0.125 mL of CLP-CAL-2 and CLP-CAL-3. 25 mL of HCl and 10 mL of HNO3 are added for preservation. This solution is stored in a Teflon bottle. A portion is reserved in case of a problem with digestion. When there is a problem with the analysis of the LCS the solution is checked first before action is taken to make sure that it was made properly and has not deteriorated since it was made up. This solution is given a unique identifier. The LCS is prepared from a source independent from that used in the calibration standards. This solution is prepared daily or as needed. Note: The analysis of Molybdenum is not a routine procedure but a project-specific requirement. A customized LCSW mix must be prepared to contain this target analyte.

b. The solid Laboratory Control Sample (Soil) (LCSS) is prepared by weighing up 1.0 g of teflon chips and spiking using the same spiking solutions used to spike the sample matrix. This standard is given a unique identifier i.e. LCSS(date prepared)A,B,C etc.

c. The spiking solutions are prepared as follows:

1. Stock Multi-element Spiking Solutions: High Purity <u>CLP-CAL-1</u> <u>solution A</u>: 2000 ug/mL Al and Ba; 50 ug/mL Be; 200 ug/mL Cr; 500 ug/mL Co, Mn, Ni, V and Zn; 250 ug/mL Cu; 1000 ug/mL Fe; 5000 ug/mL Ca, Mg, K and Na; <u>solution B</u>: 250 ug/mL Ag; <u>CLP-CAL-2</u>: 1000 ug/L Sb; <u>CLP-CAL-3</u>: 1000 ug/mL As, Pb, Se, Tl; 500 ug/mL Cd. Order from the manufacturer already prepared. These solutions are given a unique identifier. Add 0.050 mL (0.20 mL for soil samples) of CLP-CAL-1 solutions A and B, and 0.0125 mL (0.05 mL for soil samples) of CLP-CAL-2 and 3 to 50 mL of sample (1gram of sample for soils) for the following spike values: 2000 ug/L Al and Ba; 50 ug/L Be; 200 ug/L Cr; 500 ug/L Co, Mn, Ni, V and Zn; 250 ug/L Cu; 1000 ug/L Fe; 5.0 mg/L Ca, Mg, K and Na, 250 ug/L Ag, Sb, As, Pb, Se and Tl; 125 ug/L Cd. A blank spike should be prepared at the time the samples are spiked to check the actual spike value and accuracy.

2. TCLP Spiking Solution: Use 0.50 mL diluted to 50 mL for digestion:
2.5 mL 10000 mg/L Ba stock standard diluted to 100 mL; 2.5 mL Cr, Pb and As 1000 mg/L stock standard diluted to 100 mL; 0.50 mL Cd and Se diluted to 100 mL. Store in a Teflon bottle. A blank spike should always be prepared at the same time a sample is being spiked. This solution should produce a spike value of 2500 ug/L Ba; 250 ug/L Cr, Pb and As; and 50 ug/L of Cd and Se. Note: Since the samples are diluted 10x when digested the spike value will appear to be 10x greater when analyzed.

3. TCLP Silver Spiking Solution: Use 5.0 mL diluted to 50 mL for digestion:
0.40 mL of 1000 mg/L stock Ag solution diluted to 200 mL. Store in a Teflon bottle. A blank spike should always be prepared at the same time a sample is being spiked. This solution should produce a spike value of 200 ug/L. . Note: Since the samples are diluted 10x when digested the spike value will appear to be 10x greater when analyzed. Also this solution is not very stable and may require fresh preparation at least weekly.

## VIII. CALIBRATION AND ASSOCIATED QA/QC

A. Set up the instrument with proper operating parameters. The instrument must be allowed to become thermally stable before beginning (usually requiring at least 30 minutes of operation prior to calibration).

B. Operating conditions - **The instrument settings can be found in method file.** For operation with organic solvents, use of the auxiliary argon inlet is recommended, as are solvent-resistant tubing, increased plasma (coolant) argon flow, decreased nebulizer flow, and increased RF power to obtain stable operation and precise measurements. Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects must be established for each individual analyte line on that particular instrument. The analyst must (1) verify that the instrument configuration and operating conditions satisfy the analytical requirements and (2) maintain quality control data confirming instrument performance and analytical results.

C. Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the typical mixed calibration standard solutions. **(See SOP-106, *ICP Instrument Operation*)** Flush the system with 2% $HNO_3$ / 5% HCl between each standard or as the manufacturer recommends. (Use the average intensity of multiple exposures for both standardization and sample analysis to reduce random error.) The calibration curve consists of a blank and three standards ($r \geq 0.995$). If a three point

calibration curve is not required for the client samples being analyzed Empirical Laboratories may use a blank and one standard as referenced in USEPA - CLP protocols.

D. Before beginning the sample run, analyze the Iron and Aluminum standards at their linear range to check for IEC drifts. Analyze these standards first as QC samples with an IEC check table and action taken should be to calculate IECs. Then reanalyze the standard as a sample to check to see if the correction was made successfully. If the IEC correction overcompensated or under compensated for any one metal which is affected by it, the standard should be run again as a QC as before. Make sure to rinse thoroughly after running these linear range standards, they can cause carry over into the initial QC samples which are analyzed next. The analysis order follows as: ICV (± 10%) for 200.7 (± 5%) and ICB (< ±MDL or ±RL/CRDL for others or CLP, **for CCB, DOD QSM Ver. 3 no analytes detected >2xMDL**) first, then reanalyze the highest mixed calibration standard(s) as if it were a sample. Concentration values obtained should not deviate from the actual values by more than 5%. If they do, follow the recommendations of the instrument manufacturer to correct for this condition.

E. For CLP projects,verify the validity of the curve in the region of 2x the contract required detection limit ( CRDL ) before and after each batch of 20 samples in the specific order of CRI, ICSA, ICSAB, CCV and CCB(CCB criteria: < ±MDL or ±RL/CRDL for others or CLP, **for CCB, DOD QSM Ver. 3 no analytes detected >2xMDL, beginning and end of sequence and after every 10 samples**) or twice during every 8-hour work shift, whichever is more frequent. Results should be within ±20%. Supervisor must be notified if the control limit is not met. Supervisor will dictate corrective action if required. The final analytical report must document this situation. (For Internal QC)

F. Verify the interelement and background correction factors at the beginning and after each batch of 20 samples in the specific order of CRI, ICSA, ICSAB, CCV and CCB(CCB criteria: < ±MDL or ±RL/CRDL for others or CLP, **for CCB, DOD QSM Ver. 3 no analytes detected >2xMDL, beginning and end of sequence and after every 10 samples**) or twice during every 8-hour work shift, whichever is more frequent. Do this by analyzing the interference check solution A and AB. Results should be within ±20% of the true value for ICSAB. **For ICSA DOD QSM Ver 3. , absolute value of concentration for all non-spiked analytes < 2xMDL**.( ICSAB required at the end for CLP projects)

G. *When analyzing samples associated with North Carolina or with DOD QSM Ver. 3 work, a solution containing analytes at their reporting limit must be analyzed prior to sample analysis. The concentrations must be within 20% DOD( 20 or 30% depending on project) of their true values to be acceptable.*

Note: Supervisor must be notified if the control limit is not met. Supervisor will dictate corrective action if required. The final analytical report must document this situation.

H. The instrument must be calibrated once every 24 hours if performing straight CLP work.

I. **Note: The instrument software is set up to go to the "L" rack for calibration standards, initial IEC standards and initial and continuing calibration verifications (ICV/CCV). This QC is inserted by the analyst when setting up the autosampler table which is what the instrument software uses for a RUN LOG (The software does not allow the analyst to print the actual run log but prints an autosampler report). The autosampler report is used to load the instrument and does not include ICV and CCVs in racks 2 through 5 because all calibration QC is loaded in the "L" rack and then samples and CCB's are loaded in Racks 2 through 5. When the samples are analyzed the software inserts ICVs and CCVs from the actual run log set up by the analyst. Since each CCV location is visited twice by the autosampler during the run, two locations will be called CCV1 and two locations will be CCV2 and so on. The time and date are printed on the instrument print out with the data so that the data user can distinguish between the two CCVs.**

J. Instrument Autosampler Report example:

**Calibration or "L" Rack(used by instrument software to insert QC)**

1) STD 1-blank
2) NAK-100ppm
3) NAK-high
4) Fe-500,000ppb
5) Mn-10,000ppb
6) QC 21-high
7) Al IEC-QC(calculate IEC)
8) FE IEC-QC(calculate IEC)
9) ICV
10) CCV1
11) CCV2
12) CCV3

**Sample Sequence RACK 1**

1) Al IEC-(readback)
2) Fe IEC-(readback)
3) ICB-initial
4) RL-reporting limit standard

5) NAK 100ppm-(readback)

**Sample Sequence RACK 1 (cont.')**

6) NAK High-(readback)
7) QC 21 High-(readback)
8) Rinse
9) CRI-0
10) ICAS-0
11) ICASB-0
12) Rinse
13) CCB 1A
14) Preparation Blank
15) Laboratory Control Sample
16) Sample 1
17) Sample 2
18) Sample 3
19) Sample 4
20) Sample 5
21) Sample 6
22) Sample 7
23) Sample 8
24) CCB 1B
25) Sample 9
26) Sample 10
27) Sample 11
28) Sample 12
29) Sample 13
30) Sample 14
31) Sample 15
32) Sample 16
33) Sample 17
34) Sample 18
35) CCB2A
36) Sample 19
37) Sample 20
38) Sample serial dilution
39) Sample matrix spike
40) Sample matrix spike duplicate
41) Sample post digestion spike
42) CRI-1
43) ICSA-1
44) ICSAB-1
45) Rinse
46) CCB 2B
47) Preparation Blank
48) Laboratory Control Sample

**RACK 2**

1) Sample 1
2) Sample 2
Etcetera…

Each rack holds 48 samples and there are 4 racks that are used for samples and CCBs and run QC other then ICV/CCV's.

## IX. PROCEDURE

A. Once the instrument has been calibrated, begin the analysis of samples.

B. If particulates are visible in the digestate, the sample must be filtered prior to analysis. If filtration is required, a filter blank must be prepared by filtering reagent grade water which has been properly acidified. **In the event USACE samples are filtered, all USACE samples and the QC samples in that QC batch must be filtered. All USACE solid samples and their associated batch QC samples must be filtered prior to analysis.**

C. Flush the system with 2% $HNO_3$ / 5% HCl for at least 1 minute before the analysis of each sample.

D. Dilute and reanalyze samples that are more concentrated than the linear calibration limit or, for 200.7, ± 10% of the linear range standard. **In the case of USACE samples, the criterion changes and requires dilution and reanalysis of all samples which produce a concentration that exceeds the highest calibration standard. Sample results detected between the MDL and RL are flagged as estimated with a "B" flag.**

E. Verify calibration every 10 samples or every 2 hours, whichever is more frequent and at the end of the analytical run, using a continuing calibration verification (CCV) sample and a continuing calibration blank (CCB) sample.

- The results of the CCV are to agree within 10% for 6010 (5% for 200.7) on initial verification of the expected value, with relative standard deviation (RSD) < 5% from replicate ( minimum of two integrations ). If not, terminate the analysis, correct the problem, and reanalyze the previous ten samples. The analyst may continue the analytical run, and after conferring with the section manager it may be necessary to reanalyze a group of samples. The analyst must notify the section manager within 24 hours.

- The results of the calibration blank (this is not the method/preparation blank) are to agree within < ±MDL(SW-846 Method 6010B), and 3 x IDL or CRDL for CLP, for **DOD QSM Ver. 3 no analytes detected**

**>2xMDL**. If the calibration blank is not in control, evaluate the impact upon the previous 10 samples. Reanalysis may be required after an evaluation of the data. If the blank < 1/10 the concentration of the action level of interest, and no sample is within 10% of the action limit, samples need not be reanalyzed. One must also evaluate the reporting limit (RL) as it relates to 3X the IDL/MDL. If the RL is significantly above 3X IDL or MDL then reanalysis may not be required (Na, K, Mg and Ca are good examples of this situation).

- Total hardness is reported from $HNO_3$ preserved sample. The final concentration is calculated from the calcium and magnesium results as follows:

Ca mg/L x 2.5 + Mg mg/L x 4.1 = total Hardness in mg/L as $CaCO_3$

F. Documentation of Capability (DOC) – Each analyst must perform a DOC to demonstrate proficiency with this method. Refer to SOP-413 for guidance.

## X. CALCULATIONS

A. The instrument will generate data results in mg/L or µg/L ( labeled appropriately). Each result represents an average of three individual readings per metal channel.

B. For aqueous samples, if a post/predigestion dilution is performed , the result must be multiplied by this factor or the dilution factor must be entered into the instrument data table in which case the instrument will generate data corrected for the dilution.

C. For solid samples, if a postdigestion dilution is performed , the result must be multiplied by this factor or the dilution factor must be entered into the instrument data table in which case the instrument will generate data corrected for the dilution. Also, the result must be converted to reporting units which are usually mg/kg.

SR ( ug/g or mg/kg ) = IR*DF*FED/SM

SR = Sample result
IR = Instrument result ( µg/L )
DF = Dilution factor ( post digestion )
FED = Final volume of digestate ( L )
SM = Sample mass digested( g )

## XI. QUALITY CONTROL

**A. Daily**

1. See sections VIII and IX above.

**B. Quarterly**

1. Linear range standards must be analyzed at a frequency no less than once every three months. The linear range standard represents the second standard required for verification that samples are actually linear to the degree claimed. The analyst is responsible for completing this task in a timely manner. The linear range standard must be within +/-5% of true value.
2. The interelement correction factors ( IEC ) should be verified at the time the linear range standards are analyzed.
3. IDL's if CLP work required.

**C. Digestion**

1. All quality control data should be maintained and available for easy reference or inspection.
2. Employ a minimum of one method blank per sample batch to determine if contamination or any memory effects are occurring. A method blank, sometimes referred to as the preparation blank is a volume of reagent water acidified with the same amounts of acids as were the standards and samples. These blanks are taken through the same digestion/preparation steps as the sample being tested. The result for the method blank should not indicate contamination greater than ± ½ RL (USACE) or ±RL/CRDL for other or CLP. If exceeded, the impact upon the data should be evaluated and the associated sample(s) should be either redigested or the data should be qualified.
3. Employ a minimum of one laboratory control sample ( LCS ) for aqueous samples or one teflon chip spiked sample per sample batch to verify the digestion procedure. These LCSs are taken through the same digestion/preparation steps as the sample being tested. The control limits are ±15% method 200.7 - aqueous and soil samples or ±20% for all other methods aqueous and soil samples. If the LCS is not in control, the impact upon the client data should be evaluated and the associated sample(s) should be redigested. Consult your supervisor for further action. Qualifying the associated data may not be permissable for CLH.

**D. Sample**

1. Analyze one replicate sample for every twenty samples or per analytical batch, whichever is more frequent. A replicate sample is a sample brought through the whole sample preparation and analytical process in duplicate. It is acceptable to substitute a matrix spike duplicate for the sample

replicate. Project specific requirements will take precedence in these situations. NJDEP demands that this requirement be met with a client specific duplicate rather than a spike duplicate. The control limits are 20% RPD (if both are >5x CRDL) or ± the CRDL ( if either are <5X CRDL).

2. Analyze one spiked sample and spiked sample duplicate for every twenty samples or per analytical batch, whichever is more frequent. A replicate sample is a sample brought through the whole sample preparation and analytical process in duplicate. Project specific requirements will take precedence in these situations. If the analyte level in the sample is not greater than 4X the spiking level, the spike recoveries should be within ±25% of the true value (**± 20% for DOD projects**). If not, a post digestion spike should be analyzed.

3. The relative percent difference (RPD) between replicate determinations is to be calculated as referenced in the laboratory QA manual. (A control limit of ± 20% RPD (non-aqueous samples may routinely exceed this amount) shall be used for sample values greater than five times the contract required detection limit.) Supervisor must be notified if the control limit is not met. Supervisor will dictate corrective action if required. The final analytical report must document this situation.

4. *The following should be analyzed with each preparation batch containing a matrix spike.*

- Serial dilution: If the analyte concentration is sufficiently high (minimally, a factor of 50 above the instrumental detection limit after dilution), an analysis of a 1:4 dilution (volumetric glassware must be used) should agree within ±10% of the original determination. If not, a chemical or physical interference effect should be suspected. The analyst and or section manager must note this situation on the final analytical report.

- Post digestion spike addition: An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 85% to 115% of the known value and is required if the pre-digestion matrix spike (low-level only for CLH) is outside of control limits. The spike addition should produce a minimum level of 10 times and a maximum of 100 times the instrumental detection limit. If the spike is not recovered within the specified limits, a matrix effect should be suspected. The analyst and or section manager must note this situation on the final analytical report.

**E. Method Detection Limit (MDL), Empirical Laboratories Reporting Limit (ERL), Contract Required Quantitation Limit (CRQL) and Analyte Wavelength:**

## TABLE I

**Aqueous and Soil Method Detection Limits(MDL), Empirical Laboratories Reporting Limits(ERL), CLP OLM04.1 & OLM05.2 Contract Required Quantitation Limits (CRQL)**

| Analytes by EPA 200.7,3005A/3050A- 6010B SOW 4.1 & 5.2 | AQUEOUS MDL (ug/L) | AQUEOUS ERL (ug/L) | AQUEOUS CRQL ILMO 4.1 (ug/L) | AQUEOUS CRQL ILMO 5.2 (ug/L) | SOLID/SOIL MDL (mg/Kg) | SOLID/SOIL ERL (mg/Kg) | SOLID/SOIL CRQL ILMO 4.1 (mg/Kg) | SOLID/SOIL CRQL ILMO 5.2 (mg/Kg |
|---|---|---|---|---|---|---|---|---|
| **Silver** | **1.0** | **10** | **10** | **10** | **0.20** | **2.0** | **2** | **2** |
| **Aluminum** | **50** | **200** | **200** | **200** | **10** | **40** | **40** | **40** |
| **Arsenic** | **3.0** | **10** | **10** | **15** | **0.6** | **2.0** | **2** | **3** |
| **Barium** | **5.0** | **200** | **200** | **200** | **1.0** | **40** | **40** | **40** |
| **Beryllium** | **1.0** | **5.0** | **5** | **5** | **0.20** | **1.0** | **1** | **1** |
| **Calcium** | **200** | **5000** | **5000** | **5000** | **20** | **1000** | **1000** | **1000** |
| **Cadmium** | **1.0** | **5.0** | **5** | **5** | **0.20** | **1.0** | **1** | **1** |
| **Cobalt** | **5.0** | **50** | **50** | **50** | **1.0** | **10** | **10** | **10** |
| **Chromium** | **2.0** | **10** | **10** | **10** | **0.40** | **2.0** | **2** | **2** |
| **Copper** | **4.0** | **25** | **25** | **25** | **0.40** | **5.0** | **5** | **5** |
| **Iron** | **30** | **100** | **100** | **100** | **1.0** | **20** | **20** | **20** |
| **Potassium** | **200** | **5000** | **5000** | **5000** | **40** | **1000** | **1000** | **1000** |
| **Magnesium** | **200** | **5000** | **5000** | **5000** | **40** | **1000** | **1000** | **1000** |
| **Manganese** | **1.0** | **15** | **15** | **15** | **0.20** | **3.0** | **3** | **3** |
| **Sodium** | **200** | **5000** | **5000** | **5000** | **40** | **1000** | **1000** | **1000** |
| **Nickel** | **3.0** | **40** | **40** | **40** | **1.0** | **8.0** | **8** | **8** |
| **Lead** | **1.5** | **5.0** | **3** | **10** | **0.60** | **2.0** | **0.6** | **2** |
| **Selenium** | **3.0** | **10** | **5** | **35** | **0.60** | **2.0** | **1** | **7** |
| **Antimony** | **5.0** | **60** | **60** | **60** | **1.0** | **12** | **12** | **12** |
| **Thallium** | **3.0** | **10** | **10** | **25** | **0.60** | **2.0** | **2** | **5** |
| **Vanadium** | **5.0** | **50** | **50** | **50** | **1.0** | **10** | **10** | **10** |
| **Zinc** | **5.0** | **20** | **20** | **60** | **1.0** | **4.0** | **4** | **12** |

## TABLE 2

| METAL | WAVELENGTH |
|---|---|
| Aluminum | 308.2 |
| Antimony | 206.8 |
| Arsenic | 189.0 |
| Barium | 493.4 |
| Beryllium | 313.0 |
| Cadmium | 226.5 |
| Calcium | 317.9 |
| Chromium | 267.7 |
| Cobalt | 228.6 |
| Copper | 324.7 |
| Iron | 271.4 |
| Lead | 220.3 |
| Magnesium | 279.0 |
| Manganese | 257.6 |
| Nickel | 231.6 |
| Potassium | 766.4 |
| Selenium | 196.0 |
| Silver | 328.0 |
| Sodium | 330.2 |
| Thallium | 190.8 |
| Vanadium | 292.4 |
| Zinc | 206.2 |

## XII. CORRECTIVE ACTIONS

### A. INSTRUMENT RELATED

1. ICV not within ± 10% or ± 5% for 200.7
   a. Is the problem with the solution?
      i. Reprepare or obtain new stock.
   b. Is the problem with the calibration?
      i. Recalibrate through analysis of appropriate standards and recheck ICV.

2. ICB not ±MDL or within ± 3X IDL or CRDL for CLP, **DOD QSM Ver. 3 no analytes detected >2xMDL**

a. Is the problem with the solution?
   i. Reprepare
b. Is the problem with the calibration?
   i. Recalibrate with the blank solution or the low level standard. Restart analysis with the ICV.

3. Check standards not within ± 5%
   a. Is the problem with the solution?
      i. Repour, reprepare or obtain new stock.
   b. Is the problem with the calibration?
      i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.

4. CRI not within ± 20% (Internal QC, only required for CLP work).
   a. Is the problem with the solution?
      i. Reprepare or obtain new stock.
   b. Is the problem with the calibration?
      i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.

5. ICSA metals not present are not less than the CRDL for that metal, **for ICSA DOD QSM Ver 3. , absolute value of concentration for all non-spiked analytes < 2xMDL.**
   a. Is the problem with the solution?
      i. Reprepare or obtain new stock.
   b. Is the problem with the calibration?
      i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.

6. ICSAB not within ± 20%
   a. Is the problem with the solution?
      i. Reprepare or obtain new stock.
   b. Is the problem with the calibration?
      i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.

7. CCV not within ± 10%
   a. Is the problem with the solution?
      i. Reprepare or obtain new stock.
   b. Is the problem with the calibration?
      i. If appropriate, continue the analysis. Discuss effect of the out of control situation with your supervisor. The samples will be reanalyzed or the data will be qualified. Note: CLH data must always be reanalyzed back to the last compliant CCV and not qualified.

8. CCB not ±MDL or within ± 3X IDL or CRDL for CLP, **DOD QSM Ver. 3 no analytes detected >2xMDL**

a. Is the problem with the solution?
   i. Reprepare
b. Is the problem with the calibration?
   i. Apply SW846 guidance. (See Section IX-E for additional guidance). Note: CLH data must always be reanalyzed back to the last compliant CCB and not qualified.

## B. DIGESTION RELATED

1. Preparation blank not within ± ½ RL and ± RL for common contaminants USACE or RL/CRDL for other or CLP
   a. Is the problem with the instrument?
      i. Evaluate with respect to instrumental bias or reanalyze when instrument is in control.
   b. Is the problem with the digestion?
      i. If associated samples are less than 10X the level of the preparation blank but above the RL, the sample must be redigested or the data must be qualified on the final report.

2. LCS not within control limits
   a. Is the problem with the instrument?
      i. Evaluate with respect to instrumental bias or reanalyze when instrument is in control.
   b. Is the problem with the digestion?
      i. If biased low, associated samples must be redigested.
      ii. If biased high, the impact upon the data user must be evaluated. The samples will be redigested or the data will be qualified on the final report.

## C. SAMPLE MATRIX RELATED

1. Replicate analysis RPD not within ±20% (if both are >5X CRDL) or ± the CRDL ( if either are <5X CRDL).
   a. The associated sample data must be qualified on the final report.

2. Spike analysis recovery not within ±25%(**± 20% for DOD projects**)
   a. Is the analyte level in the sample greater than 4X the spiking level?
      i. If yes, the spike recovery is not evaluated.
      ii. If no, a post digestion spike must be analyzed and the associated sample data must be qualified on the final report.

3. When required, post digestion spike analysis recovery not within ±15%.
   a. The associated sample data must be qualified on the final report.
   b. For USACE analysis by MSA is required.

4. Serial dilution analysis percent difference not within ±10%
   a. Is the analyte concentration a factor of 50 above the instrumental detection limit after dilution?
      i. If no, the serial dilution data can not be evaluated.
      iii. If yes, a chemical or physical interference effect should be suspected. The analyst and or section manager must note this situation on the final analytical report.

## XIII. WASTE DISPOSAL and POLLUTION PREVENTION

Please see Waste Disposal SOP-405 fot instruction of proper disposal of waste generated from this area.

Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

## XIV. REFERENCES

1. *Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III)*; Method 6010B

2. USEPA *Code of Federal Requlations, 40, CH 1,PT 136;* Method 200.7; APX-B

3. USEPA *Contract Laboratory Program(CLP) for Inorganics ILM04.1; ILM05.2*

Refer to SOP-431 for common environmental laboratory definitions.

## Addendum for USEPA CLPILM 05.2

1. The control limit for the ICSA is at 20% or ±CRQL whichever is greater.
2. Preparation codes are required in the digestion log See SOW Exhibit B for a listing of these codes with definitions.

3. The CRQL check standard is run at the concentration of the respective CRQLs. For a listing of CRQL for this SOW see Exhibit C. Several of the metals concentration levels have changed.
4. The spiking level for CLP ILM 05.2 is at 50 ug/L for selenium. All other spike levels remain the same as in SOW ILM 04.1.
5. The CCV shall be analyzed at a different concentration then the ICV (at or near one-half of the calibration standard concentration.
6. The post digestion spike must be analyzed at 2x the indigenous level of the sample or two times the CRQL whichever is greater.
7. A Non-prepared MDL study must be analyzed and the results of this study used for MDL reporting when sample volumes are not digested.

## CHANGES TO FORMS for SOWCLPILM 05.2

1. Forms must be double-sided
2. A photocopy of the instrument's direct sequential readout shall be included.
3. Undiluted samples must be reported as well as diluted samples.
4. J flags are used in place of B flags when a sample has a concentration less the CRQL but greater then or equal to the MDL.
5. A D flag is used for samples reported from a dilution.
6. All results are reported down to the MDL not the IDL.
7. Preparation codes are used on form 13.

   The form for method of standard additions (MSA) has been removed and all subsequent QC has move up one form number in other words form 8 is now serial dilution when it used to be the MSA form,etcetera.

**ANALYST DATA REVIEW CHECKLIST Sample Number(s):**

**Batch Number(s):**

**Method: 6010B (ICP)**

| QA/QC Item | Yes | No | NA | Second Level Review |
|---|---|---|---|---|
| 1. Were samples analyzed within USACE holding times? | | | | |
| 2. Was initial calibration curve QC criteria met? | | | | |
| 3. Was all continuing calibration criteria in control? | | | | |
| 4. Did any sample exceed the highest calibration standard? (If yes, were appropriate dilutions made to generate samples concentration within calibration range?) | | | | |
| 5. Did LCS or blank spike meet control limits? | | | | |
| 6. Did MS/MSD meet control limits? | | | | |
| 7. Was the preparation (Method) Blank below the project required detection limits? | | | | |
| 8. Did you return samples back to cold storage immediately after use? | | | | |
| 9. Was hot plate temperature monitored/documented and did you apply the thermometer correction factor? | | | | |
| 10. Sample preparation information is correct and complete. | | | | |
| 11. Analytical results are correct and complete. | | | | |
| 12. The appropriate SOP's have been used and followed. | | | | |
| 14. "Raw data" including all manual integration's have been correctly interpreted. | | | | |
| 15. "Special" sample preparation and analytical requirements have been met. | | | | |
| 16. Documentation complete (e.g., all anomalies in the analytical sequence have been documented, corrective action forms are complete. | | | | |

Comments on any "No" response:

Analyst: _____ Date: _____

Second-Level Review: _____ Date: _____

Empirical Laboratories, LLC

# ELECTROMETRIC DETERMINATION OF pH

## METHODS
## (USEPA) 150.1, Standard Methods 4500-H⁺B AND 9040B FOR WATERS, LIQUIDS AND LIQUID WASTES, 9045C FOR SOILS AND SOIL WASTES

**SOP NUMBER:** SOP-187

**REVISION NUMBER:** 6

**APPROVED BY:**

SECTION MANAGER

TECHNICAL DIRECTOR

**EFFECTIVE DATE:** 09/05/06

**DATE OF LAST REVIEW:** 02/08/08

**Empirical Laboratories, LLC**

# ELECTROMETRIC DETERMINATION OF pH

## I. SCOPE AND APPLICATION:

A. This SOP is specifically for the Orion 420A pH meter with Orion Triode pH electrode.

B. USEPA Method 150.1/SM4500-$H^+$B is applicable to drinking, surface, and saline waters, domestic and industrial wastes and acid rain.

C. Method 9040B is applicable to aqueous wastes and those multiphase waste where the aqueous phase constitutes at lease 20% of the total volume of the waste. This method also fulfills the requirement for characterization of Corrosivity according to SW846.

D. Method 9045C is applicable to soils and waste samples. Wastes may be solids, sludges, or non-aqueous liquids. If water is present, it must constitute less than 20% of the total volume of the sample.

## II. METHOD SUMMARY:

The pH of a sample is determined electrometrically using either a glass electrode in combination with a reference potential or combination electrode.

## III. SAMPLE HANDLING AND PRESERVATION:

A. pH, when used for permit reporting on water samples, is intended to be a field test. It is often monitored on a continuous basis when 24 hour composites are collected. If the pH is to be analyzed on a discreet grab sample, most States require that analysis occur within 15 minutes of collection to be reported without qualification for regulatory purposes. Nevertheless, occasionally, samples will be transported to the laboratory for analysis. Frequently this is done for the information purposes only, to be used by the client or the laboratory for the purposes other than regulation. To avoid confusion or misuse of the data, these analyses will clearly be labeled as "laboratory pH" on the final report. They must be analyzed as soon as possible in the laboratory. Soil or solid samples are subject to the sample holding time restrictions, and should also be analyzed as soon as possible and qualified on the final report.

B. High-purity waters and waters not at equilibrium with the atmosphere are subject to changes when exposed to the atmosphere, therefore the sample containers should be filled completely and kept sealed prior to analysis.

C. Samples must be unpreserved.

## IV. INTERFERENCES:

A. The glass electrode, in general, is not subject to solution interferences from color, turbidity, colloidal matter, oxidants, reductants, or high salinity.

B. Sodium error at pH levels greater than 10 can be reduced or eliminated by using a "low sodium error" electrode.

C. Coatings of oily material or particulate matter can impair electrode response. These coatings can usually be removed by gently wiping the electrode or washing it with a detergent followed by rinsing with distilled water. An additional rinsing with dilute Hydrochloric Acid (1:10) may also be necessary to remove any remaining film.

D. Temperature fluctuations will cause measurement errors, however pH measurements made with the Orion 420A meter and the Orion Triode electrode have automatic temperature compensation features.

E. A dirty electrode may also cause problems. See the maintenance sections or the electrode manual.

## V. EQUIPMENT/APPARATUS:

A. pH meter: This SOP is specifically for the Orion 420A. This meter has set-up features which have been pre-set. In order to change these, refer to the instruction manual, pages 8-9. This meter has an automatic temperature compensation feature.

B. Electrode: There are many different types of pH electrodes available. This SOP is for the Orion Triode pH electrode which has an epoxy outer body and contains an internal filling solution of Ag/AgCl.

NOTE: REFER TO THE INSTRUCTION MANUAL FOR ANY QUESTIONS OR PROBLEMS WITH EITHER THE METER OR THE ELECTRODE. ALSO REFER TO THE INSTRUCTION MANUAL FOR PROPER CARE AND MAINTENANCE OF THE pH METER AND PROBE.

C. Magnetic stirrer and Teflon-coated stirring bars. Put a piece of cardboard between the stirrer and the beaker containing the sample so that the heat from the stirrer will not affect the temperature of the sample.

D. Small beakers or cups.

**Empirical Laboratories, LLC**

## VI. REAGENTS:

A. Standard buffers: 4, 7, and 10. The pH meter must be calibrated using standard buffers daily or, if not daily, with each use. Separate solutions of the three buffers should be kept in 120 ml. plastic bottles with the caps on when not in use. Pour up fresh buffers each use. (Record the date in the log book).
An additional pH 12 buffer is required for the Corrosivity test of alkaline wastes per Method 9040B.

B. Reference electrode filling solution.

1. Use Orion #900011 which contains AgCl. Make sure that the filling solution is the correct one for your electrode. If you use the wrong solution you may damage the electrode.

2. Never allow the electrode to dry out.

3. Change the filling solution at least once a month. (Record the date in the log book). When you change the filling solution you should soak the electrode in pH 7 buffer for at least one hour before analyzing samples.

C. Deionized water.

D. Hydrochloric acid (HCl): 1:3 mixture with reagent water.

## VII. CALIBRATION:

A. Initial calibration (2 point)

This meter is capable of both autocalibration and manual calibration. We calibrate manually.

1. Turn the power on. It will display a quick check mode and then the reading will stabilize. The meter should be in the pH mode, if not, press the "mode" key until the pH mode indicator is displayed.

2. Uncover the hole on the upper part of the electrode. This allows a uniform flow of the filling solution. (It should be kept covered when not in use.)

3. Rinse the electrode with deionized water and blot dry with a kimwipe (do not wipe the electrode).

4. The meter must be calibrated prior to use with two pH buffer standards which bracket the expected value of the sample to be

measured (4 and 7, or 7 and 10). Place the electrode in pH 7 buffer first, with stirring bar turning. Make sure the electrode is not touching the bottom or sides of the beaker. Buffers must be at room temperature.

5. Press the "2nd" key, followed by the "cal" key. It will display the time and date of the last calibration and then "P1" will be displayed in the lower field -- this means it is ready to read the first calibration standard.

6. Wait for a stable pH display and the meter will say "ready". Press the "no" key. The first digit will start flashing. Scroll up or down using the arrow-head keys until the correct value appears in the first digit (it will be 7 for the 7.00 buffer) then press "yes". Continue in the same manner until all the digits have been correctly entered, then press "yes" to enter the new value. The display will remain frozen for a few seconds, then "P2" will be displayed which means that the meter is ready to read the second calibration standard.

**NOTE:** Due to the temperature compensation of the meter, at 20° C the 7 buffer should read 7.01.

7. Rinse the electrode and place in the pH 4 **or** 10 buffer (depending on the expected pH of the sample to be analyzed) with stirring bar turning. Wait for a stable pH reading and then enter the correct value as you did for the 7 buffer.

**NOTE:** As with the 7 buffer, due to the temperature compensation of the meter, at 20° C the 4 buffer should read 4.00 and the 10 buffer should read 10.05.

8. After both points have been calibrated, the electrode slope will be displayed in the main field. The slope should be between 92 and 102. (Record the slope on the data sheet.) If it is not within this boundary you should inspect the electrode and meter and recalibrate with fresh buffers. If the slope is still out of range, check the manual for troubleshooting.

9. The meter will automatically advance to the "measure" mode and you are now ready to check the calibration and read the samples.

B. Calibration for Corrosivity characterization by Method 9040B

1. For corrosivity characterization, the calibration of the pH meter should include a buffer of pH 2 for acidic wastes and a pH 12 buffer for caustic wastes; also, for corrosivity characterization, the sample must be measured at 25±1 °C if the pH of the waste is above 12.0. Repeat adjustments on successive portions of the two buffer solutions until

2. The reference chamber hole should be covered overnight and the power to the pH meter should be turned off.

3. White crystals will probably form on the electrode. These are from the potassium chloride and will not damage the electrode, however, the electrode should be rinsed with distilled water to get rid of this build-up.

4. If storing the electrode for more than one week, the reference chamber should be filled and the fill hole should be securely covered. Cover the sensing element with its protective cap containing a few drops of storage solution. Before reusing it, prepare it as a new electrode. (See manual).

B. Maintenance

1. Drain the reference chamber, flush it with fresh filling solution and refill the chamber. Allow the electrode to set overnight before using it. This should be done on a monthly basis. Record the date in the log book.

2. Inspect the electrode for scratches, cracks, salt crystal build-up, and general physical integrity.

3. If there is build-up of salt or any grease or dirt on the electrode it may be necessary to soak it in dilute HCl (1:10) for half an hour. Then drain and refill the reference chamber and soak the electrode in storage solution for at least half an hour before using. Also record this in the log book.

For more information on storage and maintenance of the electrode, see the instruction manual.

## X. QUALITY CONTROL:

A. Analyze a laboratory control sample (LCS) or second-source standard once a month or every 300 measurements, whichever is more frequent. The tolerance range is $\pm$ 0.25 units.

B. Run a duplicate every 10 samples (count frequence per method and matrix). The specification limit is $\pm$ 0.09 units. If exceeded, repeat the measurement once or twice more. If the specification is still not met, you must recalibrate and reanalyze the sample in question.

C. Read one of the buffers every 20 samples (all methods and matrices included to count frequency), or at the beginning and end of the

1. To 20 g of waste sample in a 50-mL beaker, add 20 mL of reagent water, cover and continuously stir the suspension for 5 minutes. Additional dilutions are allowed if working with hydroscopic wastes and salts or other problematic matrices.

2. Let the waste suspension stand for about 15 minutes to allow most of the suspended waste to settle out from the suspension or, if necessary, filter or centrifuge off aqueous phase for pH measurement.

NOTE: If the waste is hygroscopic and absorbs all the reagent water, begin the experiment again using 20 g of waste and 40 mL of reagent water.

NOTE: If the supernatant is multiphasic, decant the oily phase and measure the pH of the aqueous phase. The electrode may need to be cleaned (Step IV.C) if it becomes coated with an oily material.

3. Adjust the electrodes in the clamps of the electrode holder so that, upon lowering the electrodes into the beaker, the glass electrode will be immersed just deep enough into the clear supernatant solution to establish a good electrical contact through the ground-glass joint.

4. Solutions should be a room temperature when analyzed. The Triode electrode is capable of adjusting for temperature inconsistencies, however, you should still adjust the samples to room temperature before analyzing. (Record the temperature of **each** solution on the data sheet.)

5. Rinse the electrode with deionized water and blot it dry with a kimwipe. Place it into the solution as described in step 3.

6. The display will start to change. Once the reading stabilizes the meter will beep and the number will freeze. Record this number on the data sheet.

7. If you calibrated with 7 and 4 buffers, the sample pH must fall between 7 and 4. If the pH falls out of this range, you must go back and recalibrate using the 7 and 10 buffers and rerun the sample.

8. Be sure you rinse off the electrode with deionized water in between samples.

## IX. ELECTRODE STORAGE AND MAINTENANCE:

A. Storage

1. Store the electrode in a solution made up of 1 gram KCl or NaCl in 200 mL of pH 7 buffer. This solution may be purchased from Fisher.

6. Be sure you rinse off the electrode with deionized water in between samples.

B. Drinking Water and Acid-rain Samples by Method 150.1

Treat these samples exactly as you would other water samples except for the stirring. It is imperative that the stir plate is set on its' lowest setting and that the sample is not stirred vigorously. You should use the smallest stir bar possible. Make sure that you do not aerate the sample.

C. Soil Samples by Method 9045C

1. To 20 g of soil in a 50-mL beaker, add 20 mL of reagent water, cover, and continuously stir the suspension for 5 minutes. Additional dilutions are allowed if working with hygroscopic soils and salts of other problematic matrices.
2. Let the soil suspension stand for about 1 hour to allow most of the suspended clay to settle out from the suspension or, if necessary, filter or centrifuge off the aqueous phase for pH measurement.
3. Adjust the electrodes in the clamps of the electrode holder so that, upon lowering the electrodes into the beaker, the glass electrode will be immersed just deep enough into the clear supernatant solution to establish a good electrical contact through the ground-glass joint.
4. Solutions should be a room temperature when analyzed. The Triode electrode is capable of adjusting for temperature inconsistencies however, you should still adjust the samples to room temperature before analyzing. (Record the temperature of **each** solution on the data sheet.)
5. Rinse the electrode with deionized water and blot it dry with a kimwipe. Place it into the solution as described in step 3.
6. The display will start to change. Once the reading stabilizes the meter will beep and the number will freeze. Record this number on the data sheet.
7. If you calibrated with 7 and 4 buffers, the sample pH must fall between 7 and 4. If the pH falls out of this range, you must go back and recalibrate using the 7 and 10 buffers and rerun the sample.
8. Be sure you rinse off the electrode with deionized water in between samples.

B. Solid Waste Samples by Method 9045C

readings are within 0.05 pH units of the buffer solution value. The specific steps to follow match the explanations in Section A.

C. Calibration Check

1. Rinse the electrode and place it in the 7 buffer. Read this buffer as a sample and not as a calibration standard. Press "measure". The display will start to fluctuate as the pH is being measured. Once the reading stabilizes the meter will beep and the display will freeze.

2. Record the reading. (It should be 7.00± 0.09. If it is not within this range, inspect the electrode and meter and recalibrate with fresh buffers. If the reading is still out of range, check the manual for troubleshooting.)

3. If the 7 buffer reads correctly, read the 4 buffer and 10 buffer. Record these readings. (The reading should not deviate from the true values by more than 0.09 units. If they are not within this range, inspect the electrode and meter and recalibrate with fresh buffers. If the reading is still out of range, check the manual for troubleshooting.)

## VIII. PROCEDURE:

A. Water and Liquid Waste Samples (Non Drinking Water) by Methods 150.1 or 9040B

1. Sample(s) should be at room temperature when analyzed. The Triode electrode is capable of adjusting for temperature inconsistencies, however, you should still adjust the samples to room temperature before analyzing. (Record the temperature of **each** sample on the data sheet.)

2. Rinse the electrode with deionized water and blot it dry with a kimwipe. Place it into the sample with the stirring bar turning. Make sure that the electrode does not touch the sides or the bottom of the beaker.

3. The display will start to change. Once the reading stabilizes the meter will beep and the number will freeze. Record this number on the data sheet.

4. If you calibrated with 7 and 4 buffers, the sample pH must fall between 7 and 4. If the pH falls out of this range, you must go back and recalibrate using the 7 and 10 buffers and rerun the sample.

5. If you are determining Corrosivity by 9040B, use a pH 12 buffer to bracket the alkaline upper range.

# TOTAL ORGANIC CARBON (TOC)

## SM5310C, SW846 METHOD 9060 AND LLOYD KAHN METHOD

**SOP NUMBER:** **SOP-221**

**REVISION NUMBER:** **7**

**APPROVED BY:**

**SECTION MANAGER**

**TECHNICAL DIRECTOR**

**EFFECTIVE DATE:** **10/27/08**

**DATE OF LAST REVIEW:** **10/27/08**

measurement session; whichever is more frequent. If it does not read its true value ± 0.09 units, then you must recalibrate.

D. Record the QC data (LCSs and duplicates) on the QC sheet. Make sure that this information is written legibly and is complete, including specification of the method number.

E. Electrodes must be rinsed thoroughly between samples.

F. Document any problem experience during the measurements. Complete a corrective action where necessary. This must be done within 24 hours of occurrence of the incident.

G. Refer to SOP-431 for common environmental laboratory definitions.

## XI. REFERENCES

A. USEPA Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, March 1983, Method 150.1.
B. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), Methods 9040B and 9045C.
C. Standard Methods 18th Edition 1992 p. 4-65.

## XII. WASTE MANAGEMENT AND POLLUTION PREVENTION

A. Please see Waste Disposal SOP-405 for instruction of proper disposal of waste generated from this area.

B. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

# TOTAL ORGANIC CARBON (TOC) BY SM5310C, SW846 METHOD 9060 AND Lloyd KAHN

METHOD "*DETERMINATION OF TOC IN SEDIMENT*"

## I. SCOPE AND APPLICATION

This SOP describes the measurement of TOC by SM5310C, SW-846 Method 9060 and Lloyd Kahn Method for determination in soil /sediment matrix.

SM5310C is used to determine the concentration of organic carbon in source and drinking water, SW-846 Method 9060 is used to determine concentrations of carbon in saline waters, domestic and industrial wastes and SW846 Method 9060 is modified for soil determination and the L1oyd Kahn Method is used for determination of TOC in soil/sediment and solid matrices. SW846 Method 9060 and the Lloyd Kahn Method require quadruplicate analysis of samples, where as SM5310C requires a minimum of two analyses. These methods should be read over carefully by the analyst and any restrictions should be noted.

## II. SUMMARY OF METHOD

The organic carbon is measured using an Shimadzu Total Organic Carbon Analyzer (aqueous samples) and an OI Analytical Solids TOC Analyzer model 1010 (soil/sediment samples). The Shimadzu instrument converts the organic carbon in a sample using wet chemical oxidation. The $CO_2$ formed is then measured by an infrared detector (replaces ultraviolet detector in SM 5310C). With the model 1010 Solids TOC analyzer, TOC is determined by acidifying a sample and heating it to 250°C to remove the TIC. The sample is then heated to 900°C to combust the remaining TOC. The resulting carbon dioxide from the TOC is detected by a non-disperse infrared (NDIR) detector that has been calibrated to directly display the mass of carbon dioxide detected. This mass is proportional to the mass of TOC in the sample.

The limit of detection for the water method is 0.50 mg carbon/L and the Limit of quantitation is 1.0 mg carbon/L. The limits of detection and quantitation with the soil method depends on the how many grams of sample is used for the analysis. For a 250 mg sample the limit of detection is 460 mg/kg and the limit of quantitation is 1600 mg/kg.

## III. SAMPLING HANDLING AND PRESERVATION

3.1 Sampling and storage in glass bottles is preferable. Sampling and storage in plastic bottles such as conventional polyethylene and cubitainers is permissible if it is established that the containers do not contribute contaminating organics to the samples. NOTE 1: A brief study performed in the EPA Laboratory

indicated that distilled water stored in new, one quart cubitainers did not show any increase in organic carbon after two weeks exposure.

3.2 Because of the possibility of oxidation or bacterial decomposition of some components of aqueous samples, the lapse of time between collection of samples and start of analysis should be kept to a minimum. The holding time is 28 days for waters and soils with the exception of the Lloyd Kahn method soils, which requires a 14 day holding time. Also, samples must be kept cool (4°C) and protected from sunlight and atmospheric oxygen.

3.3 When water samples cannot be analyzed immediately, the sample is preserved by acidification to (pH </= 2) with HCl or $H_2SO_4$. Both water and soil samples are stored at 4°C.

## IV. INTERFERENCES

### 4.1 WATER METHOD

4.1.1 Removal or carbonate and bicarbonate carbon by acidification and purging with purified gas results in the loss of volatile organic substances. The volatiles also can be lost during sample blending, particularly if the temperature is allowed to rise. Another important loss can occur if large carbon-containing particles fail to enter the needle used for injection. Filtration although necessary to eliminate particulate organic matter when only DOC is to be determined, can result in loss or gain of DOC, depending on the physical properties of the carbon-containing compounds and the adsorption of carbonaceous material on the filter, or its desorption from it. Check filters for their contribution to DOC by analyzing a filtered blank. Note that any contact with organic material may contaminate a sample. Avoid contaminated glassware, plastic containers, and rubber tubing. Analyze treatment, system, and reagent blanks.

4.1.2 This procedure is applicable only to homogenous samples which can be injected into the apparatus reproducibly by means of a pipette. The openings of the pipette limit the maximum size of particles which may be included in the sample.

### 4.2 SOIL METHOD

4.2.1 All materials must be routinely demonstrated to be interference –free under the analysis conditions by running blanks. Use high purity or purified reagents and gases to help minimize interference problems.

4.2.2 The infrared detector is sensitized to $CO_2$ and accomplishes virtually complete rejection of response from other gases that absorb energy in the infrared region.

## V. DEFINITIONS

5.1 ANALYTICAL BATCH-The set of samples extracted /distilled/ or digested at the same time to a maximum of 20 samples.

5.2 CALIBRATION BLANK (CB)- A volume of reagent water in the same matrix as the calibration standards, but without the analyte.

5.3 CALIBRATION STANDARD (CAL)- A solution prepared from the primary dilution standard solution or stock standard solutions. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

5.4 FIELD BLANK (FMB)- An aliquot of reagent water or equivalent neutral reference material treated as a sample in all aspects, including exposure to a sample bottle holding time, preservatives, and all preanalysis treatments. The purpose is to determine if the field or sample transporting procedures and environments have contaminated the sample.

5.5 FIELD DUPLICATE (FD)- Two samples taken at the same time and place under identical circumstances which are treated identically throughout field and laboratory procedures. Analysis of field duplicates indicates the precision associated with sample collection, preservation, and storage, as well as with laboratory procedures.

5.6 LABORATORY BLANK (LRB)- An aliquot of reagent water or equivalent neutral reference material treated as a sample in all aspects, except that it is not taken to the sampling site. The purpose is to determine if the analytes or interferences are present in the laboratory environment, the reagents, or the apparatus.

5.7 LABORATORY CONTROL SAMPLE (LCS)- A solution prepared in the laboratory by dissolving a known amount of one or more pure compounds in a known amount of reagent water. Its purpose is to assure that the results produced by the laboratory remain within the acceptable limits for precision and accuracy. (This should not be confused with a calibrating standard, it must be prepared from a source other than the same source as the calibration standards).

5.8 LABORATORY DUPLICATE (LD)- Two aliquots of the same environmental sample treated identically throughout a laboratory analytical procedure. Analysis of laboratory duplicates indicates precision associated with laboratory procedures but not with sample collection, preservation, or storage procedures.

5.9 QUALITY CONTROL CHECK SAMPLE (QCS)- A sample containing analytes of interest at known concentrations (true value) of analytes. The QCS is obtained from a source external to the laboratory or is prepared from standards obtained from a different source than the calibration standards. The purpose is to check laboratory

performance using test materials that have been prepared independently from the normal preparation process.

5.10 METHOD DETECTION LIMIT (MDL)- The lowest level at which an analyte can be detected with 99 percent confidence that the analyte concentration is greater than zero.

## VI. REAGENTS/STANDARDS

Store all reagents and standards according to recommendations. All standards should be stored away from light and at 4°C (± 2°C).

6.1 The laboratory reagent blank water used for TOC analysis is obtained from the Modulab Analytical water purification system in the analytical laboratory. **Boiling the water is not necessary as the method states.**

6.2 Potassium hydrogen phthalate, primary stock solution, 1000 mg/L: Dissolve 0.2128g of potassium hydrogen phthalate (primary standard grade) in 100.0 mL water.

6.3. Potassium hydrogen phthalate, standard solutions : A 100 mg/L standard is prepared by transferring 10 mL of the stock solution to a 100 mL volumetric flask and diluting to the mark with water. This solution is prepared on a daily basis.

6.4. The carbonate-bicarbonate solutions are not needed for this instrument.

6.5 Calibration Standards

1. For the water method, calibration standard is Potassium Hydrogen Phthalate. Standards are made from dilutions of the stock 1000 mg/L standard as follows:

   1.0 mg/L = 0.10 mL of 1000 mg/L -> 100 mL
   2.5 mg/L = 0.25 mL of 1000 mg/L -> 100 mL
   5.0 mg/L = 0.50 mL of 1000 mg/L -> 100 mL
   10.0 mg/L = 1.0 mL of 1000 mg/L -> 100 mL
   25.0 mg/L = 5.0 mL of 1000 mg/L -> 200 mL
   50.0 mg/L = 10.0 mL of 1000 mg/L -> 200 mL
   100 mg/L = 10.0 mL of 1000 mg/L -> 100 mL

   A low level standard curve must be run for drinking water samples with the standards made as follows:

   0.25 mg/L = 0.025 mL of 1000 mg/L -> 100 mL

0.50 mg/L = 0.050 mL of 1000 mg/L -> 100 mL
1.0 mg/L = 0.10 mL of 1000 mg/L -> 100 mL
1.5 mg/L = 0.15 mL of 1000 mg/L -> 100 mL
2.5 mg/L = 0.25 mL of 1000 mg/L -> 100 mL
5.0 mg/L = 0.50 mL of 1000 mg/L -> 100 mL
10.0 mg/L = 1.0 mL of 1000 mg/L -> 100 mL

2. The soil method the calibration standard is prepared by using an OI commercially prepared 30% carbon sucrose solution.

6.6 Laboratory Control Sample:

1. For the water method, the Laboratory Control Sample is normally made from a performance evaluation solution of which the true value is known. This solution is given a unique identifier.

2. For the soil method, the Laboratory Control Sample is made from a 30% sucrose solution which is made by weighing up 7.125 grams of EM Reagent Grade Sucrose and diluting to 10 mL with deionized water volumetrically.

6.7. Persulfate oxidation solution: This solution is made by dissolving 60g of sodium persulfate in DI water, adding 15 ml of phosphoric acid and diluting to 500 ml.

6.8 Phosphoric acid solution: Dilute 100 mL of concentrated 85% phosphoric acid in 500 mL of water. This is used for water.

6.9 Phosphoric acid solution 5%: Dilute 59 mL of concentrated 85% phosphoric acid in 1000 mL of water. This is used for soil.

## VII. INSTRUMENTATION

7.1 The instrument used for the Water TOC analysis is a Shimadzu Total Carbon Analyzer. An OIC 1010 soil/sediment carbon analyzer is used for soil samples.

7.2 There is a Shimadzu autosampler which will hold 68 samples.

7.3 The corresponding data for each sample is obtained from the Shimadzu software for the water samples. The soil/sediment data are printed out at the organic GC printer.

## VIII. AQUEOUS SAMPLE PROCEDURE

8.1 Wearing labcoat, gloves and safety glasses, the standards and check solutions should be taken out of the refrigerator and allowed to warm to room temperature. Also, remove samples from sample storage signing them out appropriately on the internal chain of custody form. Fresh acid and oxidation solutions should be poured into the appropriate containers on the front of the instrument.

**8.2** Follow the instructions for operation of the instrument in Chapter 4, section 4.3 of the Shimadzu Model TOC-VWS User Manual. **See Appendix I. for Basic TOC start-up notes for analysis.**

8.3 **Following is a list outlining the order in which the samples should be run.** Each sample VOA vial should be numbered and its identity entered into the TOC schedule. Note: All blanks should be acidified to pH 2 to match the matrix of the samples analyzed.

1. 100 ppm
2. 50 ppm
3. 25 ppm
4. 10 ppm
5. 5.0 ppm
6. 2.5 ppm
7. 1.0 ppm
8. Method blank
9. LCS + 9 samples (including any sample QC
10. 25 ppm
11. 10 samples (including any sample QC)
12. 50 ppm

8.8 Insrument printouts are generated from the software. Normal procedure is followed for preparing reports and the data is second checked before being given to the supervisor.

## IX. SOIL/SEDIMENT SAMPLE PROCEDURE

A sample is introduced into the Solid Module via a conditioned sample cup. Once the sample has been introduced the entire analysis sequence is automatic. Please reference Chapter 4 of the OI 1010 Solid Module instrument manual for instrument states and configuration when initially setting the instrument methods up.

**TC Mode Instrument Settings:**

Analysis Temp:900°C
Analysis Time: 6.5 minutes
Nitrogen Gas Flow: 60-100 psi (external regulator regulator)
**Oxygen Gas Flow: 40-60 psi (external regulator)**

**This is a step by step description of a routine soil TOC analysis.**

9.1 The standards and check solutions should be taken out of the refrigerator and allowed to warm to room temperature. The nitrogen and oxygen (internal regulator should be set at 50-60 psi) turned on allowing a nitrogen flow of 350-400mL/minute and an oxygen flow of 180 mL/minute (± 3 mL/minute).

**NOTE: DO NOT TURN THE ANALYZER ON BEFORE TURNING THE GAS ON!**

9.2 Let the gas flow through the instrument for a few minutes. The instrument should now be turned on and let to stabilize for 30 minutes.

9.3 Condition the cups (with quartz wool in them) using Diagnostics under Instrument Menu commands, (don't condition too many cups at a time since setting in contact with the air can cause contamination).

9.4 Set up the subdirectory (using the current date to ID it) under WinTOC output.

9.5 If doing an initial calibration curve use an appropriate µL syringe to make the following measurements of the sucrose standard in order to achieve the indicated concentrations. Make sure that there are no air bubbles in the syringe. Turn the syringe with the needle pointed up and vibrate the barrel and disperse any air from the syringe. To enter the calibration information on the instrument go to Instrument Cal Menu, type in the calibration standard values and save the file as the cal.. date analyzed.

| µL 30% Sucrose STD | Concentration (mg) |
|---|---|
| 0 | 0 |
| 2.0 (1:6 solution) | 0.10 |
| 3.0 | 0.90 |
| 50 | 15 |
| 100 | 30 |

Note: The 1:6 solution of the 30% Sucrose standard is prepared by mixing 100µL of the 30% Sucrose standard with 500µL of water.

9.6 Enter the sequence to be analyzed as listed below:

1. CCV(CCV1+ date analyzed for ID) or Initial calibration – single analyses
2. Method Blank(MB + date analyzed for ID) – single analyses
3. LCS, 15 mg dextrose (LCS + date analyzed for ID) – single analyses
4. NY Cert – 4 replicates
5. Sample – 4 replicates
6. Sample – 4 replicates
7. Sample – 4 replicates
8. Sample – 4 replicates
9. Sample – 4 replicates
10. CCV(CCV1+ date analyzed for ID)2 – single analyses
11. Sample – 4 replicates
12. Sample – 4 replicates
13. Sample – 4 replicates
14. Sample – 4 replicates
15. Sample – 4 replicates
16. CCV (CCV2+ date analyzed for ID) – single analyses
17. Sample – 4 replicates
18. Sample – 4 replicates
19. Sample – 4 replicates
20. Sample – 4 replicates
21. Sample – 4 replicates
22. CCV(CCV3+ date analyzed for ID) – single analyses
23. Sample – 4 replicates
24. Sample – 4 replicates
25. Sample – 4 replicates
26. Sample – 4 replicates
27. Sample – 4 replicates
28. CCV(CCV4+ date analyzed for ID) – single analyses
29. SampleMS – 4 replicates
30. SampleDUP – 4 replicates
31. FCV(CCV4+ date analyzed for ID) – single analyses
32. FCB(FCB4+ date analyzed for ID) – single analyses

9.7 Samples should be stored away from light and at 4°C (± 2°C). Wearing labcoat, gloves and safety glasses remove samples from sample storage signing them out appropriately on the internal chain of custody form.

**9.8** Transfer a homogeneous aliquot(~5 g) of the sample into a small pre-labeled aluminum weighing pan. Label each pan with the appropriate sample ID then add enough phosphoric acid (1-2 ml) to remove the Total inorganic carbon (TIC) when the sample is placed in an oven at 250°C. Place the samples in the 250°C oven for 10 minute and begin prepping the sample cups to weigh 0.2g-1.0g of each sample(in quadruplicate). Limit the time that the cups are

exposed to the atmosphere as to reduce potential contamination. **Note: Since the samples are dried in this manner, before the sample aliquot it taken, a % solids determination and calculation is NOT necessary to report the sample concentrations in dry weight.**

9.9 Set the OI 1010 to the TC Mode and start running the sequence beginning with the initial calibration or calibration verification standard as illustrated above. Weigh each sample in quadruplicate making sure to limit the time that samples are exposed to the atmosphere.

9.10 The Excel file for calculations is located in "V:\WCM\TESTS\TOC soil\". The sample identity, its corresponding mgC reading, and the sample weight are entered into the appropriate columns. The Excel worksheet is self explanatory. Normal procedure is followed for preparing reports and the data is second checked before being given to the supervisor.

## X. QC REQUIREMENTS

10.1 Analyze a laboratory control sample (LCS) for each batch of samples **(maximum of 10 samples per day).** If the LCS does not fall within the control limits of 80 to 120%, corrective action must be taken to find and correct the problem.

10.2 Run a method blank (PB) for each batch of samples (maximum of 20 samples per day). The PB should be less than 1/2 the reporting limit.

10.3 One matrix spike and matrix spike duplicate must be run per set of 20 samples. For water analysis, a spike and spike duplicate are made by mixing 20 mLs of sample with 0.30 mLs of stock 1000 mg/L standard using an ependorf pipette. The true value is 15 mg/L. The percent recoveries on a MS and a MSD should be within 75 and 125%. Relative percent difference (RPD) on duplicates should be less than 20%. If not, a corrective action (CAR) must be approved by your supervisor.

10.4 Analyze an initial calibration verification (ICV) immediately after the calibration curve. Analyze a calibration check verification (CCV) standard every tenth sample and at the end or after every fifth sample when analyzing samples in quadruplicate. Analyze a CCV after every 5th sample when analyzing soil/sediment samples. The percent recoveries should be in the range of 90 to 110%. The CCV %RSD warning limits are $\leq$15% for aqueous samples and $\leq$20% for soil/sediment samples. If the CCV % RSD exceeds 15%(aqueous) or $\leq$30% (soil/sediment) and the correlation coefficient is less than 0.990 correct the problem and re-analyze the CCV.

10.5 When analyzing water samples, all water blanks before samples and standards must be below the detection limit, otherwise the samples must be rerun.

10.6 Analyze an initial calibration blank (ICB) following the ICV. Analyze a continuing calibration blank (CCB) following each CCV. The ICB and CCB should be less than ± the MDL.

10.7 Calculate all percent recoveries and relative percent differences on duplicates and show calculations on data.

10.8 Calculate all percent recoveries and relative percent differences on duplicates and show calculations on data.

Calculate spikes as follows where everything is in concentration.

$$\% \text{ Recovery} = \frac{\text{Spike} - \text{Sample}}{\text{True Value}} \times 100$$

Relative percent difference is calculated as follows, with everything in concentration:

$$\text{RPD} = \frac{\text{Higher Concentration} - \text{Lower Concentration}}{\text{Average of Concentrations}} \times 100$$

10.9 SM5310B requires that the analyst repeat injection until consecutive measurements are obtained that are reproducible to within ±10%. A minimum of two injections is required for water samples with three replicates preferred. SW-846 Method 9060 requires quadruplicate analysis of each sample. The Loyd Kahn soil method suggests 1 sample per 20 be run in quadruplicate. Some clients may request that all samples to be done in quadruplicate. Please check with your supervisor if you have any questions about the required numbered of sample replications.

10.10 **For aqueous samples check an acidified 20mg/L inorganic carbon standard quarterly, to assure that purge gas flow is adequate to remove inorganic carbon. The result should be below the reported quantitation limit.**

## XI. CORRECTIVE ACTIONS

### 11.1 INSTRUMENT RELATED

1. ICV not within ± 20% (Soil) or ± 10% (SM 5310C0
   a. If the problem is with the solution.

i. Re-prepare, obtain new stock if necessary.

b. If the problem is with the calibration.

i. Recalibrate through analysis of appropriate standards and recheck ICV.

2. CCV not within ± 30% (Soil) or ±15% (SM 5310C)

a. If the problem is with the solution.

i. Re-prepare, obtain new stock if necessary.

b. If the problem is with the calibration.

i. Recalibrate through analysis of appropriate standards and re-prepare /reanalyze the previous ten sample according the following guidelines.

a. If the CCV was biased high, any of the previous ten samples which were below the minimum detection limit do not require reanalysis.

b. If the CCV was biased low, the previous ten samples must be reanalyzed.

*** Incorrectly set gas flow is a common instrument related problem which requires corrective action. Verify that all gas flows are adjusted properly.**

### 11.2 SAMPLE MATRIX RELATED

1. Replicate analysis RPD not within ±20% aqueous or ±50% soil/sediment

i. The associated sample data must be qualified on the final report.

2. Spike analysis recovery not within ±25% aqueous or ±50% soil/sediment

i. If the analyte level in the sample is greater than 4X the spiking level, the %recovery can not be evaluated and no action is taken.

ii. If the analyte level in the sample is not greater than 4X the spiking level, the associated sample data must be qualified on the final report. A corrective action report must accompany the data and be emailed or given to the supervisor.

## XII. HEALTH AND SAFETY

A. Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of blue nitrile gloves and lab coats is highly recommended.

B. Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples which need special consideration have applicable notes on the sample logs.

C. MSDS are available for all reagents and standards, which have been purchased. These are located in the administrative section next to the break room.

D. Please see *Waste Disposal; SOP-405* for proper disposal of the waste generated from this area.

## XIII. WASTE DISPOSAL and POLLUTION PREVENTION

Please see Waste Disposal SOP-405, for instruction of proper disposal of waste generated from this area. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

## XIV. METHOD PERFORMANCE

**14.1 Precision and Bias for Total Organic Carbon (TOC) by Persulfate-Ultraviolet Oxidation. (Water samples)**

| Characteristic Of Analysis Concentration determined, mg/L: | Spring Water | Spring Water +0.15 mg/L KHP* | Tap Water | Tap Water +10 mg/L KHP* | Municipal Wastewater Effluent |
|---|---|---|---|---|---|
| Replicate 1 | 0.402 | 0.559 | 2.47 | 11.70 | 5.88 |
| Replicate 2 | 0.336 | 0.491 | 2.49 | 11.53 | 5.31 |
| Replicate 3 | 0.340 | 0.505 | 2.47 | 11.70 | 5.21 |
| Replicate 4 | 0.341 | 0.523 | 2.47 | 11.64 | 5.17 |
| Replicate 5 | 0.355 | 0.542 | 2.46 | 11.55 | 5.10 |
| Replicate 6 | 0.366 | 0.546 | 2.46 | 11.68 | 5.33 |
| Replicate 7 | 0.361 | 0.548 | 2.42 | 11.55 | 5.35 |
| Mean, mg/L | 0.35 | 0.53 | 2.46 | 11.53 | 5.32 |
| Std. Deviation: mg/L | 0.02 | 0.03 | 0.02 | 0.21 | 0.23 |
| % | 6 | 6 | 1 | 2 | 4 |
| Actual Value, mg/L | - | 0.50 | - | 12.46 | - |
| Recovery, % | - | 106 | - | 93 | - |
| Error, % | - | 6 | - | 7 | - |

***KHP = potassium acid phthalate.**

**14.2 There was no method performance data available for the soil procedure.**

## XV. REFERENCES

1. Annual Book of ASTM Standards, Part 31, "Water," Standard D 2574-79, p. 469 (1976).

2. Standard Methods for the Examination of Water and Wastewater, 19th ED., Method 5310C (1999).

3. EPA SW-846, Method 9060.

4. Lloyd Kahn Method, *"Determination of Total Organic Carbon in Sediment"*

**APPENDIX I.**

1. Power up the lamp for warm –up, check reagents inside instrument cavity to make sure all are filled before starting the run.
2. Fill Fresh DI water in 1 gallon jug; DI squirt bottle and 1 L plastic
3. Label and load VOA vials with standards and samples into round tray.
4. Place round tray onto autosampler, get a final sample count for end point and replace lid.
5. Make sure that round tray fits down flush onto the autosampler.
6. On computer screen, select "TOC-Control V" icon.
7. Then select "Sample Table Editor"
8. Enter user name: "analyst initials" select OK.
9. Under "File" select "calibration curve" "OK".
10. Under system select Shimadzu TOC-BWS Enter/next
11. Select Edit Calibration points manually Enter/next
12. Under "Analysis" select "NPOC" then make up your file name (use today's date) Enter/next.
13. Calibration Measurement Parameters are default; Just hit "next"
14. Select "ADD" and enter calibration points starting at (1) 100 mg/L (2) 50 mg/L (3) 25 mg/L (4) 10 mg/L (5) 5.0 mg/L (6) 2.5 mg/L (7) 1.0 mg/L (8) 0.0 mg/L. After 8 points it should show 0.00 mg/L first and 100 mg/L eigth if so "next"
15. Put a check mark in "Correlation Coefficient" check box "next"
16. "next"
17. "finish"
18. Go to file and select "new", "sample run" "ok" "ok" enter file name: user date "save"
19. Now go to insert and select "calibration curve" then scroll till you find your file name/date should have .cal after date "select" the "open"
20. You should now see the sparging /acid addition page which shows a picture of the round sample tray. Under vial manually enter "1"beside 0.00 mg/L.
21. manually enter "2" beside 1.0 mg/L and "3" beside 2.5 mg/L and so on and so forth all the way to "8" this shows what order they ar loaded on the tray. "Enter/OK"
22. Then a screen with your filename/date and all info should be in row 1 only with vial column showing. 1,2,3,4, etc.
23. Select the lightening bolt symbol then enter "use PC settings" this will start initializing wait till screen goes away then you will see the stop light symbol appear with green light showing, select that icon select "keep running" select "standby"
24. Sparging/acid addition page will re-appear just hit "OK"
25. Start ASI tray screen will appear hit "Start"
26. The instrument should start establishing the baseline and move auto tray into position – Lid must be on and samples loaded into corret position will take almost

3 hours to finish. Can view data as its coming off by selecting "view" "sample window". After calibration is done review.

27. Select "File" then "New" then "sample run" "ok"
28. General information screen: No change select "ok"
29. Save as screen: Select today's date for file name example 00month/00day/00year
30. Select "save"
31. Sample Table Screen: Select "insert" then select " auto generate" enter
32. **Page 1** sample group wizard sample source: select "calibration curve" then double click on box with 3 dots ...
33. Open latest curve from calibration curves file
34. Highlight latest curve and select "open"
35. Should send you back to page 1 with calibration curve info submitted. Select "next"
36. **Page 2** Sample Parameter: Enter final sample count for "number of samples" select "next"
37. **Page 3** Calibration Curves: No changes Select "Next"
38. **Page 4** Calibration Checks: No changes Select "Next"
39. **Page 5** Controls: No changes select "finish", Select "ok" on "Sparging/ Acid page.
40. Type sequence as they are loaded on tray: ICV, ICB, LCSW, Sample #, client,etc.
41. Once everything is typed in double check that it matches the way samples and QC are loaded..
42. Click or select the lightening bolt symbol then select "use settings on PC". Wait for initializing. When screen goes awy the traffic light symbol should appear next to the lightning bolt symbol. Click on the traffic light symbol.
43. Click or select "shut down Instructions". Then select "standby" Sparging/ Acid addition screen will appear so you can confirm your tray is loaded the wax things are highlighted in blue. Select "OK" if it looks the same.
44. Start ASI measurement: External acid addition should have a check mark click on "start" analysis should begin to start.
45. Click on view and chose "sample window" to watch curves come off and to see beginning values.

# GC/MS LOW LEVEL PAH'S

# BY SW846 METHOD 8270C SIM

**SOP NUMBER:** **SOP-231**

**REVISION NUMBER:** **3**

**APPROVED BY:**

**SECTION MANAGER**

**QUALITY ASSURANCE OFFICER**

**EFFECTIVE DATE:** **01/16/09**

**DATE OF LAST REVIEW:** **01/16/09**

# GC/MS LOW LEVEL PAH'S BY SW846 METHOD 8270C

## 1.0 SCOPE AND APPLICATION

This SOP (based primarily on SW-846 Method 8000B/8270C using SIM) is used for the analysis of semi-volatile organic compounds in a variety of matrices (soils, sediments, waters, etc.). The normal laboratory list of PAH analytes with their water and soil LCS limits are found attached in the appendix. Any questions left by this SOP should be answered by reading the method SW-846 8000B/8270C. If questions still remain unanswered, check with the Organic Lab Manager, QA/QC Officer and/or Technical Director. A documentation of capability (DOC) should be performed by each analyst using this method.

## 2.0 METHOD SUMMARY

After sample preparation using the appropriate extraction technique, the sample is introduced into the GC/MS using direct injection. The analytes are separated in the gas chromatograph by a combination of the temperature program, the pressure program and the capillary column. The analytes are then detected by the mass spectrometer. Analytes are identified by comparing the mass spectra of known standards with the mass spectra from the sample. Analytes are quantitated relative to known standards using the internal standard method.

## 3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Section 3.0 and tables 3-1 and 3-2 of the Empirical Laboratories, LLC Quality Assurance Manual include details concerning sample preservation, containers and handling of semi-volatile samples and extracts. All water and soil samples are stored in their respective walk-in coolers at a temperature of 4°C. All extracts are stored in the Hobart in the Extraction lab at a temperature of 4°C. Water samples have a holding time of 7 days from date of sampling. Soil samples have a holding time of 14 days from date of sampling with 40 days from extraction to analysis for waters and soils. (unless otherwise specified for the project).

## 4.0 INTERFERENCES AND POTENTIAL PROBLEMS

4.1 All raw data (samples & QC) must be evaluated for interferences. If contamination occurs, determine whether the source of interference is in the preparation or clean-up of the samples and take corrective action to eliminate the problem.

4.2 Contamination by carryover can occur when samples of high-concentration and low-concentration are analyzed sequentially. To reduce carryover, the sample syringe must be rinsed with solvent between injections. If an unusually high sample is detected, a solvent blank should be analyzed for cross contamination.

## 5.0. EQUIPMENT AND APPARATUS

5.1 HP 7890 GC complete with electronic pressure control and temperature programmable gas chromatograph suitable for splitless injection.

5.2 Column: RTX-5MS (or equivalent) 30 m x 0.25 mm I.D. x 0.25 µm film thickness fused silica capillary column or RTX-5 SIL-MS 30 m x 0.28 mm I.D. x 0.5µm film thickness.

5.3 HP 5975 mass spectrometer capable of scanning from 35 to 500 amu every second or less and capable of using Selected Ion Monitoring (SIM), using 70 volts electron energy in electron impact ionization mode. The mass spectrometer is capable of producing a mass spectrum for decafluorotriphenylphosphine, DFTPP, which meets all the tuning criteria of the EPA methods.

5.4 HP 7683B autosampler capable of reproducibility from one injection to another proven by meeting QC and calibration criteria.

5.5 HP GC/MS interface that gives acceptable calibration points at 10 ng per injection for each compound of interest and achieves acceptable tuning performance criteria.

5.6 Acquisition Software: HP Chemstation system is interfaced to the GC/MS. The system acquires and stores data throughout the chromatographic programs.

5.7 Data Processing Software: Target DB on Windows NT server data system is interfaced to the HP Chemstation. The system accepts and stores acquired data. It plots by extracted ion current profile (EICP). The system is also capable of integrating the abundances in any EICP between specified time or scan-number limits. The most recent NBS mass spectral library is installed.

## 6.0 REAGENTS

6.1 Methylene chloride **(Please read SOP-336 before handling this solvent in our laboratory.)** – Trace analysis grade.

6.2 Stock standards are purchased in mixtures from reputable vendors. The date they are received is noted on the label and recorded in the BNA standards log book. The date they are opened is noted on the label and recorded in the BNA standards log book along with their lot number and vendor. Each standard that is prepared is recorded in the BNA standards log book and given a sequential number. Each standards label is completed with the standard number, name, preparation date, expiration date, solvent and analyst initials. All stocks and standards are stored in the freezer at a temperature of -15°C ± 5°C from the date they are received/prepared. Standards are brought to room temperature before being used to make standards. Sonication is used if precipitation is observed after bringing to room temperature. The refrigerator and freezer temperature is monitored daily with an annually calibrated thermometer and recorded with calibration correction in the Extraction temperature/calibration logbook. Makeup of common standards is detailed below. See the BNA standards log book for makeup of other standards.

6.2.1 The Decafluorotriphenylphosphine (DFTPP) tuning standard is prepared as follows (includes benzidine, pentachlorophenol and 4,4'-DDT): Using a 10µL syringe, 10µL (GCM-150, Ultra Scientific @ 1000µg/mL, or equivalent) is injected into a 1.0mL volumetric flask containing approximately 0.900mL methylene chloride (Trace Grade) and diluted to volume with same making a

10µg/mL standard. It is stored in the freezer at -15°C ± 5°C for up to 6 months. A direct injection of 1.0µL is used to tune the instrument.

6.2.2 Calibration standards are prepared from a 50µg/mL intermediate standard at a minimum of five concentrations. Calibration standards are prepared semi-annually unless the initial calibration verification standard indicates a problem. To makeup the 50µg/mL intermediate standard inject the indicated amount of the following standards (or equivalent) into a 10mL volumetric containing approximately 1mL methylene chloride (Trace Grade) and dilute to volume with the same. After capping and inverting several times, the solution is transferred into a labeled 10ml, teflon-lined, screw-capped vial and stored in the freezer at -15°C ± 5°C for up to 12 months. See the Appendix for analytes contained in these mixes.

| Standard | Conc (µg/mL) | Amount(µL) | Final Conc(µL) |
|---|---|---|---|
| PAH Mix (Vendor,Lot#) | 2000 | 250 | 50 |
| BN Surrogate (Vendor,Lot#) | 5000 | 100 | 50 |

To makeup the calibration standards, inject the indicated amount of a 5.0µg/mL or 0.5 ug/ml intermediate standard, made from a 10x cut of the 50 and 5.0 ug/ml, or the 50 ug/ml into a 2mL vial containing methylene chloride (Trace Grade ) and diluted to 1000ul with the same. After capping and inverting several times and adding 5ul internal standard, the standards are transferred into labeled 2ml, teflon-lined, screw-capped vials and stored in the freezer at -15°C ± 5°C for up to 6 months.

| Standard | Amount of standard | Amount of 100µg/mL Internal Standard |
|---|---|---|
| 0.005 µg/mL | 10 µL of 0.5 µg/mL | 10 µL |
| 0.01 µg/mL | 20 µL of 0.5 µg/mL | 10 µL |
| 0.05 µg/mL | 10 µL of 5 µg/mL | 10 µL |
| 0.1 µg/mL | 20 µL of 5 µg/mL | 10 µL |
| 0.25 µg/mL | 50 µL of 5 µg/mL | 10 µL |
| 0.5 µg/mL | 100 µL of 5 µg/mL | 10 µL |
| 1.0 µg/mL | 20 µL of 50 µg/mL | 10 µL |
| 5.0 µg/mL | 100 µL of 50 µg/mL | 10 µL |
| 10.0 µg/mL | 200 µL of 50 µg/mL | 10 µL |
| 20.0 µg/mL | 400 µL of 50 µg/mL | 10 µL |
| 30.0 µg/mL | 600 µL of 50 µg/mL | 10 µL |
| 50.0 µg/mL | 25 µL of 2000 µg/mL | 10 µL |

The makeup of the 5.0µg/mL CCV standard is detailed below. Note: MS list spikes and full list spikes for LCS and/or MS/MSD are prepared from an alternate source or lot number than the calibration standards.

5.0µg/mL CCV standard: Using a 100µL syringe, 100µL of a 50µg/mL working standard is injected into a 2 ml vial containing 900µL methylene chloride (Trace Grade) to make a 5.0µg/mL standard. After capping and inverting several times, 10µL of internal standard (100µg/mL) is then added and the standard is stored in the freezer at -15°C ± 5°C for up to 1 week.

6.2.3 The Initial Calibration Verification (ICV) standard is prepared from a vendor stock standard at a concentration of 50µg/mL as detailed below.

5.0 µg/mL ICV standard: Using a 50µL syringe, 50µL (C-507, NSI Solutions @ 100µg/mL, or equivalent) is injected into a 2mL vial containing 950µL methylene chloride (Trace Grade) making a 5.0µg/mL standard. After capping and inverting several times, 10µL of internal standard (100µg/mL) is added and the standard is stored in the freezer at -15°C ± 5°C for up to 6 months.

***For some clients an SPCC curve and CCV are analyzed along with the PAH standards to check for SPCC min. responses. See standard log for preparation of these standards.**

## 7.0 PROCEDURE

Prior to using SW-846 8270C (semivolatile method) the samples are prepared for chromatography using the appropriate sample preparation and clean up methods (generally SW-846 methods 3510, 3520, 3541, or 3550).

7.1 Chromatographic conditions:

7.1.1 GC

| | |
|---|---|
| Capillary Column | RTX-5MS 0.25 mm ID |
| Inlet mode | Pulsed Splitless |
| Pressure | 15.17 psi (on) |
| Pulse Pressure | 20 psi |
| Pulse time | 0.2 minutes |
| Purge flow | 60 mL/min. |
| Purge time | 0.15 minutes |
| Injector temperature | 300°C |
| Transfer line temperature | 280°C |
| Initial column temperature | 50°C for 0.5 minutes |
| Initial column temperature ramp | 20°C/min |
| Intermed. column temperature | 290°C for 0 minutes |
| Intermed. Temperature ramp | 5°C/min |
| Final column temperature | 320°C for 4.9 minutes |

7.1.2 MS

| | |
|---|---|
| Electron energy | 70 volts (nominal) |
| Mass range | 35 to 500 amu or SIM |
| A/D Samples | 3 |
| Solvent Delay | 4 minutes or as needed |

7.2 Tuning - Prior to any calibration or analysis, DFTPP tuning criteria must be met for a 1.0µL injection of the tuning standard [see below]. Tune must be met every 12 hours sample analysis is to be performed.

| Mass | Ion Abundance Criteria |
|---|---|
| 51 | 30-60% of mass 198 |
| 68 | <2% of mass 69 |
| 70 | <2% of mass 69 |
| 127 | 40-60% of mass 198 |
| 197 | <1% of mass 198 |
| 198 | Base peak, 100% relative abundance |
| 199 | 5-9% of mass 198 |
| 275 | 10-30% of mass 198 |
| 365 | >1% of mass 198 |
| 441 | Present, but less than mass 443 |
| 442 | >40% of mass 198 |
| 443 | 17-23% of mass 442 |

7.3 Calibration

7.3.1 Initial Calibration - An initial calibration curve at no less than five concentration levels must be analyzed and shown to meet the initial calibration criteria before any sample analysis may be performed. Method 8270C requires that the %RSD be less than 15% to use the average response factor for quantitation, the curve is to be used otherwise as long as the correlation factor is ≥0.99. The lowest standard must be less than or equal to the reported quantitation limit and the highest standard must not exceed the linear range of the detector. Generally, levels for the curve are 0.005 µg/mL, 0.01 µg/mL, 0.05 µg/mL, 0.1 µg/mL, 0.25 µg/mL, 0.5 µg/mL, 1.0 µg/mL, 5.0 µg/mL, 10µg/mL, 20µg/mL, 30µg/mL, and 50µg/mL. Any manual integrations are documented by inclusion of the integrated signals(**before and after manual integration** )initialed, reason and dated with the quantitation report and chromatogram. All integrations are checked for acceptability. Response factors of less than 0.050 must be supported by the mass spectrum of the lowest standard.

7.3.2 Initial Calibration Verification (ICV) - A second source standard at the 5.0 µg/mL level is used to check the validity of the curve. The standard recovery for all analytes must be between 75 and 125%. If the second source recovery is above 125%, it is possible that the main standard has deteriorated for that compound. That standard should be remade and reevaluated. If that does not correct the problem, the standard should probably be replaced and a new curve generated. If the second source recovery is below 75%, the second source standard may have deteriorated for that compound. The standard should be remade and reanalyzed. If this does not correct the problem, the standard should probably be replaced. If any compound in the ICV exceeds the criteria above, it may be evaluated and initialed by the organic section

manager. If deemed acceptable, the analyst may continue analysis. Any manual integrations are documented by inclusion of the integrated signals with the quantitation report and chromatogram. All integrations are checked for acceptability.

7.3.2 Continuing Calibration Verification (CCV)- Every 12 hours a CCV at 5.0 µg/mL must be analyzed and calculated against the initial calibration curve, then shown to meet the calibration criteria before any sample analysis may be performed. Acceptance criteria for 8270C analytes must be less than or equal to 20% difference or drift (%D, calculations follow in section 7.9). Internal standard areas should be within 50 to 200 percent of the area of the curve midpoint or the previous CCV. Retention times for the internal standards should be within 30 seconds of the retention time of the curve midpoint or the previous CCV. Any manual integrations are documented by inclusion of the integrated signals with the quantitation report and chromatogram. All integrations are checked for acceptability. Samples are then quantitated against the initial calibration curve.

7.4 LCS - The LCS is extracted 1 per extraction batch of up to 20 samples. The LCS is spiked using an alternate source or lot number than the calibration standards. See the LCS report forms in the appendix for the laboratory generated limits. Recoveries for the MS/MSD spike analytes in LCSs are charted annually to generate control limits for samples analyzed by method 8270C. In all cases, the lowest upper limit would be 110% and the lowest lower limit would be 10%. If enough data points are not present to generate limits, the limits default to 10-110% for all other analytes. See Section 8.3 below for corrective action.

7.5 Method Blank - Method blanks are extracted at a minimum of 1 per extraction batch - up to 20 samples. See Section 8.4 below for criteria and corrective action.

7.6 Samples - Prior to analysis, 0.5 mL samples are prepared by verifying volume and spiking with 5uL of the internal standard solution.

7.7 Data Reduction/Evaluation - Each sample analysis sequence is documented using the computer run log generated on the chemstation. This run log is signed, dated and paginated then placed in a 3 ring binder for that instrument. After the sample has been analyzed, the data is processed through Target DB on the Windows NT data system. The following must be checked to determine if the sample will need any reanalysis or dilution. Formal data evaluation is detailed in SOP-200 and documented using the Analyst Data Review Checklist (see Appendix). **See SOP-224 for guidance on manual integrations.**

7.7.1 Internal Standards - Areas should be within 50 to 200 percent of the area of the curve midpoint. Retention time should be within 30 seconds of the retention time of the curve midpoint. If not, the sample and historical data should be evaluated to determine the cause of the problem. If matrix effect is confirmed by reextraction/reanalysis or historical data, complete a corrective action

report and flag the affected compounds on the final report for matrix effect. Note: criteria applies to the continuing calibration, not samples, but is used as an indication of the sample analysis validity.

7.7.2 Surrogates – Control limits are determined annually by charting LCSs and method blanks. In all cases, the lowest upper limit would be 110% and the lowest lower limit would be 10%. All of the three surrogates for each fraction must be within the control limits in order for the extraction batch to be in control. If a surrogate exceeds the limits, the reason for the malfunction must be determined and a corrective action report must be completed. The sample must be reanalyzed, reextracted or flagged for QC problems.

| Surrogate | Water | Soil/Sediment |
|---|---|---|
| 2-Fluorobiphenyl | 34-167 | 14-129 |
| Terphenyl-d14 | 34-167 | 14-129 |

7.7.3 Analyte concentration must be within the range of the calibration curve after rounding to 2 significant figures. If an analyte exceeds the curve, a dilution must be performed and the next sample must be checked for carryover. Any dilution should keep the concentration of the analyte in question within the top half of the curve.

7.7.4 The qualitative identification of compounds is based on retention time and a comparison of the sample mass spectrum, after background subtraction, with characteristic ions in a reference mass spectrum from the NBS database (NBS75K.I). This database is used as it contains relatively uncontaminated mass spectra of each target compound which cannot be obtained from the daily calibrations during each 12 hour analytical period due to overlapping peaks in the mixes. Characteristic ions from the reference mass spectrum library are defined as the three ions of greatest relative intensity, or any ions over 30% relative intensity, if less than three such ions occur in the reference spectrum. In addition, the following criteria must be met. The RRT of the sample analyte must be within 0.06 RRT units of the RRT of the standard analyte. The relative intensities of the characteristic ions must agree within 30% of the relative intensities of the same ions in the reference spectrum. Structural isomers that produce very similar mass spectra should be identified as individual isomers so long as their GC retention times differ substantially. A library search may be made for analytes not associated with the calibration for the purpose of tentative identification. NOTE: The GC/MS analyst uses intelligence guided by experience to make the identifications. In uncontaminated spectra where ions are missing due to low concentration, if the major ions are present in the correct ratios at the correct retention time, the identification will be considered positive. In contaminated spectra, special emphasis will be placed upon higher mass ions, and the major ions will usually need to be present as major components of the spectrum (either unsubtracted or subtracted) for the identification to be positive. All assessments of relative intensities of ions will be made by visual estimates from the spectra.

7.8 Quantitation - Once a compound has been identified qualitatively, the concentration must then be quantitated. If the RSD of the compound's response

factor is 15% or less, then the concentration may be determined using the average response factor ($\overline{RF}$) from the initial calibration data. Otherwise, the concentration must be determined from equations based on internal standard calibration using either linear or non-linear calibration. Calculations follow in Section 7.9.

7.9 Calculations:

7.9.1 The RF is calculated as follows:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

where:

$A_s$ = Peak area (or height) of the analyte or surrogate.

$A_{is}$ = Peak area (or height) of the internal standard.

$C_s$ = Concentration of the analyte or surrogate.

$C_{is}$ = Concentration of the internal standard.

7.9.2 Calibration verification involves the calculation of the percent drift (linear or quadratic) or the percent difference (average) of the instrument response between the initial calibration and each subsequent analysis of the verification standard. Use the equations below to calculate % Drift or % Difference, depending on the calibration procedure used.

$$\%\ Drift = \frac{(\text{Calculated concentration} - \text{Theoretical concentration}) * 100}{\text{Theoretical Concentration}}$$

where the calculated concentration is determined from the initial calibration and the theoretical concentration is the concentration at which the standard was prepared.

$$\%\ Difference = \frac{(\text{CCV RF} - \text{Average RF}) * 100}{\text{Average RF}}$$

where CCV RF is the response factor from the analysis of the verification standard and Average RF is the average response factor from the initial calibration. The % difference or % drift calculated for the calibration verification standard must be within ±20% for each analyte before any sample analyses may take place.

7.9.3 Concentration in water samples is calculated as follows: [Note: Using the units specified here for these terms will result in a concentration in units of ng/mL, which is equivalent to µg/L.]

$$\text{Concentration } (\mu g/L) = \frac{(A_s)(C_{is})(D)(V_i)}{(A_{is})(\overline{RF})(V_s)(1000)}$$

where:

$A_s$ = Area (or height) of the peak for the analyte in the sample.

$A_{is}$ = Area (or height) of the peak for the internal standard.
$C_{is}$ = Concentration of the internal standard in the volume extracted in ug/L.
D = Dilution factor, if the sample was diluted prior to analysis. If no dilution was made, D = 1. The dilution factor is always dimensionless.
$V_i$ = Volume of the extract injected (µL). The nominal injection volume for samples and calibration standards must be the same.

$\overline{RF}$ = Mean response factor from the initial calibration.
$V_s$ = Volume of the aqueous sample extracted (mL). If units of liters are used for this term, multiply the results by 1000.

The 1000 in the denominator represents the number of µL in 1 mL. If the injection ($V_i$) is expressed in mL, then the 1000 may be omitted.

7.9.4 Concentration in non-aqueous samples is calculated as follows: [Note: Using the units specified here for these terms will result in a concentration in units of ng/g, which is equivalent to µg/kg.]

7.9.5

$$\text{Concentration } (\mu g/kg) = \frac{(A_s)(C_{is})(D)(V_i)}{(A_{is})(\overline{RF})(W_s)(1000)}$$

where: $A_s$, $A_{is}$, $C_{is}$, D, and $\overline{RF}$ are the same as for aqueous samples, and
$W_s$ = Weight of sample extracted (g). Either a dry weight or wet weight may be used, depending upon the specific application of the data. If units of kilograms are used for this term, multiply the results by 1000.

The 1000 in the denominator represents the number of µL in 1 mL. If the injection ($V_i$) is expressed in mL, then the 1000 may be omitted.

## 8.0 QUALITY ASSURANCE/QUALITY CONTROL/CORRECTIVE ACTIONS

8.1 Internals - All samples and QC are spiked with internal standards. See section 7.7.1 above for criteria and corrective action.

8.2 Surrogates - All samples and QC are spiked with surrogates. The surrogate recoveries from method blanks and LCS are used to generate control control limits for the surrogates. See section 7.7.2 above for criteria and corrective action. If any surrogate recoveries are below 10%, samples must be re-extracted if sample is available.

8.3 LCS Sample - The LCS is extracted 1 per extraction batch of up to 20 samples. The LCS is spiked using an alternate source or lot number than the calibration standards. If the LCS compound has a recovery above the upper limit, but the same compound is not detected in any of the batch samples, no corrective action is required. For all

other situations, the LCS should be reanalyzed for the failed analytes only. If the second analysis fails, all associated samples should be reextracted/reanalyzed for the failed analytes only or the data must be evaluated for flagging due to QC problems.

8.4 Method Blanks - The concentration of all method target analytes should be below the MDL for each method target analyte(**< ½ RL for DOD QSM**). The first step of corrective action is to assess the effect on the samples. If an analyte is found only in the method blank, but not in any batch samples, no further corrective action may be necessary. Steps should be taken to find/reduce/eliminate the source of this contamination in the method blank. If an analyte is found in the method blank and some, or all, of the other batch samples, then corrective action is required. The source of contamination must be investigated and appropriate action taken and documented to find/reduce/eliminate the source of this contamination. The method blank, and any samples containing the same contaminant, may need to be reextracted/reanalyzed. For the common laboratory contaminants, meeting the above requirements is not practical. Random cases of contamination are difficult to control, however, daily contamination is not acceptable and corrective action is essential. If a contaminant is found in the method blank and the samples, the compound concentration must be flagged with a 'B' on the final report unless the concentration is greater than 10x that found in the method blank.

8.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Sample - 1 in 20 samples are spiked for a MS/MSD (for full list spikes, the full list spiking solution is used). Control limits for the MS/MSD recoveries are the same as those for the LCS found in the appendix. RPD limits are found on the LCS report forms in the appendix. Samples which do not meet these criteria due to matrix must be evaluated for flagging on the final report due to QC problems. Generally, batch control is not based on MS/MSD results unless general method failure is determined to be the problem. In that case, the samples and associated QC would be reanalyzed for the failed analytes only. MS data evaluation must include the consideration of the following factors.

8.5.1 Sample matrix - If the sample is a soil, grab sample or sequentially collected water sample it may affect the %R and RPD of the MS/MSD. Corrective action must be taken in the form of reanalysis if a method problem is indicated.

8.5.2 Original sample concentration - If a spiked compound has a problem and the concentration of that compound in the original sample was two or more times the concentration of the spike, no further corrective action may be necessary other than the generation of a corrective action report to document the problem.

8.5.3 MS vs. MSD - If a spiked compound has a similar problem in both the MS and MSD and is not traced to a method problem, no further action may be necessary other than the generation of a corrective action report to document the problem.

8.5.4 Non-target Interference - The presence of significant non-target interference should be brought to the immediate attention of your supervisor who should discuss the problem with the client/project manager to determine the action to be taken.

8.5.5 Demonstration of Capability (DOC) – Each new analyst must complete a demonstration of capability by analyzing four LCSs with acceptable precision and accuracy. This also must be done when a new instrument is installed or a significant change to the method has been made.

## 9.0 HEALTH AND SAFETY

9.1 Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of gloves and lab coats is highly recommended.

9.2 Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples which need special consideration have applicable notes on the sample logs.

9.3 MSDS sheets are available for all reagents and standards which have been purchased. These are located on the bookshelf outside the office supply storage room.

## REFERENCES

1. *Test Methods for Evaluating Solid Waste*, SW-846,Method 8270C, Third Edition.
2. *DOD Quality Systems Manual for Environmental Laboratories*, 6/2002.
3. *AFCEE QAPP, Version 4.0, July 2004.*

## 10.0 EXAMPLE FORMS

Definitions and examples of the LCS report sheets (625 water limits, in-house water limits and in-house soil limits), the analyst data review checklist and figure 13 from *Federal Register* method 625 for the tailing factor calculation are located in the appendix.

# APPENDIX

## TABLE OF DEFINITIONS

1. amu- atomic mass unit
2. PAH-Polyaromatic hydrocarbons
3. BNA-base neutral acid
4. °C- degrees Centigrade
5. CCC- Calibration Check Compound
6. CCV- Continuing Calibration Verification
7. CLP- Contract Laboratory Program
8. %D- percent difference
9. DFTPP- Difluorotriphenyphosphine
10. EICP- extracted ion current profile
11. EPA- Environmental Protection Agency
12. g- gram or grams
13. GC- Gas Chromatograph
14. GC/MS- Gas Chromatograph/Mass Spectrometer
15. ICV- Initial Calibration Verification
16. I.D.- inner diameter
17. ISTD- internal standard
18. LSC- Laboratory Sample Concentrator
19. MDL- method detection limit
20. MS- Matrix Spike
21. MSD- Matrix Spike Duplicate
22. M.S.- Mass Spectrometer
23. µm- micometer
24. µL- microliter
25. mL- milliliter
26. mm- millimeter
27. ng- nanogram
28. NPDES- National Pollutant Discharge Elimination System
29. P&T- purge and trap
30. QC- quality control
31. %R- percent recovery
32. RPD- relative percent difference
33. RRT- relative retention time
34. %RSD- percent relative standard deviation
35. SOP- Standard Operating Procedure
36. Surr.- surrogate
37. SPCC- System Performance Check Compound
38. TCLP- Toxicity Characteristic Leaching Procedure
39. USACE- United States Army Corps Of Engineers

Refer to SOP-431 for additional definitions

FORM 3

WATER PAH LAB CONTROL SAMPLE (8270C)

Lab Name: ELAB Contract:

Lab Code: NA Batch No.: NA SAS No.: NA SDG No.:

Matrix Spike - Client Sample No.:

| COMPOUND | SPIKE ADDED (ug/L) | SAMPLE CONCENTRATION (ug/L) | LCS CONCENTRATION (ug/L) | LCS % REC # | QC. LIMITS REC. |
|---|---|---|---|---|---|
| Acenaphthene | 1.00 | 0.0000 | 1.00 | 100 | 41-132 |
| Acenaphthylene | 1.00 | 0.0000 | 1.00 | 100 | 43-140 |
| Anthracene | 1.00 | 0.0000 | 1.00 | 100 | 50-139 |
| Benzo(a)anthracene | 1.00 | 0.0000 | 1.00 | 100 | 58-141 |
| Benzo(b)fluoranthene | 1.00 | 0.0000 | 1.00 | 100 | 42-156 |
| Benzo(k)fluoranthene | 1.00 | 0.0000 | 1.00 | 100 | 49-165 |
| Benzo(g,h,i)perylene | 1.00 | 0.0000 | 1.00 | 100 | 12-171 |
| Benzo(a)pyrene | 1.00 | 0.0000 | 1.00 | 100 | 31-142 |
| Chrysene | 1.00 | 0.0000 | 1.00 | 100 | 51-155 |
| Dibenz(a,h)anthracene | 1.00 | 0.0000 | 1.00 | 100 | 28-153 |
| Fluoranthene | 1.00 | 0.0000 | 1.00 | 100 | 47-158 |
| Fluorene | 1.00 | 0.0000 | 1.00 | 100 | 40-140 |
| Indeno(1,2,3-cd)pyrene | 1.00 | 0.0000 | 1.00 | 100 | 20-167 |
| Naphthalene | 1.00 | 0.0000 | 1.00 | 100 | 39-125 |
| Phenanthrene | 1.00 | 0.0000 | 1.00 | 100 | 46-144 |
| Pyrene | 1.00 | 0.0000 | 1.00 | 100 | 39-158 |
| 2-Methylnaphthalene | 1.00 | 0.0000 | 1.00 | 100 | 36-121 |
| 1-Methylnaphthalene | 1.00 | 0.0000 | 1.00 | 100 | 35-131 |

# Column to be used to flag recovery and RPD values with an asterisk

* Values outside of QC limits

COMMENTS: _____

page 1 of 1

FORM III SV
FORM 3

SOIL PAH LAB CONTROL SAMPLE (8270C)

Lab Name: ELAB Contract:

Lab Code: NA Batch No.: NA SAS No.: NA SDG No.:

Matrix Spike - Client Sample No.:

| COMPOUND | SPIKE ADDED (ug/Kg) | SAMPLE CONCENTRATION (ug/Kg) | LCS CONCENTRATION (ug/Kg) | LCS % REC # | QC. LIMITS REC. |
|---|---|---|---|---|---|
| Acenaphthene | 67.00 | 0.0000 | 67.00 | 100 | 28-110 |
| Acenaphthylene | 67.00 | 0.0000 | 67.00 | 100 | 23-126 |
| Anthracene | 67.00 | 0.0000 | 67.00 | 100 | 28-136 |
| Benzo(a)anthracene | 67.00 | 0.0000 | 67.00 | 100 | 31-146 |
| Benzo(b)fluoranthene | 67.00 | 0.0000 | 67.00 | 100 | 30-139 |
| Benzo(k)fluoranthene | 67.00 | 0.0000 | 67.00 | 100 | 42-129 |
| Benzo(g,h,i)perylene | 67.00 | 0.0000 | 67.00 | 100 | 21-149 |
| Benzo(a)pyrene | 67.00 | 0.0000 | 67.00 | 100 | 28-128 |
| Chrysene | 67.00 | 0.0000 | 67.00 | 100 | 39-134 |
| Dibenz(a,h)anthracene | 67.00 | 0.0000 | 67.00 | 100 | 30-138 |
| Fluoranthene | 67.00 | 0.0000 | 67.00 | 100 | 30-142 |
| Fluorene | 67.00 | 0.0000 | 67.00 | 100 | 27-116 |
| Indeno(1,2,3-cd)pyrene | 67.00 | 0.0000 | 67.00 | 100 | 17-164 |
| Naphthalene | 67.00 | 0.0000 | 67.00 | 100 | 29-106 |
| Phenanthrene | 67.00 | 0.0000 | 67.00 | 100 | 32-127 |
| Pyrene | 67.00 | 0.0000 | 67.00 | 100 | 28-130 |
| 2-Methylnaphthalene | 67.00 | 0.0000 | 67.00 | 100 | 30-111 |
| 1-Methylnaphthalene | 67.00 | 0.0000 | 67.00 | 100 | 30-111 |

# Column to be used to flag recovery and RPD values with an asterisk

* Values outside of QC limits

**For 8270-SIM projects, the AFCEE QAPP Ver. 4.0 limits for PAHs in soil and water will be used.**

**PAH Mix**

Acenaphthene
Acenapthylene
Anthracene
Benzo(a)anthracene
Benzo(b)fluoranthene
Benzo(k)fluoranthene
Benzo(g,h,i)perylene
Benzo(a)pyrene
Chrysene
Dibenz(a,h)anthracene
Fluoranthene
Fluorene
Indeno(1,2,3-cd)pyrene
2-Methylnaphthalene
1-Methylnaphthalene
Naphthalene
Phenanthrene
Pyrene

**BN Surrogates**

2-Fluorobiphenyl
Terphenyl-d14

**Internal Standards**

Perylene-d12
Phenanthrene-d10

## ANALYST DATA REVIEW CHECKLIST

| | |
|---|---|
| **Sample Number(s):** | |
| **Batch Number(s):** | |
| **Method:** | **8260/8270 (Circle One)** |

| QA/QC Item | Yes | No | NA | Second Level Review |
|---|---|---|---|---|
| 1. Is the BFB/DFTPP tune performed every 12 hours and is the tuning criteria met? | | | | |
| 2. Are the RRFs and % RSDs within QC limits for appropriate analytes for the initial calibration? Check the retention times for compounds with the same spectra. Check compounds with different conc.( eg. m/p-xylene, ketones,etc.). | | | | |
| 3. Was the initial calibration curve verified by a second source calibration standard (ICV) and have criteria been met? | | | | |
| 4. Does the Continuing Calibration Standard (CCV) meet the criteria for the CCCs and SPCCs. | | | | |
| 5. Is the Method Blank run at the desired frequency and is its concentration for target analytes less than the MDLs? | | | | |
| 6. Are the LCS, MS, MSD within control limits and run at the desired frequency? | | | | |
| 7. Are all sample holding times met, analytes within calibration range, IS areas and surrogate recoveries within QC limits? | | | | |
| 8. Was the Method Blank, LCS, MS, MSD and samples loaded to the GCMS_LFSYS Tablespace within the Target DB Database? | | | | |

Comments on any "No" response:

Primary-Level Review: _____ Date: _____

Second-Level Review: _____ Date: _____

# GC/MS SEMI-VOLATILE BNA-AQUEOUS MATRIX EXTRACTION USING SW-846 METHOD 3510C FOR 8270C/625 ANALYSIS

**SOP NUMBER:** **SOP-300**

**REVISION NUMBER:** **17**

**APPROVED BY:**

**SECTION MANAGER**

**QUALITY ASSURANCE OFFICER**

**EFFECTIVE DATE:** **09/23/08**

**DATE OF LAST REVIEW:** **09/23/08**

# GC/MS BNA - AQUEOUS MATRIX EXTRACTION USING SW846 METHOD 3510C/8270C, 625

## I. SCOPE AND APPLICATION/SUMMARY

1. This SOP describes the extraction of BNAs from water by separatory funnel extraction using SW846 Method 3510C and 625. Samples are extracted with methylene chloride and concentrated to an appropriate final volume.

## II. INTERFERENCES

1. Solvents, reagents, glassware, and other sample processing apparatus can add interferences to sample analysis. Method blanks must be extracted under the same conditions as samples to demonstrate freedom from interferences.
2. Phthalate esters commonly found in plastics can interfere with the analysis. Plastics should be avoided.
3. Soap residue can degrade certain analytes such as aldrin and heptachlor. Glassware should be solvent rinsed to avoid this problem.

## III. APPARATUS AND MATERIALS

- Separatory Funnel - 2-Liter with Teflon stopcock
- Beaker - 400 mL
- Drying /Chromatographic column - 20 mm I.D. x 300 mm or funnel
- Turbo-Vap evaporation tube - 200 mL tube made by Zymark to fit into Turbo-Vap evaporator
- Metal rack - capable of holding six glass evaporation tubes
- Turbo-Vap Evaporator - heated and capable of temperature control (±5°C); the bath should be vented into a hood.
- Vials - 2 mL glass amber, with Teflon-lined screw cap and 40 mL with Teflon lid.
- pH indicator paper - close range (1.0 - 2.0) and (10.0 - 12.0); wide range (1.0 - 12.0)
- Syringe - 1 mL, 500 mL
- Graduated cylinder - Glass, Class A, 1000 mL, 500 mL, and 100 mL
- Pasteur pipette - length 9" and 5-3/4"
- Pasteur pipette bulb
- Labels - Avery
- Teflon Bottles - 250 mL and 1000 mL
- Ring stand - 3 prong
- Aluminum foil - heavy duty
- 10 mL disposable pipette
- Nitrogen tank - equipped with pressure regulator

## IV. REAGENTS

- Reagent Water - Reagent water is gathered in a carboy from source in the instrument lab daily. Remaining water in the carboy is dumped at the end of each day.

- Sodium Hydroxide Solution - (10N), Weigh 400 g NaOH (purchased in a plastic container from Fisher # S318-3 or equivalent) into a 1200 mL fleaker beaker and cover with reagent water. Swirl until all pellets are dissolved. This mixture gets very hot. Let stand until cool. Transfer to a 1-liter volumetric flask with several rinses of reagent water and dilute to 1 liter with reagent water. Transfer to a 1000-mL Teflon container.

- Sodium Sulfate - Granular, anhydrous, trace pure 10 - 60 mesh (purchased in plastic bulk containers from Fisher # S415-10S or equivalent) placed in Pyrex tray and heated at 400°C for a minimum of 4 hrs, removed and cooled in open air in the extraction lab, placed in a 2.5 kg glass amber jug and left at room temperature.

- Glass Wool - Silane Treated (purchased from Supelco #2-0410 or equivalent).

- Sulfuric Acid Solution - (1:1), slowly add 500 mL of H2SO4 (Baker, suitable for trace metal analysis #9673-33 or equivalent) to 500 mL of reagent water in a 1000 mL Teflon container. This mixture will get very warm. Allow to cool before use.

- Extraction Solvent - Methylene Chloride **(Please read SOP-336 before handling this solvent in our laboratory.)** (Dichloromethane - Omnisolv - suitable for spectrophotometry and gas chromatography #DX0831-1 or equivalent).

- The GC/MS operator makes up all surrogates and spikes. Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes.

  **BNA Surrogate** - The base neutral and acid surrogates are normally mixed together in one solution. This solution is purchased from a reputable vendor. Use 0.5 mL of this solution per 1000 mLs of aqueous sample for surrogate amount of 100:200 ug/mL per sample. **(For low level PAHs use 1.0ml of a 1.0µg/mL BN Surrogate spiking solution.)**

  **BNA Spiking Solution** - The base neutral and acid spiking solutions are normally mixed together in one solution ( **This spiking solution contains all the compounds that are normally calibrated by GC/MS** ). This solution is prepared in methanol by making a dilution of stock purchased from a reputable vendor. Use 0.5 mL of this solution per 1000 mLs of aqueous sample for LCS amount of 100ppm per sample. There are two separate spiking solutions available – one solution has a more complete list of BNA compounds than the other which is called the short or matrix spike list. The long list should be used on all extractions unless your supervisor has approved the short list. The short

list may be used for any 'phenol only' extractions. **(For low level PAHs use 1.0 ml of a 1.0ppm of the LLPAH spiking solution.)**

**BNA TCLP Spike** – 0.5 mL is added per 100-mL volume. Each matrix type must have its own TCLP spike. TCLP spike should be added after the TCLP has been filtered but prior to refrigeration. From the volume provided by Wet Chemistry, remove a 100-mL aliquot into a suitable container with a teflon lid, and spike as indicated above.

## V. PROCEDURE

1. All waters have a seven-day holding time counted from the hour they are sampled. Determine the samples necessary to extract from the following sources (Note: never extract samples of unknown origin without discussion with supervisor):
   - Each day a backlog report will be provided indicating sample numbers with the respective analysis required. Line through all the extractions that have been completed and plan to do the remaining analysis within the required holding time.
   - Samples requiring RUSH turn around time may be logged in throughout the day which will require your immediate attention. Log-in personnel will generally communicate this need.
   - Check with log-in throughout the day and examine the COC (chain of custody) forms that arrive with each set of samples. If an analysis is ongoing, extra QC may be avoided by picking up those extractions on the same day.

2. Wearing lab coat, gloves and safety glasses, get samples from refrigerator. Samples must be signed out of the walk-in refrigerator. Enter the sample numbers, your initials and the date and time removed on the log provided. Inspect as to whether they are in glass amber jar and have a Teflon lid. Find out if any special dilutions need to be made for this client. Routine procedures for difficult matrices are listed below:

   **SLUDGE** - use only 100 mL and dilute to 1000 mL with reagent water.
   **TCLP EXTRACT** - use only 100 mL and dilute to 1000 mL with reagent water. A separate matrix spike of 100 mLs (which has already been spiked as explained in the BNA TCLP Spike section above) should be set up at the same time. Dilute to 1000 mL with reagent water.
   **BAD MATRIX** – for example a liquid that is partially sediment, see your supervisor to find out what dilution, if any should be made. **SPLP extract- use 1 liter.**
   **NPDES client** - a special list of compounds is required including benzidine. Method 625 requires that there be a spike every ten samples. The sample must be extracted and concentrated in the same day. A GC/MS screen is recommended; therefore this extraction should be coordinated with the GC/MS operator. 1 mL is added to the LCS and the matrix spike.
   **ACID EXTRACT WITH BAD MATRIX** - a cleanup step is added. Samples are taken to a high pH, extracted with 60 mLs methylene chloride one time as explained below in the BASE NEUTRAL EXTRACTION section. This extract is discarded. The samples

are then taken to a low pH and extracted as an acid extraction. Acid extractions may be concentrated in the TurboVap.

3. **LOW LEVEL POLYAROMATIC HYDROCARBONS (PAHs)** – Samples require a BNA extraction. Use the Surrogate and BN spiking solutions specified. Low level PAHs are normally concentrated on the Turbo-Vap using Round-Bottom TV tubes to a final volume of 0.5 mL

4. Mark the amber glass container of each sample at the water meniscus with "white out" for later determination of sample volume. Check the pH by inverting the sample and touching the wide range pH paper to the portion that remains on the lid. Record this pH in the logbook.

5. Get out enough separatory funnels to extract the number of samples you have plus any additional spikes and a method blank. A method blank and an LCS must be processed with each set of samples. If the sample is a TCLP, blank fluid may be provided along with the extracted TCLP sample(s). Use only 100 mL and dilute to 1000 mL with reagent water. A matrix spike and a duplicate or a matrix spike duplicate must be processed for each analytical batch (up to a maximum of 20 samples). In the event that adequate sample is not provided to do an MS/MSD, an LCS duplicate should be done. Rinse separatory funnels with methylene chloride. Place an Avery label on each separatory funnel containing the following information: Lab #, Client name, Type of Analysis, Initial Volume-Final Volume, and the Lab prepbatch code. The lab batch code is defined as MMDDYYB# where #: 1 = 1st method blank of the day; 2 = 2nd method blank of the day; etc. The Method Blank and LCS label should include all lab #s in this set of samples.

6. Using the 1000-mL glass graduated cylinder marked NANO PURE WATER ONLY, measure 1000 mL of reagent water from the carboy and transfer it to a separatory funnel for the method blank and LCS. Transfer sample to separatory funnel that corresponds to the lab # on the sample bottle.

7. Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes. Set out the surrogate/spike at least ten minutes before use to allow it to warm to room temperature.

   Generally 0.5 mL of BNA surrogate is added to each sample, spike, and blank with a syringe designated for BNA surrogate. **For low level PAHs use 1.0ml of a 1.0ppm LLPAH Surrogate spiking solution.** Someone must verify that the surrogate has been added by placing a check mark on each label as it is added.

   NOTE: Be sure to invert syringe and eliminate air bubble when obtaining surrogate solution and spiking solution.

For the sample in each analytical batch selected for spiking, use the 0.5-mL glass syringe designated for BNA spike, to add 0.5 mL of BNA spiking solution. **For low level PAHs use 1.0 mL of the 1.0ppm LLPAHs spiking solution.** Someone must verify that the spike has been added by placing a check mark on each label as it is added. **For DOD QSM projects, all target compounds will be spiked into the LCS and MS/MSD.**

Enter the ID# of the surrogate/spike used and the initials of the person that verified their addition to the sample in the BNA logbook.

8. **ACID EXTRACTION:** Adjust the pH to between 1.0 and 2.0, using 2 mL of 1:1 H2SO4. Add to each sample, spike and method blank. Stopper and shake to insure that pH throughout the sample is changed. Check the drop of liquid hanging from the lid with short-range pH paper. Compare the color to the chart on the pH paper. If the color is not within range add more H2SO4 solution in small increments, as required to attain the proper pH.

9. Add 40 mL of Methylene Chloride to each empty sample bottle and to the LCS, method blank and MS/MSD funnels. Swirl the 40-mL of methylene chloride that you added to the empty sample bottle and transfer to the corresponding separatory funnel.

10. Seal and shake the separatory funnel vigorously for 2 minutes with periodic venting to release excess pressure. Alternatively, Teflon funnels may be used and placed in the shaker apparatus with the stopcocks slightly open. When this apparatus is used, the shake should be for 3 minutes.

    **NOTE: Methylene chloride creates excessive pressure very rapidly; therefore, initial venting should be done immediately after the separatory funnel has been sealed and shaken once.**

11. Allow the sample to sit for 10 minutes, if necessary, after it has been shaken. It will separate into two layers with the solvent layer on the bottom. If it forms an emulsion (thick, cloudy, viscous, mixture that you cannot see through), drain what you believe to be 40 mL into a 250 mL centrifuge bottle. If the layers are clearly separated, drain the solvent layer into a 400-mL glass beaker.

12. Following Steps 9 and 10, extract two more times with 40 mLs of methylene chloride. Combine the three solvent extracts into the same 400-mL beaker.

13. **BASE NEUTRAL EXTRACTION: Adjust the pH to 11 or slightly greater**, using 10N NaOH. Start by adding 5.0 mLs to each sample, spike, and method blank. Stopper and shake to insure that pH throughout the sample is changed. Check the drop of liquid hanging from the lid with short-range pH paper. Compare the color to the chart on the pH paper. If the color is not within range add more 10N NaOH in small increments, as required to attain the proper pH. **BNA extraction is necessary when doing low level PAHs.**

**NOTE: This step is critical to the extraction procedure. Too much NaOH solution could cause you to lose certain Base Neutral compounds. Be careful on this step.**

14. **FOR 8270 extraction**: Extract one more time with 40 mL of methylene chloride following Steps 9 and 10. Combine BN and Acid extracts in a same 400ml beaker, unless the BN extract has large amount of emulsion; then it will be necessary to use a separate 400 mL beaker. Concentrate BN and acid extracts for one final extract.
**NOTE: It has been demonstrated that two acid and one BN extraction can be used for normal 8270 samples. This procedure cannot be used for DOD or 625 samples.**

**For 625 extraction:** extract 3 more times with 40 mL methylene chloride following steps 9 and 10. Combine BN extracts in separate 400 mL beaker. Concentrate BN and acid extracts separately for one final extract.

15. In the log book marked BNA extractions, enter the Client name, the Lab #, the date extracted, the initial volume, and 1.0 mL for the final volume and anything unusual that may have occurred with this sample. The final volume for low level PAHs is 0.5 mL.

16. Prepare to dry the sample by either of the following methods;
    16A. Get a ring stand with a double burette clamp attached to it. Cover the burette clamp ends with aluminum foil to prevent the possibility of solvent touching the plastic coated ends and dripping into the extract. Place a drying column into the burette clamp and transfer a small amount of glass wool to the top of it. Tamp it to the bottom with a glass rod so that it adequately covers the hole at the bottom. Add approximately 10 cm of Sodium Sulfate to the column. Rinse with 20 to 30 mL of methylene chloride and discard this rinse into the Chlorinated Waste container in the hood. OR

    16B. Set up a ring stand with funnels. Place a small amount of glass wool in the bottom of it, add ~2" sodium sulfate to the column and rinse with 20-30 mL methylene chloride. Discard this rinse into the Chlorinated Waste container in the hood.

17. If the extract was drained into a centrifuge bottle, at this point you will need to take it to the centrifuge. Push the "ON" button to turn the centrifuge on. Be sure that the large holders are available for the 250-mL centrifuge bottles. The sample must always be balanced. If necessary use a dummy bottle making it similar weight using reagent water. Set the rpm at 2500 and the temperature at 0°C. Close the lid and be sure to press it down until you hear it click. Move the lever at the front of the lid to the "LOCK" position. Turn the time to approximately 15 minutes and bring it back to 10 minutes. As the rotor begins to move, you will be able to see the rpm's in the digital readout. Stay with the centrifuge until it has come up to the rpm's set to insure that it does not become unbalanced. This looks like 8888 on the digital readout. Should this occur, refer to the manual. When the cycle is complete, the digital readout will read 0000. Push the "OPEN" button and the lid will pop up. Move the lever at the front of the lid to the

"UNLOCK" position. Open lid and remove sample. The sample will usually be in two layers with the extract on the bottom.

18. Remove any water layer from the extract in the beaker or centrifuge bottle, by one of two methods. Remove with a Pasteur pipette by carefully pulling up the water layer, on top, and not the solvent. Discard this layer in the sink. Use the smallest amount possible of Na2SO4 by sprinkling the top layer with Na2SO4 until it hardens, separates, and drops to the bottom.

19. TURBO-VAP CONCENTRATION

Low level PAH sample concentration is primarily done by Turbo-Vap using Round-Bottom TV tubes.

- Rinse a Turbo-Vap tube with methylene chloride and arrange it underneath a rinsed, packed drying column or funnel. Pour the extract through the column so that it will collect in the tube. Rinse the 400-mL beaker, which contained the solvent extract twice with 10 to 15 mL of methylene chloride and add each rinse to the column to complete the quantitative transfer. After all the extract has passed through the column, rinse the column with 10 to 15 mL of methylene chloride. Total volume in the glass evaporator tube should not exceed 200 mLs to avoid splattering on the lid of the Turbo-Vap.
- Record the numbers of the Turbo-Vap tube in the BNA logbook and remove the tube to a metal holder. To help prevent cross contamination, place a piece of aluminum foil over the Turbo-Vap tube and punch a small hole in the top so that the nitrogen can be accessed.
- Turbo-Vap Operation: Adjust the pressure of nitrogen gas tank to >30 psi. Make sure the tank has 200 psi or more on the main valve. The temperature of the bath should be approximately 40°C -50°C.
- Place the glass evaporator tube in the Turbo-Vap. Be sure to push tube down so the tip slides into the sensor well. Close the lid to start concentration. Check that each position with a tube has an orange light showing. If the orange light is not steady, bubbles may be in the sensor and need removal. (See Turbo-Vap manual).
- When the beep sounds indicating the end of concentration, the extract will be at approximately one half mL (half way up tip of tube). Remove the tube from the bath. Use a 9" Pasteur pipette to draw up the sample and transfer it to the 2-mL vial. THIS IS THE MOST CRITICAL PART OF THE ENTIRE OPERATION!!! A single drop represents about 10 percent of the total sample. Before you move the tip of the pipette from the tube to the vial, be sure that a drop will not form on the end and fall off.
- Draw ~0.25 mL of methylene chloride into a 0.50 mL syringe and add this aliquot to the centrifuge tube. Draw the methylene chloride into a pipette

and rinse the sides of the tube several times. Transfer this rinse to the 2-mL vial. Add methylene chloride from the syringe and repeat the rinsing process until you have ~ 1 mL in the sample extract vial. Compare this volume to a 2-mL dummy vial containing 1 mL of solvent to insure that you have not exceeded 1 mL. For low level PAHs the final volume is 0.5mL. The methylene chloride rinse volume must be adjusted to achieve this final volume. Compare the volume to a 2mL dummy vial containing 0.5 mL of solvent to insure that you have not exceeded 0.5 mL. The GC/MS operator will adjust the sample to the desired final volume and add internal standard just prior to analyses. Cover the extract with a Teflon-sealed screw cap and transfer the label to the vial.

20. Determine the original sample volume by refilling the sample bottle to the mark made with "white out." Transfer the liquid to a plastic 1000-mL graduated cylinder and record the sample volume in the BNA logbook and the Avery label to the nearest 10-mL.

21. The extract is now ready to be analyzed. Refrigerate at 4°C or carry directly to the instrument operator. Samples must be signed into the Sample Extract refrigerator. On log provided, enter the sample numbers, the analyst initials, and the date and time the samples were placed into the refrigerator.

## VI. DOCUMENTATION OF CAPABILITY ( DOC)

Each analyst must perform a DOC to demonstrate proficiency with this method. Refer to SOP-413 for guidance.

## VII. WASTE MANAGEMENT AND POLLUTION PREVENTION

Please see Waste Disposal SOP-405 for the proper disposal of waste generated from this area.

Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

## VIII. METHOD PERFORMANCE

Refer to SOP-201 for method performance.

## IX. HEALTH AND SAFETY

Refer to the MSDS sheets for the chemicals used for health and safety information. Also see SOP-336 for proper use of methylene chloride.

## REFERENCES

1. *Test Methods for Evaluating Solid Waste*, SW-846, Third Edition
2. 40 CFR, Method 625.

## DEFINITIONS

BNA- base/neutral acid
°C - degrees centigrade
COC - chain of custody
DL - detection limit
g - grams
KD - kuderna danish
LCS - laboratory control sample
µg/L - micrograms per liter
µL - microliter
µg/ml - micrograms per milliliter
ml - milliliter
mm - millimeter
MS - matrix spike
MSD - matrix spike duplicate
PAH- polynuclear aromatic hydrocarbon
RL - reporting limit
SOP - standard operating procedure
v/v - volume to volume

Refer to SOP-431 for further definitions

SOP-300

Fraction: Semi-volatiles Matrix: Water EX0__

Supervisor_____

| # | Client | Lab No. | Date Extracted | Setup Initials | pH | Initial Volume(ml) | Final Volume(ml) | Surrogate Added | Surr Initials | Spike Added | Spk Initials | KD | 10ml Conc. | 15ml Cent. | Conc. Initials | Solvent Lot/Vendor Methylene Chloride | Notes/Comments | # |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | | | | | | | | | | NA | NA | | | | | | | 1 |
| 2 | | | | | | | | | | NA | NA | | | | | | | 2 |
| 3 | | | | | | | | | | NA | NA | | | | | | | 3 |
| 4 | | | | | | | | | | NA | NA | | | | | | | 4 |
| 5 | | | | | | | | | | NA | NA | | | | | | | 5 |
| 6 | | | | | | | | | | NA | NA | | | | | | | 6 |
| 7 | | | | | | | | | | NA | NA | | | | | | | 7 |
| 8 | | | | | | | | | | NA | NA | | | | | | | 8 |
| 9 | | | | | | | | | | NA | NA | | | | | | | 9 |
| 10 | | | | | | | | | | NA | NA | | | | | | | 10 |
| 11 | | | | | | | | | | NA | NA | | | | | | | 11 |
| 12 | | | | | | | | | | NA | NA | | | | | | | 12 |
| 13 | | | | | | | | | | NA | NA | | | | | | | 13 |
| 14 | | | | | | | | | | NA | NA | | | | | | | 14 |
| 15 | | | | | | | | | | NA | NA | | | | | | | 15 |
| 16 | | | | | | | | | | NA | NA | | | | | | | 16 |
| 17 | | | | | | | | | | NA | NA | | | | | | | 17 |
| 18 | | | | | | | | | | NA | NA | | | | | | | 18 |
| 19 | | | | | | | | | | NA | NA | | | | | | | 19 |
| 20 | | | | | | | | | | NA | NA | | | | | | | 20 |
| | | | | | | | | | | | | | | | | | | |
| | | | | | | | | SAS | SAS | | | | | | | SAS | | |
| | | | | | | | | SAS | SAS | | | | | | | SAS | | |
| | | | | | | | | SAS | SAS | | | | | | | SAS | | |
| | | | | | | | | SAS | SAS | | | | | | | SAS | | |
| | | | | | | | | SAS | SAS | | | | | | | SAS | | |
| | | | | | | | | SAS | SAS | | | | | | | SAS | | |
| | | | | | | | | SAS | SAS | | | | | | | SAS | | |
| | | | | | | | | SAS | SAS | | | | | | | SAS | | |
| | | | | | | | | SAS | SAS | | | | | | | SAS | | |
| | | | | | | | | SAS | SAS | | | | | | | SAS | | |
| | | | | | | | | SAS | SAS | | | | | | | SAS | | |
| | | | | | | | | SAS | SAS | | | | | | | SAS | | |
| | | | | | | | | SAS | SAS | | | | | | | SAS | | |
| | | | | | | | | SAS | SAS | | | | | | | SAS | | |
| | | | | | | | | SAS | SAS | | | | | | | SAS | | |
| | | | | | | | | SAS | SAS | | | | | | | SAS | | |
| | | | | | | | | SAS | SAS | | | | | | | SAS | | |
| | | | | | | | | SAS | SAS | | | | | | | SAS | | |

# SOXHLET EXTRACTION - BNA AND PEST/PCB USING SW846 METHOD 3541

**SOP NUMBER:** SOP-329

**REVISION NUMBER:** 16

**APPROVED BY:**

SECTION MANAGER

QUALITY ASSURANCE OFFICER

**EFFECTIVE DATE:** 09/24/08

**DATE OF LAST REVIEW:** 09/24/08

# BNA& Pesticide/PCB NON-AQUEOUS MATRIX (Soxhlet Extraction) USING SW846 METHOD 3541

## I. SCOPE AND APPLICATION

1. This SOP describes the extraction of BNAs and pesticides/PCBs from soil, sediment, sludges and waste solids by an automated method (3541).

## II. SUMMARY

1. Soil and solid samples are mixed with sodium sulfate and extracted with solvent in a Soxtherm extractor for BNAS or Pesticides/PCBs.. The extracts are then concentrated by either a TurboVap concentrator or by Kuderna- Danish.

## III. INTERFERENCES

1. Solvents, reagents, glassware, and other sample processing apparatus can add interferences to sample analysis. Method blanks must be extracted under the same conditions as samples to demonstrate freedom from interferences.
2. Phthalate esters commonly found in plastics can interfere with the analysis. Plastics should be avoided.
3. Soap residue can degrade certain analytes such as aldrin and heptachlor. Glassware should be solvent rinsed to avoid this problem.

## IV. APPARATUS AND MATERIALS

- Stainless steel spatula
- Soxtherm automated extractor unit-with 6 position condenser assemblies, internal plumbing, electronic components, stand alone controller unit and a copper collection tank for spent solvent
- Soxtherm extraction beakers-54 x 130 mm, capacity approximately 125-175 mL
- Suitable thimble (33 mm diameter by 80 to 94 mm length cellulose or equivalent
- Drying Column (Chromatographic column) - 20mm I.D. x 300mm
- Boiling chips - Teflon
- Vial – 2-mL amber with Teflon-lined screw cap
- Vial – 12-mL clear with Teflon-lined screw cap
- Syringe - 1 mL, 500 µL
- Graduated cylinder - Glass, Class A, 100 mL
- Pasteur pipette - length 9" and 5-3/4"
- Pasteur pipette bulb
- Labels - Avery
- Ring stand - 3 prong
- Burette clamp - double
- Rings

- Aluminum foil - heavy duty
- Nitrogen tank - equipped with pressure regulator
- TurboVap Concentrator with 200 ml concentrator tubes
- Balance - capable of weighing to 0.1 grams.
- Aluminum pie pans for mixing samples
- Glass wool- Contaminant free.

## V. REAGENTS

- Sodium Sulfate (Na2SO4) - Granular, anhydrous, trace pure 10-60 mesh (purchased in bulk containers from Fisher # S415-10S or equivalent) placed in a Pyrex tray and heated at 400 C for a minimum of 4 hrs, removed and then cooled in open air in the extraction lab, placed in a 2.5kg glass amber jug and left at room temperature.

- Glass Wool - Silane Treated (purchased from Supelco #2-0410 or equivalent).

- Methylene Chloride **(Please read SOP-336 before handling this solvent in our laboratory.)** (Dichloromethane) - suitable for spectrophotometry and gas chromatography (Omnisolv #DX0831-1 or equivalent)

- Acetone - suitable for spectrophotometry and gas chromatography (Omnisolv AX0116-1 or equivalent)

- Hexane - suitable for spectrophotometry and gas chromatography (Omnisolv HX0298-1 or equivalent)

- Hexane/Acetone (1:1) (v/v) of the above reagent grades.

- Methylene Chloride/Acetone (1:1) (v/v) of the above reagent grades.

- Surrogate/Spike Solutions - Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes or if the initial concentration of stock is different than that listed below:

  1. **BNA Surrogate (100 ug/ml)** - The base neutral and acid surrogates are mixed together in one solution. This solution is prepared in methanol by making a dilution of stock purchased from a reputable vendor. Use 0.5 mL of this solution per 15g of non-aqueous sample. **(For low level PAHs use 1.0 ml of 1.0 µg/mL BN Surrogate spiking solution.)**
  2. **BNA Spiking Solution (100 ug/ml)** - The base neutral and acid spiking solutions are mixed together in one solution. This solution is prepared in methanol by making a dilution of stock purchased from a reputable vendor with same compounds as for calibration. Use 0.5 mL of this solution per 15g of non-aqueous sample.**(For low level PAHs use 1.0 ml of 1.0 µg/mL PAH spiking solution.) The BNA Spiking solutions contains all targets that are**

**calibrated for GC/MS. DOD QSM requires all targets to be spiked in the LCS and MS/MSD.**

3. **TCMX/DCB (2,4,5,6-Tetrachloro-meta-xylene/Decachlorobiphenyl)Warm and sonicate** - Surrogate solution is prepared in acetone by making a cut on stock purchased from a reputable vendor. 0.5 mL at 0.5 µg/mL of this solution is added per 15g of non-aqueous sample.
4. **PCB Spiking Solution-** Arochlor 1016/1260 or the PCB of choice (1242, 1248, 1254, or 1260 are the most common) is prepared in acetone at a concentration of 5.0 ug/ml. PCB stock is usually purchased from RESTEK or equivalent. The PCB to use may be determined by viewing historical data or asking the GC operator. Use 0.5 mL per 15.0 grams of non-aqueous sample.
5. **Pesticide Spiking Solution** - A spiking solution is prepared at 1.0 ug/ml. Use 0.5 mL per 15 grams of non-aqueous sample.

## VI. SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIMES

1. Samples are collected in an appropriate size wide-mouth glass jar (4oz. or 8 oz.) with a teflon-lined cap.
2. Samples are preserved by cooling to 4° C.
3. Holding time is 14 days from collection date to extraction.

## VII. PROCEDURE

### Extraction Procedure

1. All soils have a 14 day holding time counted from the day they are sampled.Determine the samples necessary to extract using the following information (DO NOT extract samples for which you have no information.):
   - Each day a backlog report will be provided indicating sample numbers with the respective analysis required. Line through all the extractions that have been completed and plan to do the remaining analysis within the required holding time.
   - Samples requiring RUSH turn around time may be logged in throughout the day which will require your immediate attention. Log-in personnel will generally communicate this need.
   - Check with log-in throughout the day and examine the COC (chain of custody) forms that arrive with each set of samples. If an analysis is ongoing, extra QC may be avoided by picking up those extractions on the same day.

2. Wearing lab coat, gloves and safety glasses, get samples from cooler. Samples must be signed out of the walk-in refrigerator. Enter the sample numbers, your initials and the date and time removed on the log provided. Inspect as to whether they are in glass and have a Teflon lid. Find out if any special dilutions need to be made for this client. If the sample has a particularly bad matrix or a strange matrix, see your supervisor to find out if a soxhlet extraction is truly necessary.

3. Get twice the number of aluminum pie pans to prepare the number of samples you have plus any additional spikes or LCSs and a method blank. A method blank and LCS must be processed with each set of samples. A matrix spike, a duplicate or a matrix spike duplicate and a LCS must be processed for each analytical batch (up to a maximum of 20 samples). Make an Avery label for each extract containing the following information: Lab #, Client name, Type of Analysis, Initial Weight - Final Volume, and the Lab prep batch code. The lab prep batch code for BNAs is defined as MMDDYYBs# where #: 1 = 1st method blank of the day; 2 = 2nd method blank of the day; etc. The lab prep batch code for Pest/PCBs is defined as MMDDYYPs# using the same logic. The Method Blank and LCS labels must include all lab #s in this set of samples.

4. Decant and discard any water layer on a sediment sample by carefully pouring this off into a trashcan.

5. Dump the entire sample into an aluminum pie pan and mix sample thoroughly with a spatula until mixture is homogenous. Discard any foreign objects such as sticks, leaves, and rocks.

*It is extremely important that waste (when appropriate), soil and sediment samples be mixed thoroughly to ensure that the sample is as representative as possible of the sample media. The most common method of mixing is referred to as quartering. The quartering procedure should be performed as follows:*

- *The material in the sample pan(inorganic-plastic/organic-aluminum) should be divided into quarters and each quarter should be mixed individually.*
- *Two quarters should then be mixed to form halves.*
- *The two halves should be mixed to form a homogenous matrix.*

*This procedure should be repeated several times until the sample is adequately mixed.*

**NOTE: Samples that are clay type materials should be handled in a different manner. Due to these type sample matrices having an affinity to stick to most anything that touches it, another approach must be followed. Obtain a representative sub-sample aliquot from the center or middle section of the sample container**

6. Place an aluminum pie pan on the balance and zero it. Calibrate balance with ASTM class-1 Troemner weights or equivalent, bracketing desired weight (50g, 20g, 10g, 5g, 1g). Record calibration in the Extraction calibration/temperature logbook. Using a spatula, transfer the **appropriate weight, {10-20 grams depending upon client or project specific Detection Limits (DL) and/or Reporting Limits (RL)}**, of a representative sample to the nearest 0.1 gram. Normally 10 or 15 gram sample weights are used. Record this amount on your label. Put your label on the side of the 400-mL beaker. For spiking purposes, weigh 3 aliquots of the appropriate sample. Pick a sample with a good matrix, one that mixes well, non-oily, etc.

7. Add ~ 20 grams of sodium sulfate to the aluminum pie pan. Using a spatula and/or a glass rod, mix the sample thoroughly with the sodium sulfate until it becomes a sandy texture. If necessary, add additional sodium sulfate. When removing the spatula or glass rod from the mixed sample, leave behind all the sample possible. Cover the aluminum pie pan with foil and continue to weigh up the remaining samples. For the method blank and LCS, simply weigh up 15 grams of sodium sulfate. The matrix used for the method blank and LCS must be free of the analytes of interest and processed through the same analytical steps as the samples.

8. Transfer the sample and its label to a soxtherm beaker, which has been prepared using a glass rod to place glass wool in and over the hole at the bottom side.

9. Make up 1:1 Methylene Chloride/Acetone (**for BNAs**) or 1:1 Hexane/Acetone (**for Pest/PCBs**) by mixing 2000ml of methylene chloride or hexane with 2000 ml of acetone in a 4000ml amber jug. Add 120 mL of this mixture to the soxtherm beaker for each sample, method blank, and spike - add boiling chips.

10. Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes. Set out the surrogate/spike at least ten minutes before use to allow it to warm to room temperature. Someone must verify that the surrogate/spike has been added by placing a check mark on the label as it is added.

    NOTE: Surrogate and spike should be added just prior to setting on the Soxtherm.

    Using the 1-mL glass syringe designated for BNA surrogate, add 0.5 mL of BNA surrogate to each sample, spike, and blank. **(For low level PAHs use 1.0 ml of the 1.0 µg/mL BN Surrogate spiking solution.)** or using the 1.0-mL glass syringe marked TCMX/DCB surrogate, add 0.5 mL of TCMX/DCB surrogate to each sample, blank and spike.

    For the BNA sample in each analytical batch selected for spiking, use the 1.0-mL glass syringe marked Base Neutral Acid Spiking to add 0.5 mL of the Base Neutral Acid Spiking solution. . **(For low level PAHs use 1.0 ml of the 1.0µg/mL PAH spiking solution.)**
    For Pest/PCB samples, determine if the sample will require a Pesticide Spike and/or a PCB Spike. Proceed as follows:
    **Pesticide and PCB** - set up two LCS's – one for Pesticide getting a MIX A&B spike and one for PCB which should be spiked with PCB 1660. In addition to the LCSs, a matrix spike/matrix spike duplicate is necessary for the pesticide. Prepare a PCB matrix spike/ matrix spike duplicate if requested by the client.
    **Pesticide only** – To the sample in each analytical batch selected for spiking, add 0.5 mL of Pesticide Spike (Mix A&B) with a glass syringe dedicated for Pesticide Spike.
    **PCB only** - To the sample in each analytical batch selected for spiking, add 0.5 mL of PCB 1016/1260 (unless otherwise specified, 1248 for BB&L) using a 1.0 mL glass syringe dedicated to that PCB. Add 20 grams of $Na_2SO_4$.

11. **Automated Soxhlet:** The *Soxtherm* will extract any number of samples up to six per run. Generally, we set up to complete four sets of six a day or/as sample load permits with required QC. Each extraction cycle takes 1-2 hours. Reference *O.I. Analytical Operator's Manual* for programming and making adjustments to extraction cycle settings.

- The extraction thimbles are placed into the respective extraction thimble holders(the open end of the thimble is nearly flush with the upper edge of the metal ring. Transfer the sample into the extraction thimble. The appropriate spiking solution should be added at this point (surrogates, MS/MSD or LCS etc…). (Placing a small piece of glass wool sufficient to cover the diameter of the thimble will help hold the sample mixture in the thimble.)

- Place 1-2 boiling chips in each extraction beaker and place each extraction thimble into the appropriate labeled beaker then add 120 mLs of the appropriate extraction solvent. (1:1 Acetone/Hexane for Pest/PCBs **or** 1:1 Acetone/Methylene Chloride for BNAs.)

- For loading samples on the *Soxtherm,* the condensers should be in their raised position. If Viton gaskets are to be used, install them in the extraction beakers so that a good seal will form between the extraction beaker and the Teflon extraction cylinder. Install the extraction beakers by depressing the holding clamp and carefully sliding the beaker onto or off the bottom of the Teflon fitting that is situated below the glass condenser. After the extraction beakers containing the samples have been loaded on the *Soxtherm,* push all beakers against the rear limit stop of the hot plate.

- The *Soxtherm* is now ready to run with one of the programs listed below (**remember to check the sight glass on the front of the Extraction Unit to see if the solvent collection tank is near full and should be drained).**

**Pest/PCB Extraction Program**

| | |
|---|---|
| Temperature | = 165°C (Maximum temperature 200°C) |
| Boiling Time | = 30 minutes |
| Solution Reduction A | = 5x15 ml |
| Extraction Time | = 30 minutes |
| Solution Reduction B | = 0 minutes |
| Solution Cooling | = 0 minutes |
| Solution Reduction Interval | = 2.5 minutes |
| Solution Reduction Pulse | = 2 seconds |

**BNA Extraction Program**

| | |
|---|---|
| Temperature | = 160°C (Maximum temperature 200°C) |
| Boiling Time | = 30 minutes |
| Solution Reduction A | = 5x15ml |
| Extraction Temperature | = 30 minutes |
| Solution Reduction B | = 0 minutes |

| | |
|---|---|
| Solution Cooling | = 0 minutes |
| Solution Reduction Interval | = 2.5 minutes |
| Solution Reduction Pulse | = 2 seconds |

- At the end of Solution Cooling time the condensers and extraction beaker assemblies are automatically raised off the heating block and the process completion message appears on the display. Remove the beakers to a hood and continue with the process as listed below.

13. Enter the following information in the BNA, Pesticide and/or PCB extractions logbook: Client name, Lab #, date extracted, initial weight, final volume, soxtherm position, and anything unusual that may have occurred with this sample. Enter the ID# of the surrogate and spike used and the initials of the person that verified their addition to the sample in the appropriate logbook. Also record the vendor and lot number of the methylene chloride or hexane and acetone used.

14. Dry the sample by either of the following methods:

14A. Get a ring stand with a double burette clamp attached to it. Cover the burette clamp ends with aluminum foil to prevent the possibility of solvent touching the plastic coated ends and dripping into the extract. Place a drying column into the burette clamp and transfer a small amount of glass wool to the top of it. Tamp it to the bottom with a glass rod so that it adequately covers the hole at the bottom. Add approximately 10 cm of Sodium Sulfate to the column. Rinse with 20 to 30 mL of methylene chloride or hexane and discard this rinse into the appropriate waste container (make certain that methylene chloride waste goes in the chlorinated solvent waste container in the hood).

14B. Set up a ring stand with funnels. Place a small amount of glass wool in the bottom of it. Add ~2" sodium sulfate to the column and rinse with 20-30 mL methylene chloride or hexane for Pest/PCBs. Discard this rinse.

15. TURBO-VAP CONCENTRATION

A. Turn on the TurboVap and set the temperature at 40°C -50°C. This temperature is necessary to evaporate the acetone. Adjust pressure of nitrogen gas tank to >30 psi. Make sure the tank has 200 psi or more on the main valve.

B. Rinse a Turbo-Vap tube and arrange it underneath a rinsed, packed drying column/funnel. Pour the extract through the column/funnel so that it will collect in the tube. Total volume in the glass evaporator tube should not exceed 200 mLs to avoid splattering on the lid of the Turbo-Vap. Therefore the process cannot be completed in one step. Pour enough of the extract from the 400 mL beaker to reach the 200 mL level mark on the Turbo-Vap tube. To help prevent cross contamination, cover the turbovap tube with aluminum foil and punch a small hole in the top of it.

A portion of the extract will remain in the beaker. This should be set aside with the funnel used to dry the first portion of the sample placed in the mouth of the beaker.

C. Record the Turbo-Vap tube number, the vendor and lot number of hexane used in the appropriate extraction logbook.

D. Place the glass evaporator tube in the Turbo-Vap. Be sure to push tube down so the tip slides into the sensor well. Close the lid to start concentration. Check that each position with a tube has an orange light showing. If the orange light is not steady, bubbles may be in the sensor and need removal. (See Turbo-Vap manual).

E. When the extract has been concentrated to ~1.0 mL, remove it from the Turbo-Vap and find the remaining extract. Reuse the original funnel and pour the remaining extract through it so that it drains into the turbovap tube. Rinse the 400 mL beaker twice with 10 -20 mLs of methylene chloride or hexane for Pest/PCBs transferring the rinse to the funnel each time. Finally rinse the funnel with ~ 10 mLs of solvent. Place a piece of aluminum foil over the tube and with a sharpie mark an "X" on the aluminum foil. Remove the tube to a metal holder. Concentrate as before.

F. When the beep sounds indicating the end of concentration, the extract will be at approximately one half mL, remove the tube from the bath. The extracts are ready to be transferred into their appropriate vials.

- **For Pest/PCB Completion:** Hold the tube and the sample vial in one hand at about a 45° angle. Use a 9" pasteur pipette to draw up the sample and transfer it to the 10 mL vial. Be careful not to spill any sample during the transfer because one drop represents 5% of the sample and is a critical loss.
- Fill a 10 mL graduated cylinder with 9.5 mL of hexane. Add 1/2 of the hexane to the tube. Draw into the pipette and rinse down the conical portion of the tube several times. Transfer this rinse to the 10 mL vial. Add the remaining hexane and repeat this rinsing process. Cover the extract with a Teflon-sealed screw cap.

- **BNA Completion:** When the volume has been reduced to 1-0.5(~0.4)mL, remove the tube from the Turbo-Vap and place in a metal holder.
- Use a 9" Pasteur pipette to transfer the extract to a 2 mL amber vial. THIS IS THE MOST CRITICAL PART OF THE ENTIRE OPERATION!!! A single drop represents about 10 percent of the total sample. Before you move the tip of the pipette from the tube to the vial, be sure that a drop will not form on the end and fall off.
- Draw ~0.20 mL of methylene chloride into a 0.50 mL syringe and add ~ half of this aliquot to the tube. Draw the methylene chloride into a pipette and rinse the sides of the tube several times. Transfer this rinse to the 2 mL vial. Add methylene chloride from the syringe and repeat the rinsing process until you have ~ 1 mL in the sample extract vial.

- Compare this volume to a 2 mL dummy vial containing 1 mL of solvent to insure that you have not exceeded 1 mL. For low level PAHs the final volume is 0.5mL. The methylene chloride rinse volume must be adjusted to achieve this final volume.

Compare the volume to a 2mL dummy vial containing 0.5 mL of solvent to insure that you have not exceeded 0.5 mL. The GC/MS operator will adjust the sample to the desired final volume and add internal standard just prior to analyses. Cover the extract with a Teflon-sealed screw cap and transfer the label to the vial.

NOTE: If the final extract is yellow or dark in color or the matrix is oily and viscous, further cleanup may be desired. Refer to SOPs 307 to 309 and 330, 331, 333 for further cleanup information. Discuss cleanup possibilities with the analyst and/or your supervisor.

The extract obtained above may now be analyzed. Refrigerate at 4°C or carry directly to the analyst. Samples must be signed into the Sample Extract refrigerator. On log provided, enter the sample numbers, your initials, and the date and time the samples were placed into the refrigerator.

Determination of % Dry Weight – Weigh 5-10 grams of the sample from the bulk jar used for dry weight analysis in a tared crucible or aluminum pan. Dry overnight at 105°C. Allow to cool in a dessicator before weighing. Calculate % dry weight as follows:

$$\% \text{ dry weight} = \frac{\text{g of dry sample}}{\text{g of sample}} \times 100$$

## VIII. DOCUMENTATION OF CAPABILITY ( DOC)

Each analyst must perform a DOC to demonstrate proficiency with this method. Refer to SOP-413 for guidance.

## IX. WASTE MANAGEMENT AND POLLUTION PREVENTION

Please see Waste Disposal SOP-405 for the proper disposal of waste generated from this area.

Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

## X. METHOD PERFORMANCE

Refer to SOP-201 and SOP-211 for method performance.

## XI. REFERENCES

1. Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III); Method 3541.

## DEFINITIONS

BNA - Base/ Neutral/Acid
°C - degrees centigrade
COC - chain of custody
DL - detection limit
g - grams
KD - kuderna danish
LCS - laboratory control sample
µg/L - micrograms per liter
µL - microliter
µg/ml - micrograms per milliliter
ml - milliliter
mm - millimeter
MS - matrix spike
MSD - matrix spike duplicate
PAH - polynuclear aromatic hydrocarbons
PCBs - polychlorinated biphenyls
Pest - pesticides
RL - reporting limit
SOP - standard operating procedure
TCMX - tetrachloro-m-xylene
v/v - volume to volume

Refer to SOP-431 for further definitions

# LABORATORY SAMPLE RECEIVING, LOG IN AND STORAGE STANDARD OPERATING PROCEDURES

**SOP NUMBER:** SOP-404

**REVISION NUMBER:** 12

**APPROVED BY:**

SECTION MANAGER

TECHNICAL DIRECTOR

**EFFECTIVE DATE:** 01/05/09

**DATE OF LAST REVIEW :** 01/05/09

# LABORATORY SAMPLE RECEIVING, LOG IN AND STORAGE

This SOP lists in as much detail as possible our daily procedures for sample receiving, log in and storage of laboratory samples. Keep in mind that there may be project specific requirements that are more strict or different than our routine procedures. In these instances, the project specific requirements must be met and followed. Although a few project specific requirements are detailed in this SOP, i.e. USACE certification issues, not every situation can be addressed. If there is ever any uncertainty on what procedures must be followed, please see the Testing Coordinator immediately. If ever in doubt, always go with the more stringent requirements. This document will constantly be reviewed and revised as necessary.

## SAMPLE ACCEPTANCE CRITERIA

A sample may be rejected for compliance purposes if it does not meet the following criteria. Analyses may only proceed after notification and approval to proceed from the client or from the laboratory manager.

1. Sample must be properly preserved and in the proper container for the requested analysis.
2. Sample integrity must be maintained. The container shall be intact without cracks, leaks, or broken seals.
3. Adequate sample volume must be received for the requested analysis, including volume for any requested QA/QC (MS/MSD).
4. The sample ID on the bottle label must match the sample ID listed on the chain of custody.
5. The sample container label and the chain of custody must be completed with indelible ink. The sample label must be intact and list all necessary information; to include: sample date, sample time, sampler, and sample ID/location. The chain of custody shall also indicate sample date and time, requested analyses, and all necessary client information.
6. Sample temperature must be less than 6°C or received on ice.
7. Sample must be within holding time for the requested analysis.

These issues are discussed in more detail below under the "Sample Receiving" section of this document.

## I. Sample Receiving

A. Samples are received at the Empirical Laboratories on French Landing Drive in Nashville, TN.

1. The majority of samples are shipped in coolers by couriers such as Federal Express and UPS. All couriers are generally received in the Empirical Laboratories Sample Receiving (SR) area on the fifth floor. The laboratory is located close to the Federal Express (FedEx) distribution station, therefore we often pick up our coolers at the FedEx

location and transport them back directly to the laboratory. Some coolers and/or samples are delivered directly to the SR area by the sampler and/or client.

2. Some coolers and/or samples may be received directly by Empirical Laboratories Sample Receiving personnel. If samples are hand delivered by the client make sure that necessary paperwork is included and that you sign and date the chain of custody. If the ***Empirical Laboratories Chain of Custody [Attachment II]*** is used the white and yellow copy of the chain of custody is retained and the pink copy must be given to the client.

B. When going through the required steps for Sample Receiving and Sample Log In, keep in mind that a ***Corrective Action Report (CAR) for Sample Receiving [Attachment III]*** must be completed to document any problems, discrepancies, project changes, etc. encountered during the process. This includes but is not limited to incorrect sample containers, improper preservatives [chemical and temperature], chain of custody discrepancies, sample descriptions, etc. A CAR may be completed just to keep a record of a situation, which is not actually "out of compliance."

1. Make sure that all information on the CAR is stated clearly and very detailed. Many times it is necessary to refer to these documents a year or more after they were completed. Document all correspondence including name, date, company and response.

2. The CAR forms must be numbered starting with No. 001 at the beginning of the year (e.g. 01-001). No two forms should have the same number. All CARs must be forwarded to the Project Manager and/or receiving manager for approval and distribution. THIS MUST BE DONE ASAP OF WHEN THE PROBLEM/SITUATION IS DISCOVERED.

C. Visually inspect all coolers for tampering, custody seals present and intact (if applicable) leakage, etc. If a cooler has been damaged beyond repair, unpack the samples and discard the cooler as to not reuse it. If you suspect a cooler may be damaged or is extremely dirty this cooler must not be reused. If coolers were sent by Federal Express, examine the Federal Express airbills for the number of packages in the shipment and make sure that all the packages (coolers, boxes etc.) in a group have been received. If there are any problems the Project Manager must be contacted immediately. If anything looks unusual, take the time to check it out and document the situation and findings.

D. Open each cooler in order to quickly inspect the contents and to locate the chain of custody. Sample Receiving personnel should wear the following personal protection equipment: gloves, safety glasses and a laboratory coat. All coolers received from projects with the **US Army Corps of Engineering (USACE) and AFCEE** projects should be opened under the hood in the sample storage room. Sign, and list the date and time received on the chain of custody. The time received <u>must</u> reflect the actual time the samples were received even though they may be logged into the system at a later time. Samples received on Saturday may be processed on the following Monday morning, or samples received late in the day during the week may be processed the next morning. All cooler(s) <u>must</u> be opened, examined for leakage, breakage etc., the temperature measured and the chain of custody signed and dated to

reflect the actual date and time which they were received. The samples must be delivered to the appropriate analytical department or put in cold storage as soon as possible.

1. Attach any shipping receipts, work orders, etc. to the chain of custody.
2. If a chain of custody or other paperwork is not sent, the client must be contacted and the samples temporarily placed on hold in cold storage. In some instances the log-in person may complete a chain of custody. The required information may be found on the sample containers or it may be necessary to call the client to get the missing information (i.e. sample ID, collection date and time, etc.). Note on the chain of custody that it was completed by laboratory personnel and record the name of the person with whom you spoke. All attempts to encourage our customers to complete a chain of custody or submit written information for samples must be made.
3. Project specific paperwork may be required. For all projects, a ***Cooler Receipt Form [Attachment IV]*** must be completed for each cooler received. Sample receiving personnel must begin completing this form as soon as a cooler is received and complete this form as samples go through the log in process.

E. The temperature of each cooler or set of samples must be measured as quickly as possible using a thermometer with 0.1°C increments. This thermometer must be calibrated against a NIST certified thermometer once a year and this information recorded in a bound notebook. The Certificate of Calibration for the NIST thermometer is kept on file at the QAO's desk. The thermometer must be tagged with the unique identification number of SR#1 and serial #; (Sample Receiving #1), the date calibrated and the correction factor. This information must also be recorded in a bound notebook. Only this thermometer can be used for recording the temperature of sample coolers upon receipt.

1. To measure the temperature, open the temperature control blank if supplied, point the IR thermometer at the liquid surface, wait 30 seconds for temperature to stabilize. Read the temperature to the nearest 0.1 °C. The corrected value temperature must also be recorded on the chain of custody. (This value will also be recorded into the LIMS at a later point.). All regulatory compliance samples received from North Carolina that do not meet the temperature requirement will be segregated and the client will be notified of the non-compliance. The samples will not be analyzed until we receive client notification to proceed with analyses.
2. If the temperature exceeds 6°C for any sample, the Project Manager or Sample Receiving personnel must contact the client immediately. There may be tighter temperature control limits for specific project requirements. The customer must make the decision to either continue with the analyses or resample. Make sure the client is aware that if the samples are analyzed, the following qualifier is normally included on the final report: "The shipping cooler temperature exceeded 6°C upon receipt to Empirical Laboratories. This may have an impact on the analytical results. The concentration may be considered as

estimated." Not all samples for the project will be flagged, just those samples received above 6°C.

Many times we are not able to get in touch with the client quickly and the best judgment on how to handle the samples must be made after discussion with the Testing Coordinator and/or Laboratory Director or Technical Director. The samples may still need to go through the log in process although it may be eventually determined that the samples will not be analyzed or the samples may temporarily be placed on hold and not logged in. Above all do not allow the samples to set out at room temperature for an extended period of time while waiting for a decision. **A CAR outlining the problem and all correspondence must be completed.**

**The only exceptions to the 6°C rule are:**

a. Water samples for all Metals, (except Chrome 6+ and mercury) that have been preserved with $HNO_3$ to a pH of $\leq 2$. *Keep in mind that non-aqueous sample for Metals must be cooled.*

b. Samples for Fluoride, Chloride and Bromide.

c. Waste/Product samples for all parameters.

d. Samples generated in the Aquatic Toxicology laboratories and brought directly to Sample Receiving after they are collected. Sample receiving personnel should place these in cold storage as soon as possible.

e. Samples collected locally by Empirical Laboratories personnel or local customers that hand deliver their samples. In some instances these samples may not have had time to cool down, however, these samples should have been placed on ice in an attempt to cool them to the proper temperature. It should be noted if samples are "Received On Ice" (ROI).

f. Samples that are received on ice and it is evident that the client made a good faith attempt to properly cool the samples.

F. If several coolers are received at once, they must be inspected to determine the order in which the samples should be unpacked and logged in. The following priorities should be given:

1. Any analyses, which have a 24-72 hour holding, time. It is the log-in person's responsibility to notify the department manager or section group leader of such samples via e-mail and verbally. In addition to notifying the analyst of samples with short holding times, log-in personnel must log this information into a separate record book daily. It is the analysts' responsibility to review this information and initial each page at the end of each day.

2. Any sample which has almost exceeded its' holding time. (Especially watch for this with waters organic extractions, Solids and Sulfides, all of which have only 7 days). A list of parameters and holding times is posted in the log-in room.

   a. If a sample is received already out of holding time, the project manager must be contacted. The sample can be analyzed at the client's request, but it will be qualified on the final report as being analyzed out of holding time. The project manager must inform you of the client's need.

   b. If a sample is received with limited holding time remaining for any parameter it may be necessary to contact the project manager so that he/she can contact the client. If the sample has to be analyzed on a rush basis to meet the holding time a rush charge may apply. Also it may not be possible to analyze the sample within the holding time due to sample load, etc. A CAR must be completed.

3. Samples requiring rush turnaround.

   a. If sample(s) require 24-hour turnaround they will take first priority. Other rush requests also have high priority.

   b. The Project Manager and/or Section Manager must be contacted for approval concerning any unscheduled rush requests.

G. Unpack all samples from the cooler. If there are any known or suspected hazards this must be done under a hood. All coolers from USACE projects should be unpacked under a hood. It may be necessary to rinse off the outside of the containers in the sink and/or wipe them off with a paper towel.

1. Visually inspect them for tampering and custody seals (if applicable). Sort and inventory the samples against the chain of custody by arranging them in the same order as they are listed on the chain of custody. Normally samples are assigned log numbers in the same order as they are listed on the chain of custody but for certain projects or situations it is acceptable to arrange them in a manner which will make them easiest to log in.

2. Check for leakage as this could compromise the sample integrity. If any spillage occurred in the cooler make sure this is noted. Also list all the other samples in the cooler as cross contamination could occur. A CAR must be completed and the Project Manager and/or the customer may need to be notified in these situations. It may be necessary to resample.

H. Check the chain of custody information against the information recorded on the containers. If these do not agree, contact appropriate person (s) - Project Manager, sampler, client, etc. All problems must be documented with a CAR.

1. If major changes are made on the chain of custody received from an engineering job, then the PE should submit written confirmation of these changes or make the corrections and initial them directly on the chain of custody.

2. Any error found on the chain of custody must be marked through with one line, initialed and dated and the correction written in.

I. Note any unusual requests, methodology, hazards (known or suspected) to the Project Manager and/or Laboratory Section Manager or analysts before the samples are actually logged in. Keep notes of any problems (improper containers, preservatives, temperature, or descriptions, etc.) A CAR must be completed and the analyst or manager should be notified immediately. If ever in doubt, fill one out!

## II. Sample Log In

A. After samples have been unpacked, sorted and reviewed, they are then ready to be assigned log numbers and continue through the log in process. Make sure that the parameters for the samples are clearly marked on the chain of custody. If we prepared the sample kits there should be a sample kit work order form. Contact the Project Manager if there are any questions, problems, etc.

B. Assign a work-order and sample number to each individual sample and record it on each sample container and the chain of custody.

1. All containers with the same description must have the same sample number even if they have different preservatives and require different tests. However, each different fraction (bottle type and/or preservative) should be designated with a letter (A, B, C, etc.)

2. Grab and composite samples from the same sample location must be considered as separate samples. It may be necessary to use "grab" or "composite" as part of the sample description to distinguish between the samples. Only assign different log numbers to them if the parameters are clearly marked as grab and as composite. Do not assume that VOC must be analyzed from grab samples so therefore the client must have taken a grab sample.

3. Sample numbers must begin with 001 at the beginning of each year (e.g. 0101001).

C. Check the following items and record this information on the cooler receipt form to further ensure sample integrity. A CAR must be completed if any of the following requirements are not met and it may be necessary to contact the client. We can perform the analyses in most cases and will do so with the client's approval, however the results may be qualified in some manner on the final report.

Preserving sample integrity throughout the log in procedure must be one of our section's top priorities. This includes not only ensuring that the proper chemical preservatives have been added but also that the samples are received and maintained at the proper temperature. ***When samples are unpacked they must be placed in cold storage as soon as possible even if they have not been through the entire log in procedure.*** All samples for NPDES compliance monitoring from North Carolina will be stored at a temperature range of 1.0 to 4.4°C. All other NPDES samples will be stored at 4.0 ± 2.0°C. On the days we receive a large volume of samples, or are short handed, etc., we may not be able to completely log in all samples until late in the day or even the next day. Samples should not set out at room temperature if there is a delay. The samples must temporarily be placed in cold storage until you are able to complete the log in procedure. This should also be done when we take lunch breaks.

[Make sure the VOC containers are not temporarily stored in a non designated VOC only storage area.]

1. Determine if the samples were received at the proper temperature. (See section IC)

2. The sample descriptions on the bottle should match those on the chain of custody. (See section 1H)

3. Check to determine if the proper chemical preservatives were added to adjust the sample to the correct pH. All regulatory compliance samples received from North Carolina that do not meet the preservation requirement will be segregated and the client will be notified of non-compliance. The samples will not be analyzed until notification to proceed with analyses is received from the client. A list of parameters and the required chemical preservatives is posted in the log-in room. The verification of this preservation will be recorded on the Cooler Receipt Form for all projects. If Empirical Laboratories prepared and shipped out the sample containers they will have been pre-preserved unless instructed otherwise by the client. Complete traceability of the preservatives used to pre-preserve the sample containers and to preserve samples in the log-in area is required. A bound notebook must be used to trace this information and must include the following: Lot #, Type of preservative, Date Prepped, Amount and Analyst Name. This information must also be labeled on each container, re-pipetter, etc. that the preservative is stored in. Each lot of HNO3 used for Metals preservation must be tested prior to using them for preservation. These analyses are kept on file.

   a. The pH of each container (except VOA vials) which requires pH preservation must be checked. Do not open and check the pH of VOA vials in sample receiving/log-in.

   b. The pH of preserved samples is checked and confirmed using pH narrow range indicator paper. When the client request pH analysis on samples and they must be reported and measured for pH using the narrow range paper, rather then a pH meter, the accuracy of each batch of indicator paper must be calibrated to the nearest tenth versus certified pH buffer and recorded into a bound logbook in accordance with SW846 method 9041A pH Paper method.

c. When taking the pH reading, DO NOT PUT THE pH PAPER DIRECTLY INTO THE SAMPLE CONTAINER. Pour up a small aliquot and dispose of this volume after the pH is taken. For some samples (wastes) the indicator paper may not be accurate due to interferences. The observation of the appropriate color change is a strong indication that no interferences have occurred. If it appears as if there is interference, the pH must be measured using the pH meter. [See SOP ATSD-187 pH , Electrometric.]

4. The following guidelines must be followed to check pH preservation:

a. Water samples for Cyanide analyses must be preserved to a pH of >12 with NaOH upon collection. If the pH of these samples is between 11.0-12.0 upon receipt, and the samples are at the proper temperature and not over 48 hours old it will not be necessary to complete a CAR, however the sample should be adjusted to ≥12.0 unless project/client specific requirements are to contact the client first.

b. Water samples for Metals analyses must be preserved to a pH of ≤2.0 with $HNO_3$ upon collection. If the pH of these samples is between 2.0-.3.0 upon receipt, and the samples are not over 48 hours old it will not be necessary to complete a CAR, however the sample should be adjusted to ≤2.0. unless project/client specific requirements are to contact the client first.

c. Samples requiring analyses which are preserved with $H_2SO_4$ (i.e., Nitrogen compounds, Total Phenolics, Oil and Grease, Total Phosphorus, etc.) can be accepted up to a pH of 2.5 without a CAR, however the sample should be adjusted ≤2.0 unless project/client specific requirements are to contact the client first. Samples for sulfide analysis must have a pH >9.

d. If a sample is not properly preserved, log-in personnel must either do the following:

- To meet project specific requirements, including all USACE projects, the client must be notified before preserving or adding additional preservative to the sample unless otherwise instructed. If the client instructs us to add chemical preservatives to a sample, complete traceability of the preservatives used is required (See section IIC, #3). A CAR must be completed.
- For other projects it may be acceptable to preserve the sample accordingly before the sample is placed in storage. Complete traceability of the preservatives used is required (See section IIC, #3). A CAR outlining the project and the steps taken must be completed.
- All metals samples preserved upon receipt must be held 24 hours before proceeding with analysis. These samples must be entered into the Unpreserved Metals Log and a CAR generated and the client notified to see if the lab is to proceed with analysis.

e. In some instances it may not be possible to adjust the sample to the proper pH due to matrix problems which cause excessive foaming or require an unusually large amount of acid. Do not continue to add acid if a few mL's of acid does not lower the pH. Notify the Project Manager, Metals Manager and/or analyst. They will make the decision if the sample will be diluted, not analyzed, etc. A CAR must be completed in these situations. Make sure you note on the container and in the LIMS notes that the sample is not at the proper pH as well as any useful information (i.e., foaming, strong odor, etc.).

f. A CAR may not be required for samples generated in the Aquatic Toxicology Laboratories and brought directly to Sample Receiving after they are collected but before they are preserved. Log-in personnel must preserve the samples accordingly before they are placed in storage. Complete traceability of the preservatives used is required (See section IIC, #3). A CAR outlining the project and the steps taken must be completed.

5. Check to make sure samples are in proper containers and that there is adequate volume for all the parameters requested and no leakage.

6. If VOA vials are present, each vial must be inverted and checked for head space. "Pea-sized" bubbles (i.e. bubbles not exceeding 1/4 inch or 6 mm in diameter) are acceptable and should be noted, however, a CAR is not required. Large bubbles or head space is not acceptable and a CAR must be completed. If this occurs, the client must be contacted. The samples can be analyzed with their approval, however the report will be qualified and the data may be questionable. All VOA vials will be preserved with $Na_2S_2O_3$ (0.2g) when chlorine is known to be, or suspected to be present.

7. All pesticide samples to be analyzed by method 608 will be checked by the sample receiving personnel for the correct pH range of 5.0 to 9.0. The pH of the sample(s) will be communicated via E-mail to the Section Manager and appropriate analyst.

8. All chlorinated effluent samples received for Cyanide must be checked for residual chlorine. The one liter sample container should initially contain 1 to 2g/L of Ascorbic Acid. Potassium Iodide starch indicator paper will be used for detecting the presence of residual chlorine. DO NOT PUT THE TEST PAPER DIRECTLY INTO THE SAMPLE CONTAINER. Pour up a small aliquot, neutralize, test and dispose of this volume after the sample is checked. If the test paper turns blue, the sample must be treated for residual chlorine. Add Ascorbic Acid, approximately 0.6g at a time and recheck the sample until there is no residual chlorine present. If the sample required this treatment this information must be included in the LIMS notes. This must be done by log-in personnel before leaving the receiving area. It may be necessary to notify the Inorganic Manager and/or analyst.

9. Be aware of holding time requirements. (See section 1D)

D. Once sample containers have been numbered, they must be checked by another laboratory individual to ensure that the log number on the container matches the log number and sample ID on the Chain of Custody. A ***Sample Receiving Custody and Disposal Form [Attachment VII]*** must be completed each day. Samples should not leave the log-in area until this has been completed. *[see IIC; it may be necessary to temporarily store samples in cold storage until the samples can be second checked, the amount of time that the samples are at room temperature must be minimized as much as possible.]* The original is to remain in Sample Receiving until the samples are disposed of. Once the document is complete, the original will be kept on file. The following information must be logged onto this form:

1. Client and Log #s
2. Date/Time Unpacked
3. Logged In/Numbered By (Initials)
4. 2nd Checked By (Initials)
5. Date/Time Placed in Cold Storage
6. Storage Area (Walk In, VOC Cooler, Quarantined Soils, Quarantined-VOC, Other)
7. Disposed of By/Date
8. Method of Disposal

E. Notify the proper analyst if samples have been logged in for analyses which have a 24-48 hour holding time or if a 1-2 day turnaround has been requested. The log number and description on sample (s) must be second checked before it is released to the analyst. (The analyst can second check the sample, but must initial the custody form.)

## III. Sample Storage

A. After samples have been correctly logged in they are then transferred to one of the following cold storage areas and arranged in numerical order by the assigned log in/LIMS sample number. ***Note that aqueous VOC samples must be segregated from all other samples.***

1. The Blue Air refrigerator in Sample Storage Room: All aqueous VOC's must be stored in this refrigerator. Storage blanks consisting of organic free water from the laboratory may be required for specific projects. These will be analyzed for VOCs only. ***Storage blanks are required for all DOD projects.***
2. Walk In Refrigerator in Sample Storage Room: All aqueous samples for all analyses must be stored in this refrigerator.

3. Soil Walk-In Refrigerator in back BC Laboratory: All quarantined and non-quarantined soil samples for all analyses must be stored in this refrigerator.

B. Quarantined soils are those quarantined by the US Department of Agriculture. These soil samples must be segregated from other soil samples during storage. A separate disposal log must be maintained for these soils including the location, date and quantity of the soil received and processed. Soil residues from quarantined samples must be treated according to regulations after testing (see Sample Disposal SOP). Quarantined soils are defined as:

1. Soil taken from much of the southeastern US and parts of New York and Maryland at a depth of three feet or less. *Soils from three feet or more are not regulated provided they are stored separately.* A map of the regulated areas in the United States entitled ***Soil Movement Regulations [Attachment VIII]*** is posted in the log-in room.

2. All soils taken from foreign sources, US. territories and Hawaii .

***NOTE: All soils are treated as quarantined soils and are disposed of in accordance with USDA regulations. Above for information purposes only.***

C. All samples must be stored in one of the three refrigerators detailed above with the following exceptions:

1. Matrices that may be adversely affected by the cold temperature. (e.g. surfactant samples, multi-phase samples)

2. Highly contaminated waste or product type samples that could jeopardize the integrity of other samples in the walk in cooler. Often these can be stored at room temperature. If these require refrigeration see the Project Manager for other options.

D. The temperature of each sample refrigerator must be monitored and recorded each day by Wet Chem personnel by the following method. A Mercury thermometer or digital min/max thermometer with 1° increments must be used. Each thermometer must be calibrated against a NIST certified thermometer once a year**(digital thermometers quarterly)** and this information recorded in a bound notebook. The Certificate of Calibration for the NIST thermometer is kept on file at the QAO's desk. The thermometers must be tagged with a unique identification, the date calibrated and the correction factor.

The tolerance range for all refrigerators is 1 to 6°C. This range and the range using the corrected reading must be posted on the outside of each cooler. If the temperature exceeds this range, corrective action measures must be put in place immediately. A CAR must be completed specifically noting the date and time the problem was discovered. The Project Manager, Laboratory Director and Technical Director will be notified in order to assess the situation. It may be necessary to put a service call in to the refrigeration repair service.

E. All personnel removing samples from any refrigerator must sign them in and out. This is done by completing the ***Sample Custody Form [Attachment IX]*** which is attached to the door of each refrigerator. These completed forms are kept on file [see section III, #4F]

F. The water walk in refrigerator in the sample room is the largest refrigerator and stores a large majority of the samples. A back up compressor is hooked into the system and scheduled to automatically come on if the main compressor fails. There is a digital min/max thermometer, which monitors the temperature 7 days a week. This thermometer will be calibrated quarterly against the NIST thermometer.

G. As stated above the temperatures for all refrigerators that samples are stored are checked each day Monday-Friday and monitored seven days a week with min/max thermometers. Pay close attention to these readings and watch for signs of possible problems.

H. A temperature maintenance record book is kept for each refrigerator.

I. Samples must be held for a minimum of 30 days after the final report unless specified otherwise. For USACE projects, samples must be held for a minimum of 60 days after the final report unless otherwise specified. See SOP ATSD 405 entitled Analytical Laboratory Waste Disposal SOP for guidance on disposal of samples.

## IV. Laboratory Information Management System (LIMS)

A. Log the sample information into the LIMS for each sample. Every attempt should be made to get every sample logged into the LIMS by the end of the day. All information entered should be clearly stated and recorded on the COC provided. After opening the main menu of the LIMS, select the 'Work Orders' tab from the 'Sample Control' drop down menu. Now click on the 'Add' button to create a new Work Order. You will see the following:

1. ***Client*:**

   Select the client I.D. by clicking on the pull-down and choosing from the client list. This list is in alphabetical order. If the desired client is not on the list, a new client must be created by the project manager or I.T. director.

2. ***Projects*:**

   Click on 'Projects' and choose the project I.D. The projects will be client specific. After the project is chosen the "project information" areas should fill in. The 'Project Name,' 'Project Number,' 'TAT,' 'Client Project Manager,' 'Lab Project Manager,' and 'Comments' information should also appear. If there are no applicable project choices, a project must be created by the project manager or I.T. director. There are two types of projects:

a. Internal -- Empirical Laboratories projects;

b. External -- direct laboratory clients.

3. ***Comments:***

This area is to be used to note any information from the project manager for all work orders of this project. It can also be used to list any work order specific notes; this includes but is not limited to information concerning rush turnaround, deliverables or other QC requirements, analyte concentrations, safety issues, quarantined soils, CAR #s, preservation or matrix problems, etc.

4. ***Received By:***

Enter the name of the person who received the samples.

5. ***Logged In By:***

Enter the name of the person who logged in the samples.

6. ***Received:***

Enter the date and time received separated by a space and using military time. Example: *08/02/2008 08:30*

7. ***Project/Package Date Due:***

After the date and time received have been entered, the date due for both of these fields will be calculated. If this information is not correct or needs to be amended later, check with the project manager before doing so.

8. ***Shipping Containers:***

Click on the 'Coolers' button and enter the temperature and condition upon receipt. If more than one cooler was received, each cooler must be assigned a different name. For example, if these came in by dedicated courier, enter the last four numbers of the Tracking Number as the name. After all of a cooler's information has been entered, click the 'Save' button. If more than one cooler was received, click the 'Add' button and repeat the process above, then click 'Done' after all the coolers' info has been saved.

9. ***COC Number:***

If an identifiable COC number is listed, record that ID here.

10. ***Shipped By:***

Enter the courier used to deliver the samples. If the samples were picked up by a lab employee or dropped of by the client/representative, enter 'Hand-Delivered.'

***After these items have been completed, click 'Save,' then the 'Samples' button to continue. To begin entering information for a sample, click the 'Add' button on the bottom of the Samples screen.***

11. ***Sample Name:***

a. Only abbreviate if description is too long for the spaces allotted in the LIMS. This information should come directly from the chain of custody. The sample ID entered into the LIMS will be the sample ID on the final report.

b. If no sample ID is provided, or is indistinguishable from other samples listed, contact the project manager to ascertain distinction in the samples. Include date as part of the description if this is the only way to differentiate the samples.

c. When logging in trip blanks that do not have an ID assigned by the client, list them as "Trip Blank # _____". This information should be on the containers. A log book must be kept in the sample kit room which lists all trip blanks and the date they were filled. This will ensure consistency with the descriptions for trip blanks. Make sure you record the trip blank on the chain of custody if it is not listed.

12. ***Collection Date:***

Enter the date and time the sample was collected. You must use military time and separate by a space. Often the time collected is not given. Although this is a sampling requirement, this information may not be crucial unless a parameter with a short holding time or a data deliverables package is required. All attempts should be made to get all our clients to supply this information.

13. ***Lab/Report Matrix:***

Click on pull down and select matrix. Many times it is difficult to discern the matrix if it is not specified on the COC, and log-in personnel must use their best judgment with regard to analytes/methods requested. Keep in mind that the detection limits and units on the LIMS reports are linked to the matrix. In some cases it may be necessary to ask the Section Managers about the matrix selection.

14. ***Sample Type:***

This is used to differentiate between special types of samples (i.e. Field Duplicates, Equipment Blanks, Trip Blanks, etc.). If there is no definite way to determine that a sample should be classified as something else, then "SAMP-Client Sample" will be selected as the sample type. Do not list a sample as anything other than a Client Sample unless noted on the COC of are instructed by the client to do so.

15. ***Container:***

Click on the drop down list and select the appropriate bottle type. If multiple bottles are received for the same sample, then move down to the next line and select all other containers as required. Repeat this process until all containers for the sample are listed. As each container is entered, an individual number is assigned to it by the LIMS system. This number is also listed on the container labels that are printed from the LIMS, and is placed on the corresponding bottle for container tracking purposes.

16. ***Fraction:***

When more than one container is sent with the same preservation/analysis (i.e. Volatiles & Extractables), the sample containers can be grouped together using a fraction code. This is simply an alphabetical notation added to each container to allow analysts to verify the preservative and proper sample volume to use when performing analysis. **This should be in no way used to record the sample volume used during analysis or reporting**, as these codes are entered by log-in personnel on an as needed basis, and do not provide an individual container designation by which to track any given container.

17. ***pH*** (Container Preservative)***:***

Use this to document the pH check information taken during sample unpacking. If no preservative was used, then nothing is required in this field.

18. ***Comments:***

Enter any information that is applicable at the sample level.

19. ***Field Analysis:***

Click on field analysis tab and enter field information when provided.

20. ***Work Analyses:***

Select all parameters requested for the sample from this list.

a. If the required test code is not listed, and the sample matrix is not a contributing factor, click the Work Analyses tab to open the All Analyses list. When selecting from this expanded list, be careful to select the proper method as all methods available for the current matrix will be selectable.

b. If any analyses are selected from the All Analyses list, the Project Manager in charge should be notified so that the correctness of methods and pricing can be checked and updated as needed.

c. All preparation codes for analytes are entered and stored by the system independently of the test codes selected, except in the cases of Dry Weight analysis, and TCLP/SPLP preparation (tumbling). In the case of the TCLP/SPLP prep codes, these are entered alongside the other required analyses automatically by the LIMS when a TCLP/SPLP analyte is selected. As for Dry Weight, it is required for all solids testing except in the cases of TCLP/SPLP analysis, Explosives only analysis, and/or any pure product/non-soil based sample when specified by the client.

21. ***Analyses Comments:***

These comments should be used for any notes that only apply to that particular test code.

22. ***RTAT:***

If the Rush Turn-Around Time for this sample is known at the time of log-in, this information should be updated here.

23. ***Save:***

Once all applicable information is entered for a sample, click the save button. At this time the LIMS applies the Laboratory Sample ID to the sample. This is a four part ID code composed of the following:

a. A 2-digit numeral of the year. Example (**08**11248-06)

b. A 2-digit numeral of the month. Example (08**11**248-06)

c. A 3-digit numeral of the work order number. This number reset to 001 at the beginning of each month. Example (0811**248**-06)

d. A 2-digit numeral of the sample number separated by a dash. Example (0811248-**06**). This number is different for each sample in a work order, and a single work order cannot contain more than 99 samples. If more sample numbers are needed, a new work order number will have to be assigned to the applicable set of sample.

24. ***Add/Edit/Copy:***

Use these selections to add more samples to the work order, or to change existing information prior to label printing.

***Once all the tests have been selected and all samples have been added in the work order, a work order summary and all container labels are printed. Labels are checked for accuracy against the containers while being labeled. At this point log-in of this group of samples is complete.***

B. After log-in of a work order is complete, the COC can then be scanned into the system, attached to the work order on the Work Order screen, and the work order can be updated to Available status so as to be seen by the analysts.

## V. Daily Follow Up for Sample Receiving/Log In

A. Wipe out the inside of coolers and return all Empirical Laboratories coolers to the sample kit room. Discard any coolers that are cracked, broken or filthy.

B. Complete the short holding times log book as required. This must be done as early in the day as possible.

C. If any samples were received for RUSH turnaround, then a ***RUSH SHEET [Attachment XII]*** must be completed and distributed to all laboratory personnel via e-mail. If ever in doubt of which analysts should be notified, pass them out to everyone. Always give copies to the Laboratory Director, Administrative Assistant and Section Managers. It may be necessary to send out two RUSH sheets per day (one around mid-day and the other at the end of the day).

D. Complete any required CARs.

E. At the end of the day organize all paperwork received and generated for the day. The following should be given to the Project Manager (section supervisor):

1. The original chains of custody and yellow original or copy of each. The CRF will accompany the CoC for the project.

2. Any information (letters, regulatory limits, etc.) from a client which was received with any samples.

3. All CARs.

4. LIMS sample receiving logs.

5. Copies of any RUSH sheets which have been distributed

6. Sample Receiving Custody and Disposal Form.

7. Cooler receipt form.

G. All the above information from the day will be reviewed as soon as possible.

1. All LIMS logs must be 2nd checked by a different person than the person entering the information into the LIMS. Each set of logs must be initialed dated by the person 2nd checking. These will be kept on file at the Project Manager desk.

2. If any corrections or changes are required, all laboratory personnel will be notified by distributing a ***Sample Log Change Form [Attachment XIII]*** through email distribution. A ***Sample Log Change Form*** by the project manager will also be sent out if a client adds or deletes any parameters, changes sample IDs, etc.

H. The Testing Coordinator will distribute the following after they have been through the 2nd QA check:

1. Copies of the LIMS receiving reports to necessary laboratory personnel.

2. Original (white copy) chains of custody are given to the Project Manager. These will be sent with the final report to the client.

3. Finalized/approved CARs must be sent to the:

a. Organic Manager

b. Inorganic Manager

c. Laboratory Manager

d. Laboratory Director {optional}

e. Quality Assurance Officer

f. Administrative Assistant

g. Client {optional}

4. Copies of any project/sample specific information to the Section Manager and analysts.

I. Information will be filed as follows:

1. Chains of custody:

   a. Original (white copy) is returned to the customer with the final report along with the CRF.

   b. Yellow copy originals or photo copies will be filed by client and kept in the Sample Receiving Room.

   c. Photo copies will be kept in a notebook in numerical order in the Sample Receiving Room.

   d. Pink copies should be retained by the sampler.

2. CARs

   a. CARs can be found at V:\LAB\log-in\login(year)\logcar(year).

3. Sample Change Forms and RUSH Sheets

   a. Sample Change Forms are distributed by email.

   b. RUSH Sheets are found at V:\LAB\login\Rushsheets

4. At the end of each year, files for that year are boxed and archived. Make sure files are labeled properly and place them in banker's boxes. Complete a storage box file form with as much detailed information as possible. The Laboratory Administrative Assistant will label and number the boxes and incorporate the storage boxes into the laboratory file archive system. Boxes containing files from Sample Receiving are kept on site for 1-2 years and then may be moved to off site storage upon release from the Project Manager.

## VI. Miscellaneous

A. All projects which require deliverables or other QC requirements should be listed in the notes section of the LIMS.

B. If samples are received from a new client or a new job number that is not in the LIMS, a new client code must be set up. This information should be on the chain of custody or it may be necessary to contact the customer if the information is incomplete.

C. Samples from the Aquatic Toxicity Laboratory (ATL) are logged into the LIMS for billing and long-term tracking purposes. The receiving information and proper assignment of test's are reviewed by the ATL Manager. The samples are then logged in by ATL personnel.

D. A flow chart outlining sample receiving and the flow of data, reporting and invoicing is attached as ***Attachment XIV.***

E. A ***Telephone Conversation Log [Attachment XV]*** may be required to document information and may be attached to or used as a CAR.

F. All log books used in the Sample Receiving and Sample Storage Areas are numbered. The following log books are presently maintained. All log books must be "Z"ed out. The Testing Coordinator will review the log books each week to check for completeness.

| Log Book ID | Log Book Description |
|---|---|
| SRLB #3 | pH Paper Calibration |
| SRLB #4 | Tracking of VOC Trip Blanks, Organic Free Water and Chemical Preservation |

## Attachments to SOP 404

| | |
|---|---|
| II | Chain of Custody Record |
| III | Corrective Action Report for Sample Receiving/Log In |
| IV | Cooler Receipt Form |
| V | List of Short Holding Time (Immediate-72 hrs.) Parameters |
| VII | Sample Receiving Custody and Disposal Form |
| VIII | Map of Quarantined Soil Areas in the US. |
| IX | Laboratory Sample Custody Form for Walk In Refrigerator |
| X | Container Codes for the LIMS |
| XI | Routine NPDES Clients |
| XII | RUSH Sheet |
| XIII | Sample Log Change Form (Green Sheet) |
| XIV | Flow Chart, Laboratory Sample Tracking System |

*[Attachments I and VI were removed during the editing process and not added to the SOP.]*

# ANALYTICAL LABORATORY WASTE DISPOSAL

**SOP NUMBER:** **SOP-405**

**REVISION NUMBER:** **4**

**APPROVED BY:**

**TECHNICAL DIRECTOR**

**QUALITY ASSURANCE OFFICER**

**EFFECTIVE DATE:** **09/26/03**

**DATE OF LAST REVIEW:** **04/23/08**

# Analytical Laboratory Waste Disposal
# Standard Operating Procedure

## I. SCOPE AND APPLICATION:

Empirical Laboratories, LLC laboratory waste includes excess client sample waste and waste that are generated while performing an array of analytical services, some of which are hazardous. These wastes must be disposed of in a manner that is safe, cost efficient and in accordance with hazardous waste regulations.

### A. Wastes can be broken down into the following categories:

1. Unused portions of actual samples received from outside clients.

   a. Unused aliquots of completed water samples.

   b. Unused aliquots of completed non-aqueous samples.

2. Soils from quarantined areas

3. All other soils, sediments, building debris, wipes etc.

4. Hazardous waste generated within the laboratory as part of numerous analytical procedures.

## II. SUMMARY OF PROCEDURES:

### A. There are four options for disposing of unused sample portions:

1. Return completed samples and any generated waste from these samples to the client.

2. Throw the sample away after confirming that it is non-hazardous.

3. Disposal through a waste vendor in either a sealed drum or lab pack. This is normally done twice a year.

4. Treat the sample to make it non-hazardous and dispose of it as such. (Aqueous pH neutralization only.)

**B. There are two options for disposing of laboratory generated waste:**

1. Disposal through a waste vendor in either a sealed drum or lab pack. This is normally done twice a year. The waste must be stored properly until the waste is transported off site.

**For example: Solvent waste must be stored in the vented flammable cabinet.**

2. Treat the waste to make it non-hazardous and dispose of it as such. (Aqueous pH neutralization only.)

## III. EQUIPMENT/APPARATUS:

**A. Proper safety equipment in good working condition. This includes gloves, lab coat and safety glasses/goggles (voluntary use of cartridge respirator allowed see area manager or QAO).**

**B. USDOT approved drums for storing and shipping hazardous waste.**

**C. Fume hood vented outside the building.**

**D. Flammable storage cabinet which is vented to the outside**

## IV. PROCEDURE

Waste disposal is done under the management and coordination of the Sample Receiving Manager, Section Managers and the Health and Safety Officer.

**A. Disposal of completed aqueous samples:**

Completed samples are kept in cold storage for approximately three weeks after the final report has been mailed. Engineering support projects involving CLP work, litigation cases etc. may be saved for longer than three weeks at the request of the project manager.

No samples should be disposed of without approval from the responsible area manager or analyst. **At this point the area manager and/or analyst will communicate information about samples deemed as hazardous.**

1. The majority of the water samples (ground, surface and drinking) are non-hazardous and are disposed of by pouring them down the sink.

a. This must be done under the hooded area located near the walk in cooler next to the extraction lab. Make sure that the sash is closed far enough to produce sufficient ventilation. The tap water should be turned on to supply copious wash for sample disposal.

b. Proper safety equipment **must** be used including safety glasses (face shield if necessary), lab coat and gloves.

c. **Be alert to potential problems: for example, separate Cyanide waste from acid waste. Neutralize acid waste that will be poured down the acid drain and don't mix waste/samples thought to contain Cyanide with samples that are acidified. Also, look for things such as phase separation, odd color, odor etc. Check with the area manager or Health and Safety Officer before disposing of any questionable samples.**

d. Tap water must be running during the time samples are poured out and for approximately 10 minutes after so sufficient flushing and dilution takes place.

e. All containers must be rinsed out, all identifying markings defaced or removed, and thrown into the trash.

f. All samples disposed of in this manner must be documented in the bound disposal log.

2. If water samples are hazardous (known or suspected), one of the following steps must be taken.

a. Samples may be returned to the client. If you plan to ship the unused portion back to the client check with shipping and receiving to make sure that the material can be shipped in accordance with USDOT regulations. **If the samples are not returned to the client they must be stored properly until picked up by a waste vender.**

b. Treat the sample to make it non-hazardous. One example of this is if the sample is highly corrosive, the pH may be adjusted.

c. Store the sample properly until either a sealed drum or lab pack is sent out.

d. All samples disposed of in this manner must be documented in the bound disposal log.

**B. Disposal of completed non-aqueous samples:**

The majority of non-aqueous samples are soils or sediments, although there may also be building debris, wipes, oils, and occasionally product type samples.

1. If samples are non-hazardous they must have all identifying markings defaced or removed, and thrown into the trash. On specific projects we may also opt to return the unused portions to the client even if they are non-hazardous.

2. If non-aqueous samples are hazardous (known or suspected), one of the following steps must be taken.

a. Samples may be returned to the client. If you plan to ship the unused portion back to the client check with shipping and receiving to make sure that the material can be shipped in accordance with USDOT regulations. **If the samples are not returned to the client they must be stored properly until picked up by a waste vender.**

b. Store the sample properly until a lab pack is sent out.

3. Soil samples taken at a depth of three feet or less from areas, which have been quarantined by the US Department of Agriculture (USDA), must first be treated at the laboratory to prevent the spread of any plant pests. The USDA has detailed proper treatment procedures of which we use the following:

a. The sample is heated to 180°C(356°F)in a vented oven for one hour.
b. After the heating the samples are placed close to a hood to cool and are marked as being ready for disposal.

4. Once the samples have undergone treatment they can then be disposed of by one of the procedures for non-aqueous samples. **All samples disposed of in this**

**manner must be documented in the bound disposal logbook with the following information:**

a. Client
b. Sample #s
c. Date(s) treated
d. Treatment method used

**C. Disposal of laboratory generated waste:**

Generated waste is stored in satellite areas until a waste pick up occurs. These satellite areas must be maintained properly.

1. Waste handling and disposal within each laboratory section:

Each laboratory analyst and section manager is responsible to assure that **handling** operations within their area are being followed according to the laboratory requirement.

a. General Chemistry/Inorganic

Each analyst performining specific laboratory tests that generates waste is responsible to handle and dispose of the waste in a safe manner and under the guidelines listed below. If you have any questions left unanswered regarding waste disposal within your specific area contact the inorganic manager or the safety officer.

- Concentrated acid waste, **(>2% by volume) and** dilute mercury waste (mercury, chemical oxygen demand, total kjeldahl nitrogen and chloride analyses waste) are poured into the Acid Satellite Waste drum in the general chemistry laboratory. **Document the type and amount of waste in the acid waste logbook, then initial and date the entry.**
- Dilute acid waste **(<2% by volume or less)** are neutralized and poured down an acid drain with copious amounts of tap water.
- **All other non-hazardous sample waste, reagents and standards are poured down the drain with copious amounts of tap water.**

b. Metals

Each analyst performining specific laboratory tests that generates waste is responsible to handle and dispose of the waste in a safe manner and under the guidelines listed below. If you have any questions left unanswered regarding waste disposal within your specific area contact the inorganic manager or the safety officer.

- Concentrated acid waste, aqueous sample waste digestates and old unused calibration standards **(>2% by volume)** are poured into the Acid Satellite Waste drum in the general chemistry laboratory.
- Non-aqueous sample digestate wastes are decanted off the soil/solid samples into the Acid Satellite Waste drum in the General Chemistry Laboratory. **Rinse the soil/solid with tap water several times and discard the first rinsate into the Acid Satellite Waste drum and the sequential rinsates decant down an acid drain with copious amounts of tap-water.**
- **Throw the soil/solids in the trash once the acid has been rinsed free.**
- **Cr6 digestates as with all concentrated metal/acid waste are poured into the Acid Satellite Waste drum.**

c. Organic Extraction Laboratory Area

Each analyst performining specific laboratory tests that generates waste is responsible to handle and dispose of the waste in a safe manner and under the guidelines listed below. If you have any questions left unanswered regarding waste disposal within your specific area contact the organic manager or the safety officer.

- Concentrated acid waste is discarded into the Acid Satellite Waste drum in the General Chemistry Laboratory.
- Non-chlorinated solvent waste (Acetone, Ether, Hexane, Methanol ....etc...) pour into the Non-Chlorinated Waste labeled bottle located in the hood in the Organic Extraction Laboratory.
- Chlorinated solvent waste (Methylene Chloride, Chloroform, chlorinated standard and spike waste) pour into the Chlorinated Waste labeled bottle located in the hood in the Organic Extraction Laboratory.

****Note: Laboratory generated solvent waste is transferred to the appropriate Satellite Solvent Waste Drum (chlorinated or non-chlorinated) weekly or as deemed necessary. Disposal of solvent waste is done under the direction of the organic laboratory manager. These drums are located in the chemical reagent storage room and only authorized laboratory staff are allowed to add waste solvent to these drums. The date of addition to the**

**drum , type and quantity of solvent is entered into the *'Organic Solvent Waste Logbook'* located on the shelf next to the drums.**

- **Aqueous sample waste from extracted samples (once the extraction solvent has been removed) is poured down the drain and flush with copious amount of tap water.**
- Non-aqueous sample waste and sodium sulfate waste is dumped into a waste container under an extraction laboratory hood and left overnight or until the solvent is evaporated and then the waste is discarded into the trash.

d. Gas Chromatography (GC)/High Performance Liquid Chromatography (HPLC) Laboratory

- Autosampler vials are discarded into the appropriately labeled box located under the table in the GC/HPLC Laboratory.

**PCB Box** – all samples/standards
**Pesticide Box** – all samples/standards
**Herbicide Box** – all samples/standards
**8330 Box** – all samples/standards
**Methylene Chloride Box**- all samples/standards that contain methylene chloride (Diesel Range Organics, DRO)

- Sample and spike extract vials are separated according to the contents in the vial.

**Acid cleaned extracts** are combined into a separatory funnel and the acid layer separated from the solvent. The acid portion is discarded into the Acid Satellite Waste drum in the general chemistry laboratory. The solvent waste is discarded into the appropriate solvent waste bottle (chlorinated/non-chlorinated waste) located in the hood in the organic extraction laboratory.

**After the mercury has been recovered** from the mercury cleaned samples, the extracts are discarded into the proper waste solvent bottle (chlorinated/non-chlorinated waste) in the hood in the organic extraction laboratory. The mercury waste is consolidated and discarded into the mercury waste container located in the vented hood in the chemical storage room. **Mercury used for removing sulfur from samples has been discontinued in our laboratory.**

**Unused stock and working standards** are discarded into the chlorinated solvent waste bottle located in the organic extraction laboratory. The empty vials are rinsed

several (3) times with solvent and the solvent rinsate poured into the solvent waste and the vials with labels removed are discarded into the glassware waste container.

e. Gas Chromatography/Mass Spectrometry

- Volatile sample, standard and reagent waste

**Waste from the instrument** - Aqueous sample waste is collected in waste bottles via waste lines from the instrument. The bottles are emptied into buckets and poured down the drain(pH is < 2% by volume). A small amount of methanol used to clean glassware is also dumped into the bucket and poured down the drain. While disposing of sample waste always run the cold tap water 10-15 minutes. Non-aqueous waste from sample analyses is retained and dispose of in the samey as the unused sample. Unused sample is held for sample disposal by the sample receiving area, see A and B listed above.

**Standards - Unused stock and working standards** are discarded into the chlorinated solvent waste bottle located in the organic extraction laboratory. The empty vials are rinsed several (3) times with solvent and the solvent rinsate poured into the solvent waste and the vials with labels removed are discarded into the glassware waste container.

In conjunction with section managers, the sample receiving area disposes of solid sample waste and unused aqueous and solid samples, see procedures A and B listed above.

- Semivolatile sample and standard waste disposal

Methylene chloride waste solvent and standard waste in vials are poured into the chlorinated waste bottle in the hood in the organic extraction laboratory. The empty vials are rinsed with solvent and the solvent poured into the waste solvent bottle. The vials with labels removed are discarded into the glassware waste disposal container.

Auto sampler vials are collected in buckets and stored under the hood in the organic extraction laboratory. **Periodically the vials are consolidated in lab packs for disposal by a licensed waste disposal company.**

f. Bioassay Laboratory

# STANDARD OPERATING PROCEDURE (SOP) FOR LABORATORY SAMPLE STORAGE, SECURE AREAS AND SAMPLE CUSTODY

**SOP NUMBER:** SOP-410

**REVISION NUMBER:** 6

**APPROVED BY:**

SECTION MANAGER

TECHNICAL DIRECTOR

**EFFECTIVE DATE:** 09/08/08

**DATE OF LAST REVIEW:** 09/08/08

- Aqueous sample waste and a small amount of methanol are poured down the drain with copious amounts of tap water. Larger amounts of methanol used for glassware cleaning are collected in beakers and evaporated at room temperature.

- Hazardous or product samples are returned to the client.

**D. Consolidation of satellite waste for contractor disposal:**

In conjunction with the Safety Officer, the sample revieving supervisor is responsible to coordinate waste disposal operations with outside waste disposal contractors.

1. Solvent waste from the areas discussed above is periodically consolidated into two drums located in the Chemical Storage room (c. *Organic Extraction Laboratory Area,* *** Note**). A drum designated either chlorinated or non-chlorinated solvent waste is available to receive the appropriate solvent waste. When the drums become full (fluid surface six inches below the top of the drum), an authorized hazardous waste contractor will be scheduled to remove them to proper waste disposal.

2. The Acid Satellite Waste drum is also disposed through the authorized hazardous waste contractor once the drum is full to the level of six inches below the top of the drum.

3. Consolidated autosampler and standard vials are periodically Lab-Packed in drums and disposed through the authorized hazardous waste contractor.

4. The Laboratory Health and Safety Officer will administer the Waste Disposal Program and maintain current information to track quantities of waste generated and stored on-site.

**It is the continuous objective of our laboratory to find ways to decrease the amount of waste generated.**

**STANDARD OPERATING PROCEDURE (SOP) FOR LABORATORY SAMPLE STORAGE, SECURE AREAS AND SAMPLE CUSTODY**

Empirical Laboratories, LLC is located on the fifth floor of a building which is locked and monitored by a guard after normal business hours. No unauthorized personnel are permitted within the facility without a proper escort and a visitor's badge. During non business hours, all doors to the building are locked and the elevators are security coded (i.e. a code must be entered in order to get the elevator to open on the fifth floor.) All stairway doors are locked and only Empirical Laboratories, LLC personnel have a key to the fifth floor stairway door. The doors to the lab in the hallway have a key code. There is a buzzer at the door to Login to allow entry for sample and supply deliveries.

The majority of samples are shipped in coolers by couriers such as Federal Express and UPS. All couriers are generally received in the Shipping/Sample Receiving (SR) area on the fifth floor. The laboratory is located close to Federal Express (FedEx) distribution station, therefore we pick up our coolers at the FedEx location on Saturdays and transport them directly to the laboratory. Some coolers and/or samples are delivered directly to the SR area by the sampler and/or client. The SR personnel must not leave any packages/cooler without authorized receipt from laboratory personnel. Samples must be accompanied by some type of chain of custody record. Sample receiving personnel sign, and list the date and time received on the chain of custody. The time received must reflect the actual time or validation date and time of receipt for the samples although they may be placed in cold storage and logged into the system at a later time. The method of delivery is listed on the cooler receipt form(CRF). The tracking # (if available) is attached to the chain of custody.

Once sample containers have been assigned a laboratory ID number, they must be checked by another laboratory individual to ensure that the log number on the container matches the log number and sample ID on the Chain of Custody. A Sample Receiving Custody and Disposal Form (attached) must be completed each day. Samples should not leave the log-in area until this has been completed. A copy of this form must be given to the Testing Coordinator at the end of the day. The original is to remain in Sample Receiving until the samples are disposed. Once the document is complete, the original will be kept on file. The following information must be logged onto this form:

- Client and Log #s
- Date/Time Unpacked
- Logged In/Numbered By (Initials)
- 2nd Checked By (Initials)
- Date/Time Placed in Cold Storage
- Storage Area (Walk In, Blue Air-VOCs, Quarantined Soils, Quarantined-VOC, Other)
- Disposed of By/Date
- Method of Disposal

Original samples are stored in following areas of the laboratory.

1. Blue Air Refrigerator in Sample Storage Room: All water VOCs must be stored in the refrigerator.
2. Walk In Refrigerator in Sample Storage Room: All waters for all analyses except VOCs must be stored in this refrigerator.
4. Soil Walkin Refrigerator for all soils.

All soils are treated as quarantined.

All samples must be stored in one of the three refrigerators detailed above with the following exceptions:

1. Matrices that may be adversely affected by the cold temperature. (e.g. surfactant samples, multi-phase samples)
2. Highly contaminated waste or product type samples which could jeopardize the integrity of other samples in the walk in cooler. Often these can be stored at room temperature. If these require refrigeration see the Testing Coordinator for other options.

Any person removing samples from the storage areas listed above, must sign them out on a laboratory custody sheet (attached). The individual performing the processing becomes responsible for the samples at this point. The samples are maintained in the

secure possession of the individual processing the samples. When the processing is completed, the samples are returned and signed back into the appropriate storage area. It must be noted if the entire sample volume was used and that the container was discarded.

Sample extracts and digestates are stored in the following areas:

1. All metals digestates are stored in the metals instrument laboratory. The transfer from the digestion analysts to the ICAP analysts is documented in the metals digestion log book.

2. Non - ZHE TCLP extracts are returned to the refrigerator in which the original samples are stored. For ZHE samples, the extract is returned to the refrigerator in which the original VOC sample containers are stored.

3. Extracts from medium level VOC analyses are also stored in the Soill Walk – in or VOC sample freezer in the VOC Lab.

4. All Organic extracts are stored in a Beverage Air side by side refrigerator in the organic extraction laboratory.

The generation of all sample extracts/digests and their movement through the laboratory will also be tracked on a laboratory custody sheet or in a log book. The individual performing the processing becomes responsible for the samples at this point. The samples are maintained in the secure possession of the individual processing the samples. When the processing is completed, the extracts are returned and signed back into the appropriate storage area. The metals digestates are not removed from the metals instrument laboratory.

After the analytical results have been reported, the original samples, sample extracts, and digestates will remain in secure storage until they are disposed of in accordance with the Waste Disposal Standard Operating Procedure. Samples will be held for a minimum of 30 days after the final report unless specified otherwise. Sample extracts and digestates are held for a minimum of 60 days after the final report unless project specific requirements state otherwise. See SOP No. 405 entitled Laboratory Waste Disposal SOP for guidance on disposal of samples.

The following personnel as of September 08, 2008 have access to all sample storage areas:

| | |
|---|---|
| Chandra Arthur | Herbie Johnson |
| Ashley Bester | Dahae Kim |
| Roger Burr | Dustin Lynch |
| Tanisha Custer | Marcia McGinnity |
| Rick Davis | Badeen Mekael |
| Barbara Dawson | AntonioMontiero |
| Betty DeVille | Ashley Morris |
| Amanda Fei | Gino Moore |
| Kendra Gentry | E. J. Overby |
| Jason Goodman | Brenton Powers |
| Sonya Gordon | Brian Richard |
| Gwen Hallquist | Franklin Rivers |
| Andrew Holder | William Schwab |
| Jade Holliman | Christy Thompson |
| John Hughes | Renee Vogel |
| Karu Huka | Randy Ward |

In the event that an employee is terminated, the supervisor is responsible for collecting the employee's keys.

For additional information see SOP No. 404 entitled Laboratory Sample Receiving, Log-In and Storage.

# APPENDIX C

# FIELD FORMS

Tetra Tech NUS, Inc.

# SOIL & SEDIMENT SAMPLE LOG SHEET

Page___ of___

Project Site Name: _____
Project No.: _____

[] Surface Soil
[] Subsurface Soil
[] Sediment
[] Other: _____
[] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____

Type of Sample:
[] Low Concentration
[] High Concentration

**GRAB SAMPLE DATA:**

| Date: | Depth Interval | Color | Description (Sand, Silt, Clay, Moisture, etc.) |
|---|---|---|---|
| Time: | | | |
| Method: | | | |
| Monitor Reading (ppm): | | | |

**COMPOSITE SAMPLE DATA:**

| Date: | Time | Depth Interval | Color | Description (Sand, Silt, Clay, Moisture, etc.) |
|---|---|---|---|---|
| | | | | |
| Method: | | | | |
| | | | | |
| Monitor Readings (Range in ppm): | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |

**SAMPLE COLLECTION INFORMATION:**

| Analysis | Container Requirements | Collected | Other |
|---|---|---|---|
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |

**OBSERVATIONS / NOTES:**

**MAP:**

**Circle if Applicable:**

| MS/MSD | Duplicate ID No.: |
|---|---|

**Signature(s):**

Tetra Tech NUS, Inc.

# EQUIPMENT CALIBRATION LOG

PROJECT NAME : _____ INSTRUMENT NAME/MODEL: _____

SITE NAME: _____ MANUFACTURER: _____

PROJECT No.: _____ SERIAL NUMBER: _____

| Date of Calibration | Instrument I.D. Number | Person Performing Calibration | Instrument Settings | | Instrument Readings | | Calibration Standard (Lot No.) | Remarks and Comments |
|---|---|---|---|---|---|---|---|---|
| | | | Pre-calibration | Post-calibration | Pre-calibration | Post-calibration | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |
| | | | | | | | | |

Tetra Tech NUS, Inc.

# BORING LOG

Page ___ of ___

PROJECT NAME: _____ BORING No.: _____
PROJECT NUMBER: _____ DATE: _____
DRILLING COMPANY: _____ GEOLOGIST: _____
DRILLING RIG: _____ DRILLER: _____

| Sample No. and Type or RQD | Depth (Ft.) or Run No. | Blows / 6" or RQD (%) | Sample Recovery / Sample Length | Lithology Change (Depth/Ft.) or Screened Interval | MATERIAL DESCRIPTION: Soil Density/ Consistency or Rock Hardness | Color | Material Classification | USCS* | Remarks | PID/FID Reading (ppm): Sample | Sample BZ | Borehole | Borehole BZ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | | | | | | | | |

* When rock coring, enter rock brokeness.

** Include monitor reading in 6 foot intervals @ borehole. Increase reading frequency if elevated reponse read.

Drilling Area Background (ppm): [ ]

Remarks: _____

Converted to Well: Yes _____ No _____ Well I.D. #: _____

TETRA TECH NUS, INC. CHAIN OF CUSTODY NUMBER 0584 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 GRAB (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

DISTRIB WHITE (ACCOMPANIES SAMPLE) YELLOW (F COPY) PINK (FILE COPY) 4/02R

FOR NO. TNNIS 001

Tetra Tech NUS, Inc.

# FIELD PORTABLE XRF SAMPLE ANALYSIS LOG SHEET

Project Site Name: _____ Measurement Units: _____

Project No: _____ Run Length/Duration _____

Analyzer: _____ Analysis Date: _____

| Sample ID | Analysis Time | Element Analysis | Result Run 1 | Result Run 2 | Result Run 3 | Results Avg. | Comments |
|---|---|---|---|---|---|---|---|
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |

Notes:

Tetra Tech NUS, Inc.

# QA SAMPLE LOG SHEET

Page___ of ___

Project Site Name: _____ Sample ID Number: _____
Project Number: _____ Sampled By: _____
Sample Location: _____ C.O.C. Number: _____
QA Sample Type:

[] Trip Blank [] Rinsate Blank
[] Source Water Blank [] Other Blank _____

## SAMPLING DATA:

Date: _____
Time: _____
Method: _____

## WATER SOURCE:

[] Laboratory Prepared [] Tap
[] Purchased [] Fire Hydrant
[] Other _____

## PURCHASED WATER INFORMATION (If Applicable as Source or Rinsate Water):

Product Name: _____
Supplier: _____
Manufacturer: _____
Order Number: _____
Lot Number: _____
Expiration Date: _____

## RINSATE INFORMATION (If Applicable):

Media Type: _____
Equipment Used: _____
Equipment Type:
[] Dedicated
[] Reusable

## SAMPLE COLLECTION INFORMATION:

| Analysis | Preservative | Container Requirements | Collected |
|---|---|---|---|
| Volatiles | Cool 4°C & HCl | | YES / NO |
| Semivolatiles | Cool 4°C | | YES / NO |
| Pesticide / PCB | Cool 4°C | | YES / NO |
| Metals | Cool 4°C & $HNO_3$ | | YES / NO |
| Cyanide | Cool 4°C & NaOH | | YES / NO |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |

## OBSERVATIONS / NOTES:

Signature(s):

# EQUIPMENT INSPECTION

COMPANY:_____ UNIT NO._____

REQUENCY: Inspect at the initiation of the project, after repairs, once every 10-day shift.

Inspection Date: ____/____/____ Time: _____ Equipment Type:_____
(e.g., bulldozer, generator)

| | Good | Need Repair | N/A |
|---|---|---|---|
| Tires or tracks | ☐ | ☐ | ☐ |
| Hoses and belts | ☐ | ☐ | ☐ |
| Cab, mirrors, safety glass | ☐ | ☐ | ☐ |
| - Turn signals, lights, brake lights, etc. (front/rear) for equipment approved for highway use? | ☐ | ☐ | ☐ |
| - Is the equipment equipped with audible back-up alarms and back-up lights? | ☐ | ☐ | ☐ |
| Horn and gauges | ☐ | ☐ | ☐ |
| Brake condition (dynamic, park, etc.) | ☐ | ☐ | ☐ |
| Fire extinguisher (Type/Rating -_____) | ☐ | ☐ | ☐ |
| Fluid Levels: | | | |
| - Engine oil | ☐ | ☐ | ☐ |
| - Transmission fluid | ☐ | ☐ | ☐ |
| - Brake fluid | ☐ | ☐ | ☐ |
| - Cooling system fluid | ☐ | ☐ | ☐ |
| - Windshield wipers | ☐ | ☐ | ☐ |
| Hydraulic oil | ☐ | ☐ | ☐ |
| Oil leak/lube | ☐ | ☐ | ☐ |
| Coupling devices and connectors | ☐ | ☐ | ☐ |
| Exhaust system | ☐ | ☐ | ☐ |
| Blade/boom/ripper condition | ☐ | ☐ | ☐ |
| Access-ways: Frame, hand holds, ladders, walkways (non-slip surfaces), guardrails? | ☐ | ☐ | ☐ |
| Power cable and/or hoist cable | ☐ | ☐ | ☐ |
| Steering (standard and emergency) | ☐ | ☐ | ☐ |

**Safety Guards:**

| | Yes | No |
|---|---|---|
| – Around rotating apparatus (belts, pulleys, sprockets, spindles, drums, flywheels, chains) all points of operations protected from accidental contact?_____ | ☐ | ☐ |
| – Hot pipes and surfaces exposed to accidental contact?_____ | ☐ | ☐ |
| – All emergency shut offs have been identified and communicated to the field crew?_____ | ☐ | ☐ |
| – Have emergency shutoffs been field tested? _____ | ☐ | ☐ |
| – Results?_____ | ☐ | ☐ |
| Are any structural members bent, rusted, or otherwise show signs of damage?_____ | ☐ | ☐ |
| – Are fueling cans used with this equipment approved type safety cans?_____ | ☐ | ☐ |

## EQUIPMENT INSPECTION (Continued)

- Have the attachments designed for use (as per manufacturer's recommendation) with this equipment been inspected and are considered suitable for use?_____ ☐ ☐

**Portable Power Tools:**

- Tools and Equipment in Safe Condition?_____ ☐ ☐
- Saw blades, grinding wheels free from recognizable defects (grinding wheels have been sounded)? ☐ ☐
- Portable electric tools properly grounded?_____ ☐ ☐
- Damage to electrical power cords?_____ ☐ ☐
- Blade guards in place?_____ ☐ ☐
- Components adjusted as per manufacturers recommendation?_____ ☐ ☐

**Cleanliness:**

- Overall condition (was the decontamination performed prior to arrival on-site considered acceptable)?_____
- Where was this equipment used prior to its arrival on site?_____
- Site Contaminants of concern at the previous site?_____
- Inside debris (coffee cups, soda cans, tools and equipment) blocking free access to foot controls?_____

**Operator Qualifications (as applicable for all heavy equipment):**

- Does the operator have proper licensing where applicable, (e.g., CDL)?_____
- Does the operator, understand the equipment's operating instructions?_____
- Is the operator experienced with this equipment? _____
- Does the operator have emotional and/or physical limitations which would prevent him/her from performing this task in a safe manner?_____
- Is the operator 21 years of age or more?_____

**Identification:**

- Is a tagging system available, for positive identification, for tools removed from service?___

**Additional Inspection Required Prior to Use On-Site**

| | Yes | No |
|---|---|---|
| Does equipment emit noise levels above 90 decibels? | ☐ | ☐ |
| If so, has an 8-hour noise dosimetry test been performed? | ☐ | ☐ |

- Results of noise dosimetry:_____
- Defects and repairs needed:_____
- General Safety Condition: _____
- Operator or mechanic signature:_____

Site Safety Officer Signature:_____

Approved for Use: ☐ Yes ☐ No

## SITE-SPECIFIC TRAINING DOCUMENTATION

My signature below indicates that I am aware of the potential hazardous nature of performing remedial investigation activities at _____ and that I have received site-specific training which included the elements presented below:

- Names of designated personnel and alternates responsible for site safety and health
- Safety, health, and other hazards present at the sites
- Use of personal protective equipment
- Safe use of engineering controls and equipment
- Medical surveillance requirements
- Signs and symptoms of overexposure
- Contents of the Health and Safety Plan
- Emergency response procedures (evacuation and assembly points)
- Initial response procedures
- Review of the contents of relevant Material Safety Data Sheets
- Review of the use of Safe Work Permits

I have been given the opportunity to ask questions and all of my questions have been answered to my satisfaction. I further state, that the dates of my training (introductory, refresher, and supervisory, as applicable) and my medical surveillance requirements are accurate and correct to the best of my knowledge.

| Name (Printed and Signature) | Site-Specific Training Date | 40-Hour Training (Date) | 8-Hour Refresher Training (Date) | 8-Hour Supervisory Training (Date) | Medical Exam |
|---|---|---|---|---|---|
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |

## MEDICAL DATA SHEET

This Medical Data Sheet must be completed by all on-site personnel and kept in the command post during the conduct of site operations. This data sheet will accompany any personnel when medical assistance is needed or if transport to hospital facilities is required.

Project _____

Name _____ Home Telephone _____

Address _____

Age _____ Height _____ Weight _____

Name of Next Kin _____

Drug or other Allergies _____

Particular Sensitivities _____

Do You Wear Contacts? _____

Provide a Checklist of Previous Illnesses or Exposure to Hazardous Chemicals _____

_____

_____

What medications are you presently using? _____

_____

_____

Do you have any medical restrictions? _____

_____

_____

Name, Address, and Phone Number of personal physician: _____

_____

_____

_____

I am the individual described above. I have read and understand this HASP.

_____ _____

Signature Date